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Graphite for Advanced Nuclear Reactors

Deployment Readiness Review

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Deployment Readiness Review

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EPRI Project Manager
C. Wax

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EPRI
3420 Hillview Avenue, Palo Alto, California 94304-1338 USA
800.313.3774 ▪ 650.855.2121 ▪ askepri@epri.com ▪ www.epri.com

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ACKNOWLEDGMENTS

Dominion Engineering, Inc.
12100 Sunrise Valley Drive, Suite 220
Reston, Virginia 20191

Principal Investigators
C. Marks
N. Craft

Idaho National Laboratory
1955 Fremont Avenue
Idaho Fall, Idaho 83415

Principal Investigators
W. Windes
S. Johns
M. Metcalfe
A. Mack
R. Smith

Oak Ridge National Laboratory
5200, 1 Bethel Valley Rd
Oak Ridge, TN 37830

Principal Investigators
A. Campbell
N. Gallego
J. Geringer

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ABSTRACT

Historically, graphite has been used in numerous reactor technologies, including research/test reactors and, commercially, in advanced gas reactors. These non-metallic materials can play a key role as internal core structures, reflectors, or neutron moderators, making them important for deployment of certain advanced reactor technologies. This report explores the industry readiness for graphite material deployment, documenting Codes and Standards applicable to their design, qualification, and manufacturing. Discussions also examine the manufacturing processes, aging/degradation, inspection techniques, and disposal options. The report covers the current status, documents gaps, and proposes some conclusions about approaches to managing some of the current gaps.

Keywords

Degradation
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EXECUTIVE SUMMARY

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Product Title: Graphite for Advanced Nuclear Reactors: Deployment Readiness Review

Primary Audience: Graphite manufacturers, advanced reactor developers, design engineers, component engineers

Secondary Audience: End users interested in advanced reactor deployment

KEY RESEARCH QUESTION

Graphite materials will comprise key components in some advanced reactor designs. Codification, qualification, and deployment of non-metallic materials are new; the industry needs to understand the areas in which gaps remain to support prioritization of resources for gap closure.

RESEARCH OVERVIEW

The information captured in this report is the culmination of literature reviews and documentation of responses from key industry stakeholders. The goal was to take a wholistic approach to the graphite life cycle from design and qualification to manufacturing and deployment in an operating reactor. The information captured also encompasses a review of monitoring/inspection activities, as well as approaches to decommissioning and disposal of graphite. Each of these portions of the graphite life cycle is explored and the current understanding of some gaps are identified.

KEY FINDINGS

- Gaps remain in design, manufacturing, and deployment of graphite for advanced reactors.
- Aging and degradation management will be key to operational programs.
- Numerous Codes and Standards activities are ongoing to clarify graphite deployment requirements.
- Supply chain demand for nuclear-grade graphite is growing, but is still minuscule compared with graphite demand from other industries.

WHY THIS MATTERS

This research provides key insights into industry preparedness for deployment of graphite materials. The information in this report supports prioritization and effective closure of the documented gaps by providing a baseline industry understanding.

HOW TO APPLY RESULTS

This information is meant to be an input into industry plans for gap closure, leading to effective and efficient deployment of graphite materials.

LEARNING AND ENGAGEMENT OPPORTUNITIES

- This work was completed under collaborative agreement with the Department of Energy (Idaho National Laboratory and Oak Ridge National Laboratory). Each has been instrumental in further graphite materials research for the industry.

EPRI CONTACT: Chris Wax, Principal Technical Leader, cwax@epri.com

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1 INTRODUCTION

1.1 Prior Uses in Nuclear

Graphite has been used in multiple experimental nuclear reactors in the US. Enrico Fermi's Chicago Pile 1 utilized graphite as the moderator in 1942. From this development, graphite moderation was used in multiple reactors throughout World War II in order to develop and produce uranium and plutonium [1]. This included the X-10 Graphite Reactor, an air cooled, graphite moderated reactor similar to the advanced gas reactors (AGR) later operated in the United Kingdom.

After World War II, experimental graphite moderated reactors were constructed to develop methods of reliable energy production. The Magnox reactor was the first graphite-moderated, carbon dioxide, cooled reactor brought online in the UK. The Magnox design consisted of a 24-sided, 1,150-ton graphite structure consisting of interlocking bricks and tiles serving as moderators and reflectors. These plants were eventually phased out in the UK in favor of the advanced gas reactors (AGRs) with the first AGR reaching first power in 1976. The AGR design was similar to the Magnox reactors in that they used graphite moderation and were gas-cooled. The AGR produced higher coolant temperatures, expanding the temperature based operational experience of graphite. The use of CO_2 coolant resulted in oxidation of the graphite components. This combined with irradiation damage led to significant mass losses. Above a weight loss of 10%, graphite component integrity is considered threatened due to cracking [2].

The United States shifted to the use of water as a moderator in reactor designs, and graphite was relegated to experimental and theoretical applications. In 1965, the Molten Salt Reactor Experiment was brought online at Oak Ridge National Laboratory. The matrix of close-packed graphite stringers with coolant channels for molten salt to flow allowed for in depth analysis of graphite response to molten salt and irradiation. This also allowed for narrowing of graphite core component design to best survive the environment. Small pores were required to avoid fuel salt penetration, neutron irradiation resistance to inhibit degradation, and low gas permeability to avoid fission product absorption [3]. A specialty grade of graphite was developed for these issues, grade CGB produced by the Carbon Products Division of the Union Carbide Corporation. The increased density and smaller pore size produced through many impregnations and heat treatments allowed for increased stability in the environment with respect to the fuel salts and temperature [4]. This was the first nuclear-specific grade of graphite produced on a commercial scale. While in service, the graphite was not exposed to enough radiation to significantly affect the material properties, but subsequent testing after removal showed that the CGB graphite used would not be sufficient for full-scale, long-term molten salt reactor use [3].

Two high-temperature, gas-cooled reactors were built in the United States using helium coolant. These prototype reactors were Peach Bottom Unit 1 and Fort St. Vrain. These reactors operated from 1966 to 1974 and 1974 to 1989, respectively, and utilized graphite core components such as moderators and reflectors. Fort St. Vrain experienced moisture ingress into the reactor

coolant system, which resulted in oxidation of the graphite core components. However, Fort St. Vrain demonstrated that material models could accurately predict irradiation dimensional changes for the graphite fuel elements [5]. Both of these prototype reactors experienced multiple operational problems, which eventually led to their respective shutdowns.

1.2 Graphite Components for Nuclear Applications

Graphite components can be sorted into three categories which vary based on the reactor design: fuel, reflector, and support elements. These are similar to components that have been used in past reactor designs and their operational experience can be used in advanced reactor designs.

1.2.1 Fuel Elements

Graphite fuel elements are designed to maintain fuel geometry, contain fission products, and in most cases provide channels for coolant fluid to remove heat from the fuel. In most advanced reactor designs, the fuel being incorporated is TRISO fuel particles, either within prismatic fuel blocks or as fuel pebbles.

Prismatic fuel blocks house the fuel, the coolant channels, the control rod channels, as well as any instrumentation used for the fuel or graphite components. Graphite components cannot be joined via welding traditionally done to join metal components. Because of this, graphite fuel blocks will need to be held together with pins or other physical lock and key systems in order to maintain structural form. Graphite has been chosen for fuel blocks due to their ability to moderate neutrons, maintain CTE at increased temperatures, and maintain structural integrity for the core at increased temperatures [6]. Graphite fuel elements will be exposed to the most irradiation as they are closest to the fission reactions.

Pebble fuel elements are a component singularly concerned with maintaining fuel geometry and protection. These pebble fuel elements are tennis ball sized and contain about 9 grams of low enriched uranium. These small, sand-like, particles of uranium are coated and surrounded in a silicon carbide shell which is then embedded in a graphite matrix in the shape of a sphere. These pebble elements use graphite and silicon carbide to ensure the retention of fission products, as well as protect the fuel contained inside [7]. Reactors can vary between including these pebbles in graphite fuel blocks, or utilizing a pebble bed design, such as the Xe-100 reactor [8]. Pebble bed designs allow the fuel pebbles to sit freely in a fluidized bed and cycle out and back into the reactor over the course of the individual pebble's lifetime. Fuel pebbles do have failure mechanisms, including thermal decomposition, irradiation induced cracking, debonding, and pressure vessel failure [9]. Because of the retention of gaseous fission products, fuel pebbles are considered pressure vessels by some laboratories and is considered the primary containment barrier for fission product release [10].

1.2.2 Reflector Elements

Graphite reflector elements are also responsible for neutron moderations as well as reflecting neutrons back towards the active core region in order to promote fission [6]. These are normally solid graphite components that can be subcategorized based on their position within the reactor core and their expected lifetime. Figure HHA-1400-01 from the ASME BPV Code Section III, Division 5 depicts the different reflector locations in a reactor core [11]. The main subcategories for reflectors are as follows:

- Removable reflector elements surround the active core region and are designed with a limited lifetime with relation to the lifetime of the reactor. The typical lifespan of a removable graphite reflector element is typically four to ten years, depending on the core design, at which point peak radiation damage will have occurred for the graphite component [12]. Radiation damage is the major limiting factor in most graphite component designs, as dimensional changes and mechanical property changes will require the graphite components to be removed. Removable reflector elements are designed with this in mind and will be replaced a few times before the core lifetime is reached.
- Permanent reflector elements surround removable reflector elements, if present, and fuel elements. The location in the core allows for permanent reflector elements to remain in the core for the lifetime of the core. These components serve the same purpose as removable reflectors but experience a lower fluence level.
- Some designs include a central reflector, which can be permanent or replaceable, and serves to moderate and reflect neutrons. This component also serves as mechanical support for the core.
- Pebble bed reactor designs employ pebble reflectors as a form of removable side reflectors. These spherical graphite elements are placed closest to the core region and only provide moderation and reflection.

Aside from the pebble bed reflectors, the reflectors are generally designed as free-standing graphite bricks. This design minimizes the thermal and irradiation expansion experienced by the graphite components [13]. Thermal and irradiation damage can cause permanent distortion in graphite components, requiring components like removeable reflector elements to be replaced. Even for permanent reflectors, which ideally have a lifetime of 50 effective full power years (EFPY), may not be able to reach their intended lifetime and could require replacement or removal [14].

1.2.3 Core Support Elements

Core support elements consist of the graphite components which can affect the moderation of the reactor core, but primarily serve as support structures for the core interior. These components are designed with high stress tolerances, beyond the stress tolerances of other graphite components, and are designed to last the lifetime of the plant [6].

1.3 Graphite Use in Advanced Reactors

1.3.1 Advanced Reactor Designs

Currently, the major advanced reactor designs being considered are high temperature gas reactors (HTGRs), molten salt cooled reactors (MSRs), lead or sodium cooled fast reactors (LFRs and SFRs respectively), gas-cooled fast reactors (GFRs), and supercritical water cooled reactors (SCWRs). Of these designs, MSR and HTGR designs are considering the use of graphite as moderating and structural components due favorable strength, coefficient of thermal expansion, and thermal conductivity at high temperatures. In the case of MSR technology, graphite does not react with molten salts and is, therefore, not at risk of chemical degradation. MSR technology also operates in the thermal neutron spectrum, allowing for graphite to act as the moderating material as well as the structural material for the core. HTGRs are considering graphite use for almost entirely the same reasons. HTGR technology is considering helium for its coolant gas, which does not react with graphite, and the high temperatures of the reactor core will exclude traditional metals used in current reactor designs. HTGR designs also operate in the thermal neutron spectrum, allowing for the same dual use of graphite for both moderation and structural integrity.

For other advanced reactor designs, graphite is not as attractive. For SCWR technologies, graphite is easily oxidized in water coolant and would quickly lose effectiveness and strength through degradation. Graphite oxidation in water can quickly lead to loss of mechanical properties, requiring constant monitoring and replacing if implemented. Due to this, and water already being an adequate moderator for SCWR designs, graphite has not been considered. For SFR, LFR, and GFR designs, despite chemical stability when in contact with the coolant in these reactor designs, fast spectrum reactors do not require moderators, and introduction of graphite structural components could negatively affect the neutronics of the reactor core.

1.3.2 Overview of Anticipates Usages in Example ARs

Most technical information on the use of graphite for specific components is proprietary, Available information is summarized in Table 1-1. Within all of the graphite grades and components being considered for advanced reactor design, the *sameness* of the graphite grades is a necessity. This *sameness* refers to the repeatability of nuclear grade graphite performance between the different stages of processing, including the raw materials used to develop the graphite. Repeatability of nuclear grade graphite performance is necessary to provide confidence in the graphite core component designs being considered by advanced reactor designers.

Table 1-1. Example graphite components in advanced reactor designs

Reactor & Vendor	Graphite Component Incorporated in Design					
	Fuel Element	Removable Reflectors	Permanent Side Reflectors	Center Reflector	Pebble Reflectors	Core Support Structures
KP-FHR Kairos Power [15]	240,000 Pebbles	Not Present	Rings of 25, 0.5 m tall blocks 43,310 kg	10 EFPY lifetime, replaceable 5,940 kg	220,000 Pebbles 0.05-5.0 mm diameter	Present
HTR-PM INET [16]	Pebbles	Not Present	Present	Not Present	Not Present	Present
MMR UltraSafe Nuclear [17]	TRISO in prismatic block	Unknown	Unknown	Unknown	Unknown	Unknown
U-Battery** UltraSafe Nuclear/NNL [18]	Prismatic block	Not Present	Present	Present	Not Present	Present
eVinci* Westinghouse	Unknown	Unknown	Unknown	Unknown	Unknown	Unknown
XE-100 X-energy [19]	TRISO in spherical pebble	Not Present	Present	Not Present	Not Present	Present
BANR BWXT [20]	TRISO in spherical pebble	Unknown	Present	Unknown	Unknown	Unknown
Horizontal Compact HTGR MIT [21, 22]	Rectangular Block	Rectangular	Present	Present	Not Present	Present
Integral MSR, Terrestrial Energy [23]	Not Present	Unknown	Present	Not Present	Not Present	Unknown
LFTR* Flibe Energy [24]	Not Present	Unknown	Unknown	Unknown	Not Present	Present

* Limited Information Available

** U-Battery no longer under development

1.4 Emphasis on Technology Readiness

Despite the use of graphite in the past as components for research and development reactors, graphite use in the nuclear power industry represents a small fraction of existing production capability. Manufacturing of graphite is primarily focused on electrode manufacturing and metallurgy. The requirements necessary to produce nuclear grade graphite are not used for mass graphite production. Additionally, graphite is a complex, heterogenous material with high amounts of natural anisotropy. This can lead to inconsistencies in graphite products, from production lots to material properties across singular components. Because of these inconsistencies in the graphite properties, as well as the lack of experience in nuclear graphite manufacturing, there are technological readiness gaps related to nuclear grade graphite for which closure is necessary to provide reactor manufacturers, regulators, and potential owners/operators with confidence in the components being produced. Traditional reactor materials have a long history of use, and that history has been applied to further development of these materials, as well as to provide confidence in designs and construction. Nuclear graphite, while beginning with the history of reactors, has been largely removed from consideration as a material in the U.S. and other jurisdictions, and in some cases, companies no longer have the knowledge necessary to produce graphite materials used in past reactors, such as the MSRE [4].

Many reactor manufacturers are combatting this problem through the use of demonstration reactors. These controlled, heavily monitored, and in some cases scaled down versions of their intended commercial advanced reactors will serve to provide regulators with confirmation that their design is safe and reliable. Codes and standards committees can use the results of these demonstration reactors to determine operating limits, manufacturing limits, and monitoring limits for graphite core components through the information gathered during the test period. However, these demonstration reactors will be held back by the supply chain and its technological readiness for large scale nuclear graphite production. As mentioned above, not all graphite manufacturers are experienced in nuclear graphite production, and inconsistencies between them can result in different reactors having different experiences with components that should on the surface behave quite similarly.

2 CONCLUSIONS AND RECOMMENDATIONS

Graphite as a material for nuclear reactor core internals still faces many challenges before it can be successfully and confidently incorporated into commercial reactors. Throughout this report, knowledge and technological gaps have been discussed concerning nuclear grade graphite and graphite core components. Actions taken to reduce these gaps will need to come from all of the organizations involved in the production, acceptance, regulation, and implementation of nuclear grade graphite. The following sections identify broad categories of work needed to close gaps to technological readiness. Note that these categories are cross-cutting and affect many different aspects of reactor design and component procurement.

2.1 Aging and Degradation

Graphite degradation in advanced reactor environments is lacking definition and sufficient information to create standards and regulations. Without a greater body of data demonstrating how graphite is affected by different potential advanced reactor environments, advanced reactor designers do not have a good basis with which to bound their designs. Cooperation between researchers and regulators is needed to build greater understanding and, using that understanding, to develop more cohesive and encompassing codes and regulations. This effort will allow designers more confidence in their design envelopes in terms of degradation and potentially allow for nuclear graphite manufacturers to refine their production processes with new information to create a more resilient product.

The challenges facing this process occur due to the lack of experience with graphite in advanced reactor environments. For most materials, the conditions within HTRs are outside of their operational experience, and graphite is no different. Graphite core components will experience a variety of degradation modes including irradiation, oxidation, coolant abrasion, coolant intrusion, and thermal fatigue. Research on nuclear grade graphite properties with respect to the variety of degradation modes present in advanced reactors will provide a better understanding for the limits on graphite use. These limits can then be used to inform codes and standards developed to bound graphite core component design. With these bounds in place, advanced reactor designers can make confident design choices for their advanced reactors.

ASME BPV Code Section XI, Division 2 describes the reliability and integrity management (RIM) programs focused on the monitoring of plant materials in operation. Currently RIM programs are focused on metallic components in light water reactors (LWR) and there is current ongoing work to change this focus to include other materials, such as nuclear grade graphite. Without the understanding of how graphite will age and degrade in advanced reactor environments, informed RIM programs cannot be developed. This is a failure point for designers, as all U.S. nuclear plants need to have a monitoring program in compliance with U.S. code of federal regulations in order to operate. Without codes and standards for graphite monitoring, and without an understanding of graphite's aging properties, it is not possible to develop further designs for graphite core components. Finally, another major hurdle to the further development of RIM concepts is the lack of specific performance requirements information for

actual components, due either to the fact that such components are still in the early design phase or due to the proprietary nature of the designs. Without a specific performance requirement, it is not possible to determine the extent to which degradation modes would compromise the ability of a specific component to perform its design requirements.

There is a fundamental deficiency in the data available for irradiation behavior of modern graphite supplier grades. As yet, there is no overarching model that would allow the prediction of irradiated behavior from as-constructed behavior. Therefore, additional irradiation testing is required.

The interaction of graphite with molten salts intended as the primary coolant in some advanced reactor designs is an area of active research. The interactions include chemical reactions, tribological phenomena, and the intrusion of fueled salt into the porous structure of graphite components. Although graphite is generally considered to be a non-reactive material, current research indicates that there may be significant concerns with long-term exposure to molten salts. Few data are available regarding the interaction of graphite with molten salts under irradiation.

Fatigue of graphite has not been sufficiently characterized. Complicating factors include the potential effects of irradiation on fatigue properties as well as environmental fatigue issues, especially in molten salts.

2.2 Consistency of Properties

The consistency of properties in manufactured graphite is a major hurdle in the design, acceptance, and production of graphite core components. Nuclear grade graphite currently contains a large amount of variability within individual billets, for example, up to a 90% difference in the density across nuclear grade graphite billets [25]. Inconsistency in manufactured properties exist across the production of graphite, with differences between different nuclear grades, orders, production lots, billets, and components affecting the ability of designers to accurately predict the behavior of components in service and affecting the ability of regulatory bodies to develop methods for accepting design envelopes for nuclear grade graphite.

The issue of graphite inconsistency is currently being studied in an attempt to understand the mechanisms creating this amount of variation. Raw materials are a starting point, and ASME and ASTM standards have been put in place to ensure that nuclear grade graphites are always manufactured with the same raw materials in order to avoid requalifying the particular nuclear graphite grade [11, 26, 27]. However, more clarity on what constitutes *sameness* in the raw materials may be necessary if large quantities of graphite will be needed over an extended period. Further studies from researchers will be needed to continue to close gaps on the understanding of nuclear graphite variation. While raw materials may be a controllable factor, billet, lot, and order variability will need to be understood to create design limits and more complete qualification methods for graphite. Research on nuclear grade graphite properties and responses to reactor conditions are ongoing and their findings will aid in the development of ASME and ASTM regulation. Collaboration between researchers and manufacturers has

begun with projects like the baseline graphite characterization program through INL. Programs such as these aim to expand the concept of design by test by developing a database of tests for each graphite grade throughout the production process. Methods such as this will require a larger industry focus in order to create a large enough database to source component properties and models for qualification and development.

Issues of consistency are also a major factor in pre-service inspections.

Significant work has been carried out by the U.S. DOE in its Baseline Graphite Characterization (BGC) program. However, the data from this program are not generally available to the industry or to regulators for assessment. Additionally, there are data from other testing programs by other organizations that have not been considered in conjunction with the results of the BGC program. Thus, there is a gap in the availability of data to the industry.

2.3 Supply Chain Readiness

An assessment of the mass of graphite needed to supply a robust expansion of nuclear power through deployment of advanced reactors using graphite components indicates that the nuclear industry's graphite needs will continue to be a very small fraction of the total global production. However, there appear to be no graphite suppliers that are capable, at this time, of supplying material that has appropriate nuclear safety related quality assurance. This represents a major gap. This issue has significant overlap with the issue of material consistency.

The U.S. DOE is beginning a project that will assess supply chain readiness. EPRI is producing a report concerning the procurement of nuclear grade graphite and the requirements for that process.

2.4 Component Preparation

For metals used in LWRs, component preparation has been a key element of subsequent performance as well as the effectiveness of various inspection techniques. Most understanding of preparation requirements for these metal components has come from operating experience. Such experience is lacking for graphite, and an understanding of the importance of various preparation requirements and controls is not well understood. Furthermore, graphite suppliers generally do not have the long-term quality assurance record keeping programs (see Section 2.3) that would facilitate the development of guidance as experience is accumulated. Gaps related to preparation of components include issues such as NDE at intermediate stages of production, quality assurance requirements, and cleanliness and surface preparation requirements.

2.5 System Performance

Much of the research performed on graphite as a suitable material for advanced reactor construction has focused solely on graphite performance in isolation, i.e., not considering graphite components as part of a larger system including other materials. Considerations in the context of system performance include the following:

- The potential for impurities to accelerate degradation
- The potential for graphite to accelerate degradation of non-graphite components

2.6 Summary

In general, the following list summarizes the identified gaps:

- Clear definitions of what constitutes similar or identical graphite. A generally accepted definition is required for the industry to successfully qualify components. Such a definition would need to be supported by significant quantities of data spanning different manufacturers, different grades, and different periods (times) of manufacture.
- Understanding of the relationship between as-manufactured properties and the ability to achieve performance requirements, including the effects of irradiation.
- Understanding of the effects of aging and degradation on the performance of components in the full range of environments (temperature, irradiation, chemical) for full design lifetimes.
- Interactions between graphite and molten salts (including chemical reactions, tribological phenomena, and fueled salt intrusion) are the subject of active research. A related area under active investigation is the effect of graphite particles in molten salt on the degradation of non-graphite components, e.g., by carburization.
- Fatigue of graphite is not well characterized, especially for irradiated graphite or graphite exposed to molten salt.
- Supply chain readiness is an issue, not so much with respect to quantities, but with respect to qualifications, including appropriate quality assurance and an appropriate understanding of the extent to which different individual components can be considered to have been manufactured from the same material.
- Manufacturing controls, including in-process NDE, record keeping, and cleanliness are not codified.

For the first five of these gaps, additional irradiation testing is expected to be necessary.

To a significant extent, many of these gaps are being addressed by the U.S. DOE and various committees or working groups of the ASME BPVC. However, there appears to be no significant industry efforts to address supply chain readiness issues and manufacturing controls.

In some cases, it will not be possible to fully close gaps until the design requirements of specific components are sufficiently defined to allow assessment of the effects of degradation on performance. That is, it is not enough to fully characterize the degradation modes of graphite. These degradation modes must be assessed against specific component functional requirements (including component lifetime) in order to determine the significance of the degradation modes.

3 GRAPHITE MANUFACTURE

3.1 Overview of Graphite

Graphite is a crystalline allotrope of carbon. Made up exclusively of carbon atoms, graphite is a system consisting of compact stacks of graphene layers. Each layer of graphene consists of a single sheet of hexagonal carbon rings. These stacked graphene layers are offset from each other to produce an ABA stacking pattern and held together through Van Der Waals forces. The plane parallel to these graphene layers is known as the basal plane. The plane perpendicular to the graphene layers, normally depicted along the side of graphite crystals, is known as the edge plane. This structure is shown in Figure 3-1, below. Due to this crystal structure, single crystals of carbon are anisotropic, with different materials properties in the <c> and <a> axes, also shown in Figure 3-1, below.

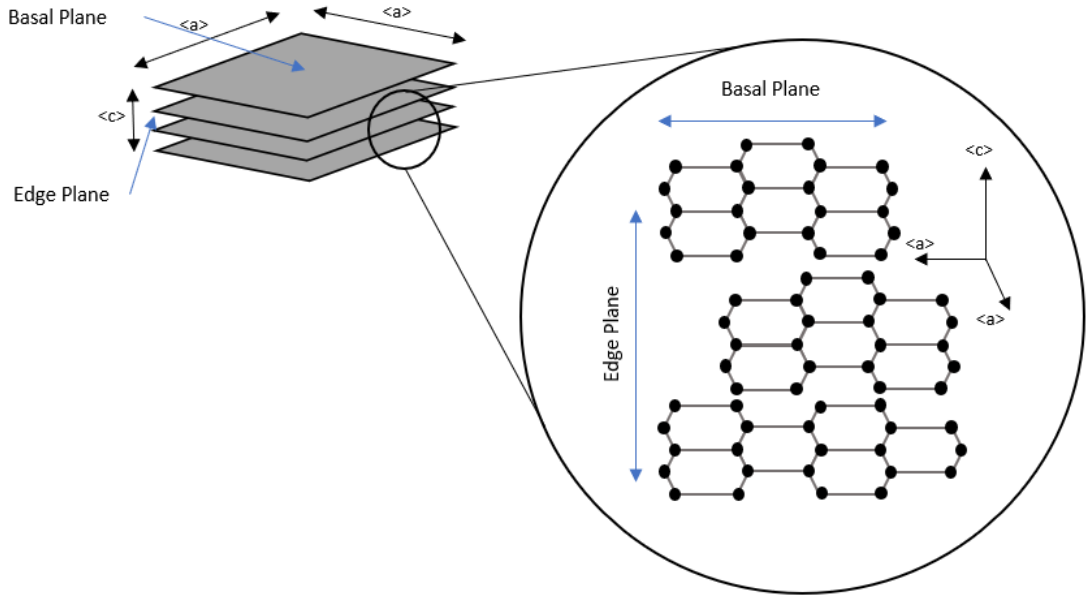


Figure 3-1. Depiction of graphite crystal structure

Graphite sublimates at 3600°C (6512°F) allowing it to survive extreme temperatures at which many normal materials would fail or melt. Graphite retains its strength at increased temperatures. This, along with a low coefficient of thermal expansion (CTE) and high thermal conductivity up to high temperatures, makes graphite useful for reactor environments. Due to the anisotropy inherent in graphite, it is important to ensure, through processing, that the average grain size of graphite materials is small. This allows for closer to isotropic behavior for graphite components and distributes the thermal conductivity and CTE among the principal directions on the components.

3.2 Graphite Processing

Because nuclear reactor designers are only considering synthetic, polycrystalline graphite, that will be the focus of this report. Polycrystalline graphite is intended to be pure carbon. The raw material used to create synthetic graphite, generally a petroleum coke filler with a coal-tar pitch binder, can contain other chemicals as well as other allotropes of carbon depending on its source. Raw materials can affect graphite material properties significantly, and, because of this, the ASME and ASTM guidelines for nuclear grade graphite production require that a nuclear grade use the same raw materials each time in order avoid the need for requalification. The petroleum coke filler is mixed until the desired grain size is obtained and to remove as much volatile matter as possible. The coke filler is then mixed with the pitch binder and shaped through one of the three following methods [11]:

- Extrusion involves mechanically pushing the slurry mixture through an opening (die) to acquire a desired shape. This extruded mass will be cut off at the end closest to the hole, which can create dissimilar properties compared to the beginning of the extrusion. Extrusion can introduce anisotropy in the final shape due to alignment of filler material with the direction of flow.
- Vibration molding is a process of placing the filler-binder mixture in a mold that can vibrate. As the mold vibrates, pressure is applied in order to force the mixture into the desired shape. The vibration system produces more isotropy compared to extrusion molding.
- Isostatic pressure is similar to the method of the same name in powder metallurgy. The graphite slurry mixture is placed in a thin mold of the desired shape, and high pressure is used to force the slurry into the desired shape. This pressure is produced through gas intrusion around the mold in order to apply even pressure at all points.

After mixing and shaping, the filler-binder mixture is baked at around 1200°C (2192°F), binding the graphite grains from the petroleum coke and coal pitch together. During baking the binder is burned off through destructive distillation in order to form an infusible carbon bond [11]. The graphite can be impregnated with more petroleum pitch and baked again in order to increase density. This process can be performed several times until the desired material properties are obtained. From here the entire product is graphitized at 2500°C to 3000ºC (4532°F to 5432°F). Graphitization is the process of heating amorphous carbon material in order to the desired graphite structure through time and temperature. Gaseous agents can be used to remove some impurities and are necessary to obtain high purity nuclear grade graphite [28].

3.3 Material Response

3.3.1 Microstructure and Pore Structure

As discussed above, the crystal structure of graphite consists of offset stacked hexagonal sheets. Nuclear graphite has a few requirements detailed in ASTM D7219-08 and ASTM 7301-08 regarding the microstructure of manufactured graphite. Nuclear grade graphite can be defined as medium grained, fine grained, superfine grains, ultrafine grained, and microfine grained. The

grain size of nuclear grade graphite is required to be below 4 mm to be considered medium grained. However, the maximum filler particle size allowed by the ASTM standards is 1.68 mm. Because there is not much grain growth during the graphitization process, this results in a practical limit on the grain size of nuclear grade graphite of 1.68 mm [26, 27].

The isotropy ratio is used to assess the differences in properties due to orientation. This isotropy ratio is based on the CTE and is determined by measuring with the grain and against the grain on extruded graphite. Extruded graphite will have grains in the extrusion direction (extrusion, as mentioned above, can result in the alignment of the filler particles). This can lead to unacceptable anisotropy. The CTE isotropy test is designed to identify such unacceptable anisotropy. A ratio is taken of the CTE with the grain and the CTE against the grain. The highest allowed value for the CTE isotropy ratio is 1.15 for any manufacturing method [26, 27].

Graphite is a porous material, with manufactured graphite generally containing at least 20% porosity. These pores are formed from the constituents used during processing as well as the manufacturing methods. Different forms of petroleum pitch will provide different amounts of porosity, and binder outgassing during baking will create pores from trapped gas. Pore morphology controls the mechanical, thermal, and electrical conductivity properties of graphite as well as the evolution of cracks under mechanical load. Larger amounts of porosity lower the Young's modulus, the CTE, and electrical conductivity [29]. Porosity can also arise from cracks that develop during cooling from graphitization temperatures, generally hypothesized to be due to the anisotropic CTE at the crystalline scale. These cracks are known as Mrozowski cracks [30]. It is a relatively unique aspect of graphite as a structural material that it begins its service life essentially guaranteed to contain flaws which have no impact on performance.

3.3.2 How Microstructure Affects Degradation Response

As mentioned above, porosity and isotropy can affect the mechanical, CTE, and electrical conductivity properties of nuclear grade graphite. Porosity also plays a major role in the response of graphite to irradiation. As graphite is irradiated, changes in the porosity determine the changes in the material properties of the graphite component. Irradiation of graphite results in anisotropic swelling due to the dislocation of carbon atoms from their crystallographic orientations. The dislocated carbons can be relocated to a position between two basal planes. This causes expansion in the <c> direction with contraction in the <a> direction [31]. This anisotropic expansion continues through the early stages of irradiation and pores known as accommodation pores absorb the swelling [29]. Once these accommodation pores have absorbed all the swelling available to them, the contraction in the <a> direction stops and bulk swelling is observed with expansion of existing porosity and generation of new pores. As pores are closed and formed, the mechanical properties as well as CTE and electrical conductivity of graphite changes [29]. The result of this is the turnaround points observed for different properties of graphite core components and discussed in later sections of this report.

Another issue that is affected by microstructure is the potential for certain coolants to infiltrate graphite and create problematic stress points. When immersed in molten salt coolant, graphite absorbs molten salt into its porous structure. The amount absorbed is controlled by the

connectivity and size of pores, which in turn are derived from the density of the graphite and the grain size. During outage periods, molten salt coolant may change phase within the graphite pores, potentially damaging graphite. Molten salt with fuel dissolved into it can create hot spots within graphite if absorbed, also causing stress imbalance across the graphite component. Studies are currently being performed on the molten salt infiltration in different grades of graphite, with large discrepancies based on processing parameters and resultant porosity [32].

Oxidation of graphite is controlled by the amount of edge planes available for oxidizing species. Edge planes have dangling carbon bonds that can be easily oxidized, resulting in more dangling bonds, continuing the process. As with other material properties, the amount of edge planes will be affected by the porosity and therefore the density of the manufactured graphite [32]. Highly dense graphite is not ideal for components expected to experience significant irradiation as inhomogeneous irradiation will cause an imbalance in expansion, which can result in cracking if the graphite is too dense [29].

Because of different processing methods and materials available for graphite manufacturing, different nuclear grade graphite grades will have different characteristics, most ultimately derived from the density and grain size. These include the amount of accommodation porosity available. Even within individual orders, lots, and billets of graphite there is heterogeneity within the graphite produced. This will result in varying material properties, as well as variation in the response to degradation from irradiation, coolant infiltration, and oxidation.

4 PRE-SERVICE ISSUES

4.1 Assessment for Service

Assessment of the readiness of graphite core components for service and installation requires an understanding of the current gaps in the guidance and specifications for graphite manufacturing and examination. Records of examination, as well as records of raw materials, process parameters, and deviations are needed in order to ensure a component is processed correctly and acceptably to meet the quality requirements for reactor service. Graphite does not have the same guidance and specification for these records when compared to metal components, and the analyses being completed and recorded are not in an adequate technological state to support graphite manufacturing and development. This chapter discusses the knowledge gaps related to the acceptance of completed graphite core components and the requirements needed or missing that will allow for confidence in the properties being reported for manufactured graphite materials.

4.2 Definitions of Material Groupings

Pre-service evaluation and acceptance of a component will generally require some confidence in the properties of the component that can only be obtained by destructive methods. Therefore, it is generally necessary to have a material grouping in which such properties are sufficiently consistent that evaluation of a sacrificial member of the group provides acceptable confidence in the properties of all other members of the group.

Heat is a term that is primarily used in the world of metal manufacturing. The definition of heat for metals is clear and precise, resulting in consistency in the reporting of material properties and traceability in the manufacturing process. For metals, a heat consists of the material from one single molten batch. For remelting of an ingot, each remelted charge is a separate heat [33]. The chemistry of the ingot is generally considered sufficiently characterized by analysis of any part of the heat. A *lot*, for metals, is a quantity of materials made using the same heat and the same manufacturing process. Consumers rely on these definitions for knowledge of how their components were processed and whether individual components differ from each other in processing or composition.

For graphite, a comparable definition of *heat* or similar term has not been universally adopted. Specifically, ASME, ASTM, and graphite suppliers do not use a consistent set of terms to describe material groupings.

The ASME BPV Code Section III, Division 5 does not define *heat*. However, definitions for other material groupings of graphite through the manufacturing process are provided in HAB-9000 and are described below [11]:

- A graphite core component is defined as a single machined graphite item complying with the requirements of subpart HAB and subsection HH, subpart A. Graphite core components are machined from billets and must comply with the applicable ASTM specifications per table HAB-7100-1.

- A graphite *billet* is considered by the ASME Code in HAB-8000 to be an extruded, molded, or isomolded graphite artifact with dimensions sufficient for the designer.
- The graphite *charge* is defined in the ASME Code in HAB-8000 as the billets of the same graphite grade in the same graphitizing furnace run.
- The graphite *grade* is defined by the ASME Code in HAB-8000 as the designation given to a graphite material by a manufacturer such that it is always reproduced from the same raw materials to the same specifications using the same process.

The ASME code uses lot numbers, charge numbers, and grades of graphite as requirements for identification of completed material in HAB-4556.3. The use of each of these terms, without a definition for graphite *lots* leads to confusion in the industry.

Graphite manufacturers have developed their own definitions for the different stages of graphite production. Graphite manufacturers generally refer to lot and charge with the same definition. However, the term lot is used more prominently in manufacturing. The following are the groupings used by manufacturers to describe the graphite manufacturing process [10]:

- Manufacturers define a *billet* as a single body of graphite from which machining is required to form any components.
- Graphite manufacturers define a *lot* as a set of billets processed and graphitized in the same manner.
- Graphite manufacturers define a higher order grouping for graphite production. This term is a *batch*, which is a single order of graphite material that can comprise several lots.
- Manufacturers consider a graphite *grade* to be the same as the ASME definition: graphite material made from the same raw materials using the same processing techniques and parameters.

Graphite manufacturers define their identification numbers based on these definitions. Confusion with respect to what each organization thinks is being referenced can originate for the designation numbers assigned to the different quantities described above in graphite manufacturing.

ASTM D7219-08, the standard specification for isotropic and near-isotropic nuclear graphites, and D7301-08, the standard specification for nuclear graphite suitable for components subjected to low neutron irradiation dose, also defines the material grouping of graphite, with differences from both the ASME BPV Code and the graphite manufacturers. These groupings are described below from the definitions section of the ASTM standards, Section 3 [26, 27]:

- The ASTM standards define *billets* in the same way that the ASME BPV Code defines billets, which is also consistent with the terminology used by graphite manufacturers.
- The ASTM standards define a *graphitization charge* as the total number of billets graphitized together in one graphitization furnace, i.e., similar to the ASME.

- The ASTM standards define a *production lot* as a specified number of billets in a purification run. This is not consistent with the use of *lot* by manufacturers interchangeably with *charge*. Lot is used in the ASME BPV Code with reference to graphite but is not defined.
- Graphite *grades* are defined by the ASTM in a similar way to the ASME and graphite manufacturers, graphite material manufactured from the same raw materials using the same processes and techniques. The ASTM does define the same mix formulation, which is a step farther than the ASME definition.

The ASTM standards for nuclear graphite grades also include a breakdown of class for nuclear graphite with respect to processing methods. These classes include isomolded, extruded, and molded, each with a specification for high and low pressure processing. These is also designation for graphite based on grain size, from medium grain, to microfine grains. However, these are just a designation, as the only requirement on grain size is a maximum grain size of 1.68 mm [26, 27].

The misalignment in standard terms among the ASME, the ASTM, and the graphite manufacturers leads to complications in the qualification process of graphite material. Currently the ASME process allows for the graphite manufacturer to qualify a batch as a specific grade based on the use of the historical data for that grade. Article HHA-III-5000 of the ASME BPV Code Section III, Division 5 states that no new testing is required if the required test data are already generated and meet the requirements of the HHA appendix for the same graphite grade [11]. This allows for new batches to be ordered for a grade that has been qualified previously without any further testing. This can include production runs that are a decade old, which runs the risk of the coke material from the supplier changing in that time, producing billets with potentially different properties, but technically of the same grade. This brings into question the validity of the "sameness" of the raw materials being received. Raw materials such as petroleum coke can vary based on mining location and can even vary within the same deposits. This can cause problems for the consistency of graphite grades, and the charges, billets, and graphite core components created under these grades [10].

Specifically for pre-service acceptance of components, the following gaps are noted:

- There is technical uncertainty in the consistency of material groupings such that destructive analysis of members of a group cannot be relied upon to provide an indication of the properties of an actual component that is a member of the same group.
- There is uncertainty arising due to the multiple terms used to describe different material groupings, some of which have different meanings in different parts of the industry.

Although these gaps are similar to those facing designers attempting to determine the acceptability of historical data for ASME qualification, they are distinct in that they are specific to the ability to determine the acceptability of a specific instance of a component, i.e., an actual physical part received for use in assembling a reactor.

4.3 Consistency

The sections below discuss general issues of consistency among different subset of graphite (Section 4.3.1) and the available results of a DOE program to evaluate the extent of inconsistency across example subsets (Section 4.3.2).

4.3.1 Consistency of Various Groupings of Graphite

The previous section discussed different material groupings of graphite used at various points in the manufacturing process. These are the grade, lots, billets, and core components according to the graphite manufacturers. While these quantities are not consistent in their naming across the ASME Code, the ASTM Standards, and the graphite manufacturers, each of these groupings also have physical inconsistencies within the group. These inconsistencies are caused by the inhomogeneity that is inherent throughout the graphite manufacturing process. Unlike metals, in which the constituent materials and elements are melted into a liquid, removing any pre-manufacturing microstructures or phases, graphite carries certain properties of its raw materials through the entire manufacturing process. Attributes of the petroleum coke or coal tar coke, raw materials used in the graphite manufacturing process, are maintained with very little change through to the graphite core component. This can cause variability among graphite parts of the same charge in some cases, leading to variability in the performance of graphite core components of the same grade, within the same charge, and sometimes cut from the same billet. Properties of the final component that can be affected include microstructure, particle size, and coefficient of thermal expansion, as well as the anisotropic tendencies in these parameters. The Baseline Graphite Characterization (BGC) Program at INL attempts to describe the extent of consistency, e.g., within billets, billet to billet, charge to charge, and grade to grade [34].

The graphite forms that will be discussed in the rest of this section will be described using the ASME BPV Code definitions consisting of grade, charge, billet, and graphite core component. The material in the groups defined by these terms have inconsistencies, such as inconsistencies from billet to billet, charge to charge, etc. The inconsistencies are described as follows:

- A graphite grade is defined in the ASME BPV Code Section III, Division 5 Article HAB-9000 as a designation given to a graphite material by a manufacturer such that it is always manufactured to the same specifications, using the same process, and reproduced from the same raw materials. Between different nuclear graphite grades, differences in properties are expected. Raw materials and forming processes will produce different graphites from different companies that will both be considered nuclear grade graphite. Within a singular graphite grade, inconsistencies are being researched by the BGC program at INL.
  - The specifications for graphite grades are defined by the ASTM standards for nuclear graphite, D7219-08 and D7301-08. Both of these standards are comprehensive for the specifications needed to determine a nuclear graphite grade in terms of grain size, equivalent boron content, isotropic ratio of CTE, and the physical properties of graphite [26, 27].

  - The processes used to form nuclear graphite grades are also defined by the ASTM guidelines as the classes of nuclear graphite. These include isomolding, extruding, and molding process at high and low pressures. These processes are repeatable per the manufacturers of nuclear graphite grades [26, 27].
  - The issue of consistency in nuclear graphite grades arises from the raw materials used to create nuclear graphite. Petroleum oil and coal tar are the two materials that are allowed for use to create the coke for graphite manufacturing [26]. Companies that provide coke to manufacturers do not have specifications for their petroleum oil or coal tar coke, leading to potential variation due to the coke being an integral part of the graphite microstructure. Due to the difficulty graphite manufacturers have placing specifications on raw materials, changes in the raw material may not be reflected by the provider, leading to changes in a graphite grade overtime without control [10]. Requirements for the procurement of graphite raw materials, and potential requirements for graphite raw material production may be necessary to ensure that consistency within graphite grades is maintained.

- A graphite charge is defined by the ASME Code as all the billets of the same graphite grade in the same graphitization furnace run. Since differences within a grade can persist through to a single furnace load, all of the within-grade variations can exist within a charge. Additionally, differences within a charge can exist due to the nature of thermal gradients within the graphitization furnace. The temperature of graphitization is addressed by the ASTM specifications through testing each billet for a specific electrical resistivity corresponding to 2700ºC per section 5.6.3 of the ASTM standards. This allows for a thorough check of the graphitization temperature of each billet, and records of both the graphitization furnace run, and the billet furnace location, required by Section 8 of the ASTM specifications allow for the reactor vendors to have confidence in their lack of charge variations from graphitization [26, 27].
- The BGC program has been able to provide information on an extruded petroleum coke graphite grade, as well as a vibration molded pitch coke graphite grade, and has reported variations in various mechanical properties through the length and width of the graphite billet. The reason for these variations have not been defined yet, but properties such as density, compressive strength, tensile strength, shear modulus, and elastic modulus have been shown to vary across the billet itself. This can lead to variation in graphite core components cut from the same billet [34]. (Further discussion of billet inconsistencies are provided below.) Graphite core components are sectioned from billets and in most cases, multiple components come from the same billet. As mentioned above, similar components could be cut from the same billet and not be identical due to property variation across the billet. Large enough components could experience that property variation within the component itself. As discussed below, this variation could be a difference of as much as a factor of 10 for the compressive strength in extruded graphite grades as well as large variations in other physical properties. Inconsistent density can affect neutronics and moderator effectiveness, while also giving way to larger flaws in less dense areas, potentially causing components to fail inspections. This could lead to local areas of

abnormal physical properties or microstructure. This potential variation will require reactor vendors to extensively test their components as they receive them if this kind of inconsistency is not resolved. Assessment of the extent to which variability in material properties is a significant issue depends on several factors with their own unknown aspects, including the following:

- The relationship between initial condition variability and the response to aging (including irradiation)
- The extent to which differences in properties affect the ability of a component to perform its design function
- The extent to which differences in properties would preclude use of prior testing of a different sample for qualification

4.3.2 Results from the Baseline Graphite Characterization Program

The Baseline Graphite Characterization (BGC) program has analyzed two billets of vibration molded graphite and three billets of extruded graphite. This analysis has been reported in Reference [25] and a discussion of this analysis is provided. This discussion focuses on the variations contained within the billets with respect to different material properties.

The density of the vibration molded (VM) and extruded (EX) graphite grades varies throughout the billets, with the VM graphite grade exhibiting less variability. This variation is shown in Figure 4-1. The density measurements vary from 1.88 g/cm^3 to 1.775 g/cm^3 for the VM graphite, and 1.85 g/cm^3 to 1.71 g/cm^3 for the EX graphite excluding outliers. These variations, while not large and above the minimum density required for nuclear grade graphite (1.7 g/cm^3 [26, 27]) will affect the precure neutronics and the predictability of the moderated neutron flux. Density variation does provide a prediction for the relative variation between the mechanical properties of the graphite billets [25].

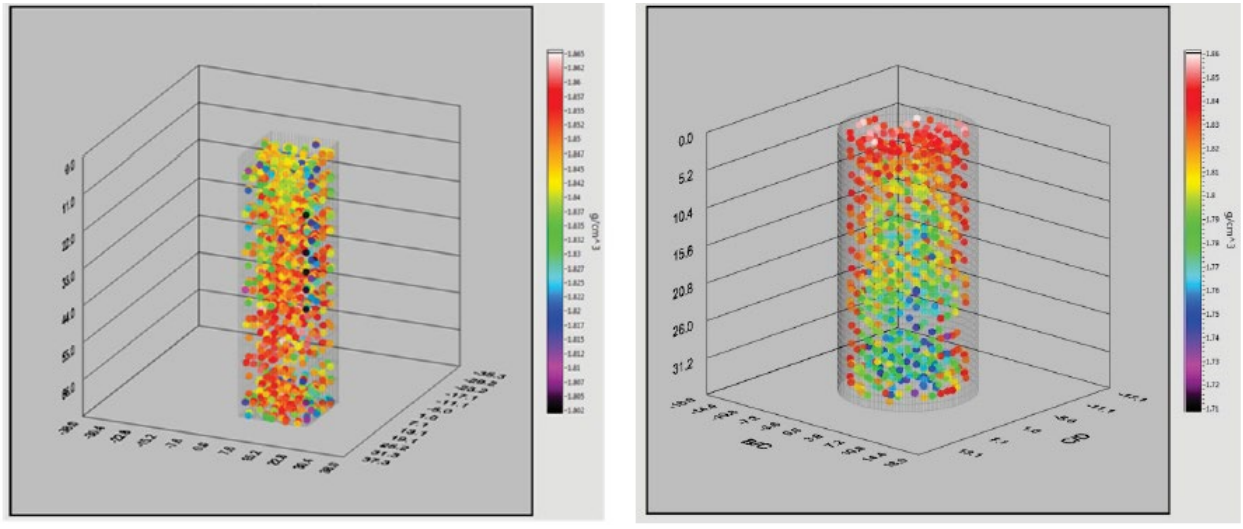


Figure 4-1. Comparison of density measurements in the VM (left) and EX (right) graphite billets [25]

Ultimate tensile strength (UTS) of the VM graphite varies much less than the EX graphite. VM graphite UTS varies from 24.9 MPa to 12.5 MPa, while the EX graphite varies from 25.1 MPa to 2.5 MPa excluding outliers. A Weibull plot of this variation is shown in Figure 4-2. This property variation is shown to vary through the height of the EX graphite billet, with a trend of decreasing values from the top to the bottom of the billet. The VM graphite displayed a relatively homogenous distribution. However, when comparing each of the EX graphite billets individually, the decreasing UTS trend is not as obvious. Each of the graphite grades discussed exhibit UTS measurements below what is specified by the ASTM standards D7219-08 and D7301-08, 15 MPa for both grades [25, 26, 27].

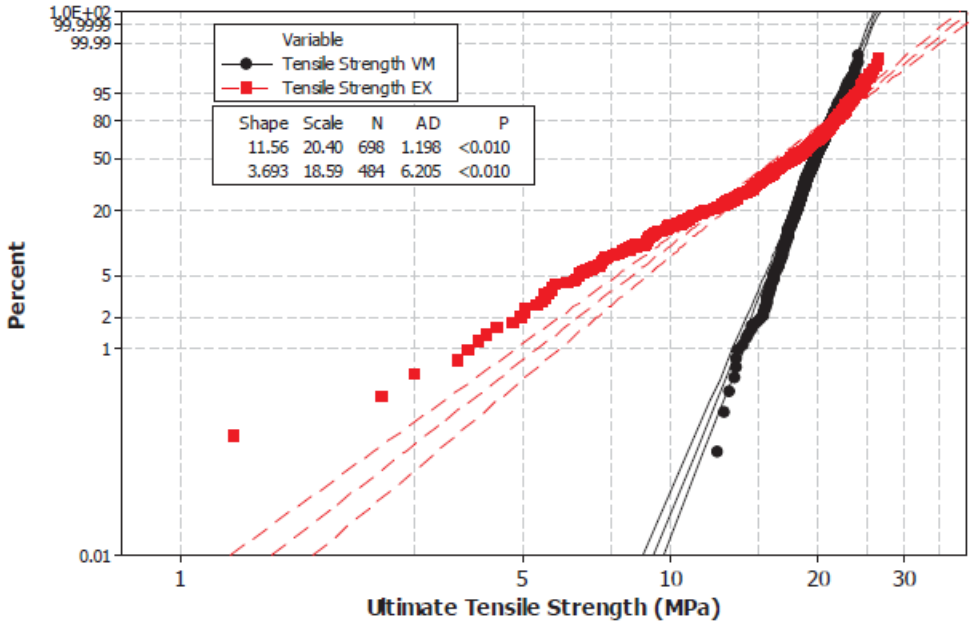


Figure 4-2. Weibull plot of the VM and EX graphite grade UTS measurements [25]

The flexural strength of the graphite grades are similar to the UTS. The VM graphite varies less, from 33 MPa to 20 MPa, while the EX graphite varies from 38 MPa to 13 MPa excluding outliers. A Weibull plot of this variation is shown in Figure 4-3. Similar to UTS and density, these results vary through the graphite billet, with the EX graphite decreasing in flexural strength from the top to the bottom of the billet. The VM graphite displayed a more homogenous distribution. The largest variance reported through analysis of variance (ANOVA) was 30.2%, and a minimum of 9%, in the EX graphite, near the bottom of the billet. The ANOVA analysis confirmed the trend of decreasing mean values for flexural strength of the EX graphite. Again, each of the graphite grades exhibited flexural strength measurements below the minimum specified by the ASTM standards, 21 MPa [25, 26, 27].

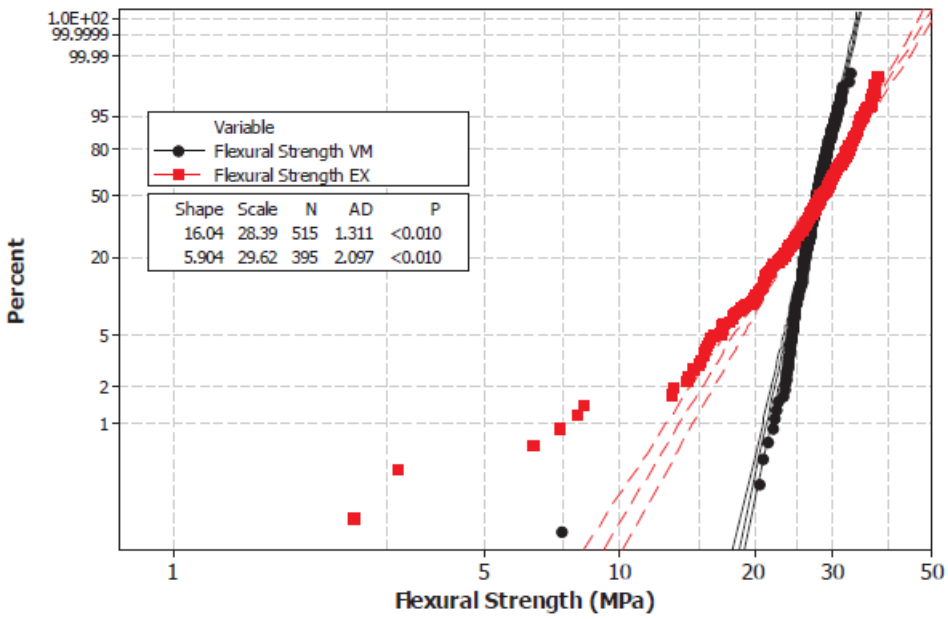


Figure 4-3. Weibull plot of the VM and EX graphite grade flexural strength measurements [25]

Compressive strength, while still mirroring the trends viewed in the other properties, provided more variation in both graphite grades as the compressive strength of graphite is considerably higher than its flexural or tensile strength. A Weibull plot of this variation is shown in Figure 4-4. The VM graphite varied from 92 MPa to 65 MPa, while the EX graphite varied from 80 MPa to 32 MPa excluding outliers. Similar to the previous properties, the EX graphite shows a trend of decreasing strength from the top to the bottom of the billet. The VM graphite displayed a more homogenous distribution. An ANOVA analysis was provided for the compressive strength of the EX graphite, with a maximum variance of 22%, and a minimum of 12.1%. Both graphite grades exhibited compressive strength measurements below the minimum specified by the ASTM standards, 45 MPa [25, 26, 27].

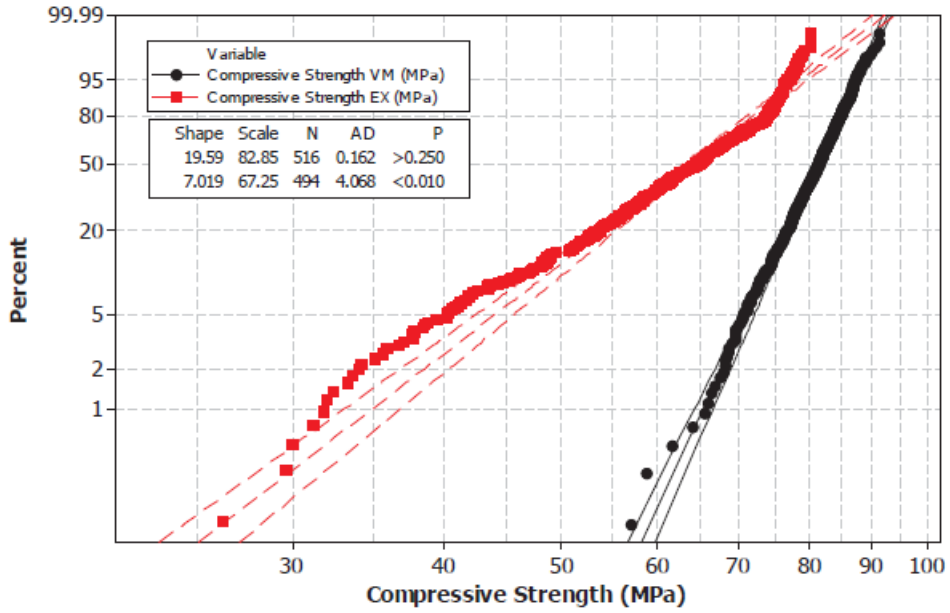


Figure 4-4. Weibull plot of the VM and EX compressive strength measurements [25]

With each of these mechanical properties (excluding density), the VM and EX billets were compared among billets of the same grade, of which there were two VM billets and three EX billets in this study. The variations among the billets were small, with consistent distribution between the grades. The VM properties were relatively consistent when compared to the EX grade [25].

The graphite grade properties were also compared in different orientations based on how the billet was processed. The VM billet was compared *with-grain* and *against-grain*, referring to the direction along the plane of hexagonal carbon atoms and the direction normal to the plane, along the stacked layers, respectively. For EX graphite the different orientations were respective to the extruding direction of the cylindrical billet. These included the following:

- Parallel to the extrusion direction, which is also the assumed with-grain direction
- Transverse the extrusion direction, across the centerline of the cylinder
- Radially, from the centerline to the edge of the billet

From these directions, UTS, flexural, and compressive strength have been measured. For UTS, the VM graphite has a much closer distribution, while the EX graphite has a much more inconsistent distribution, the smallest being the radial direction. For flexural strength, each direction of the VM graphite was almost identical, with high variation shown in the EX graphite, similar to the UTS data. For compressive strength, the higher values are consistent in the different directions for VM graphite, while the lower values start to spread more than what has been observed in the previous properties. Compressive strength of EX graphite shows a similar trend to what was observed with the other property distributions discussed [25].

From these observations, one particular trend was observed concerning the EX graphite grade. For all of the properties measured, a general trend displayed high strength and density in the upper layers of the EX graphite, with lower strength and density in the bottom center section of EX graphite. The region of lower density and strength exhibited larger flaws, and a larger distribution of flaw sizes, which in some cases were large enough to compromise physical property measurements [25].

These results are derived from only three extruded graphite billets and two vibration molded graphite billets. There will be more work to come concerning inconsistencies in the graphite billets, providing more data to understand the variations exhibited in the available nuclear graphite grades. There is still research to be performed concerning variations within components, lots, and graphite grades. This work is being performed through the BGC program.

The variation within the different material groupings of graphite manufacture are problematic for the advanced reactor vendors. Consistency between the graphite core components ordered cannot be assured through the knowledge that the grade is reported to be the same. A reactor vendor will need to test the graphite core components received in order to ensure the properties are acceptable and within an acceptable amount of variation of each other. There is currently no working definition of acceptable variation for graphite core components. The need to test graphite core components to determine properties and any variation will be design specific, and guidance in future version of the ASTM standards D7219-08 and D7301-08 as well as the ASME BPV Code Section III, Division 5 is warranted. Graphite core components ordered with long periods of time between them may have shifting properties due to shifts in raw materials, while still remaining the same grade. Billet variation could lead to components or test samples cut from the same billet having different properties or to large components contain variations throughout the volume. These will need to be considered and accounted for through NDE testing of the graphite or through better understanding, control, and documentation of manufacturing processes. Reduction in variation will put less responsibility on the reactor vendors to ensure the properties of their components are similar to each other and within their construction specifications. The BGC program through INL is working to provide closure to the gaps presented by these inconsistencies, but more work is needed to close these gaps. The BGC completed analysis of a second billet of PCEA graphite in 2016, with the first billet being analyzed in 2010. The process provides a wealth of information on the inconsistencies within graphite billets and, with further analysis, inconsistencies between billets. There is still a need for research on more billets, as well among billets of the same grade from different charges [35].

4.4 NDE Requirements

Both the ASME BPV Code Section III, Division 5 and the ASTM provide information discussing the nondestructive evaluation (NDE) at the various steps in the manufacture of graphite, from the coke to the graphite core components. The ASME is not as specific as the ASTM guidance. The ASME guidance for NDE, HHA-4212 refers only to graphite core components and discusses the need for written records of the procedures used as well as keeping the materials from being

contaminated, it does not refer to any NDE procedures or requirements. Article HHA-4230 requires at least visual inspection of post machined graphite components. In the ASME BPV Code for graphite, defects are defined as cracks, notches, inclusions, or excessive voidage [11]. The acceptance criteria for these flaws is determined by the designer in the construction specifications, outside of this there is no set definition for the acceptance of flaws and defects. The ASTM is more encompassing in its methods of analysis for graphite core components. D8093-19, *Standard Guide for Nondestructive Evaluation of Nuclear Grade Graphite*, discusses the use of NDE technologies with graphite. The guide only provides information on the techniques and does not provide any acceptance criteria for the user with respect to the NDE requirements of nuclear grade graphite.

Currently the main NDE techniques available for graphite, in addition to visual examination, include x-ray radiography, eddy current analysis, and ultrasonic inspections. These are the three techniques discussed in ASTM D8093-19 [36]. These techniques were also evaluated by INL during the Next Generation Nuclear Plant program [37]. Each of these techniques has its own weaknesses and strengths as follows:

- X-ray radiography and diffraction use the principle of x-ray beam attenuation to analyze the volume of the material of interest. This technique has been used for many materials in different fields of engineering and science for years, providing a wide breadth of information on the subject and its application. Graphite is an inhomogeneous material made of a single element. Due to this, the main property detected by X-ray radiography is density, which allows measurement of porosity. The main drawback of the use of X-ray radiography with graphite is the need for calculations to ensure adequate characterization of the attenuated X-rays for complicated graphite components [36, 37]. ASTM E1742 Standard Practice for Radiographic Examination establishes requirements for examination for metallic and nonmetallic materials [38].
- Eddy current analysis uses electrical and magnetic conductivity of materials to provide near surface information about flaws, porosity, and changes in grain structure or phase. This technology is established for use in most metals with varying electrical conductivity. Graphite has similar conductivity to some metals including Ti-6Al-4V titanium, Inconel 600, and SS 304. Graphite does, however, respond to magnetic frequencies differently from metals, leading to a need for large probe coils and lower frequencies compared to equipment used for high conductivity metals to analyze thicker sections of graphite. Grain size does produce noise in eddy current analysis, and most systems will only be able to detect flaws larger than the apparent grain size, which can exclude certain nuclear graphite grades from analysis due to large grain sizes. ASTM D7219-08 does not require graphite billets to have any surface finishing beyond wiping down after graphitization, which can complicate eddy current inspection due to non-ideal surface conditions [36]. The ASTM E2884-22 Standard Guide for Eddy Current Testing of Electrically Conducting Materials Using Conformable Sensor Arrays includes all conductive materials including metals, composite materials, carbon-carbon composites, and polymer matrices with carbon fibers [39].

- Ultrasonic inspections are limited by the range of acoustic velocities available in the graphite. Small, isolated flaws are harder to detect using ultrasonics. These can be on the same order as porosity already present, making differentiation between acceptable porosity and unacceptable small flaws difficult. Ultrasonics are viable for analyzing macro-cracks, large voids, and inclusions. The wave scattering could be used to detect variations in the microstructure for flaws, but more research will need to be done for this method [37]. Currently, no ultrasonic or acoustic inspection standard exists for general use, or specific to graphite. There are many acoustic emission testing standards including standards different metal structures, concrete, pressure vessels and storage tanks, and structural health [40].

The techniques discussed above are the most widely used NDE methods for graphite examination. ASTM D8093-19, the Standard Guide for Nondestructive Evaluation of Nuclear Grade Graphite only discusses the use of these techniques with graphite analysis and does not provide minimum requirements or further guidance than what DOE and independent research has already shown. ASTM E1742 does define minimum requirements for radiographic examination. However, no direct mention of graphite or variations in the technique for graphite are mentioned. ASTM E2884 is similarly non-specific for graphite materials. There does not currently exist a standard for ultrasonic testing that establishes requirements for graphite. More research will be needed in order to generate standards for graphite for these NDE techniques or update current standards that exist to include any necessary variations in the technique for graphite analysis. This is the main gap in knowledge for NDE examination of graphite billets and components.

Ultimately, a performance demonstration program may be necessary to provide confidence that NDE techniques are sufficient to detect flaws of engineering relevance in graphite components. Although there will likely be analogs to similar programs for metal components (e.g., EPRI's Performance Demonstration Initiative), it should be noted that graphite components are generally not pressure boundary (and therefore not radiation boundary) components and, therefore, are likely to require a lower level of ensured performance than components for which other NDE techniques have been developed (e.g., pressure vessels, piping, and other pressure boundary components).

4.5 Cleanliness

For metal components, cleanliness is well defined. The ASME NQA-1, Part II, Subpart 2.1 provides criteria and methods for maintaining the surface cleanliness of metals [41]. This document applies to various metals used in nuclear reactor systems and has different grades of cleanliness depending on the metal component's purpose. Within these grades in some cases are variations for specific metals that may be cleaned in various ways in order to ensure either appropriate surface cleanliness or lack of potential corrosion. This document discusses the cleanliness of metals from manufacturing, to storage, to installation and provides details on how the metal should be handled and kept clean at each stage. This guide only defines cleanliness for metal components in nuclear reactor systems. For graphite, there is currently no guidance analogous to Subpart 2.1. Currently in the ASME Code, graphite surface cleanliness is

not addressed in either HAB or HHA [11]. In the ASTM standards referring to the manufacture of graphite, ASTM D7219-08, the only requirement for cleanliness is that the surface is brushed clean after graphitization furnace runs [26, 27].

Graphite is a brittle, porous material, and will need to be handled differently than traditional metal components for surface cleaning. Abrasive cleaning can strip away graphite from the surface and could cause or exacerbate existing cracks, damaging the billet or component. NQA-1 Subpart 2.1 Section 304 lists different grades and types of cleaning solutions for metal components [41]. Graphite components and billets will need similar descriptions for their surface cleanliness. However, it is anticipated that most graphites will be able to use the same cleaning agents, but consideration and research on how fluid cleaning agents interact with graphite porosity will be necessary. Graphite is chemically inert, such that restrictions on cleaning chemicals are likely to be fewer than those for metals. For example, chemicals that evaporate without leaving a solid residue are likely to be acceptable. Sectioning of graphite does not generally use cutting fluid, so this would not be expected to be an issue in the manufacture of nuclear components [42]. Another consideration necessary in the production of standards for nuclear grade graphite will be the impact that potential cleaning chemicals will have on the coolant chemistry if not removed properly. Effective cleaning of graphite surfaces will also involve the removal of excess cleaning agents. However, this is not discussed in any ASME code or ASTM specification. Graphite has the potential to oxidize readily at elevated temperatures, and diffusion of surface contaminants into the porous graphite could lead to undesired effects such as internal stress from heat and irradiation.

Currently there are no ASME codes or ASTM specifications for graphite surface cleanliness at any stage of manufacture. Graphite can be affected by surface contaminants similar to metals, and there need to be specifications and requirements to keep graphite components clean. NQA-1 Subpart 2.1 describes classes of cleanliness for metal components, each of which is broken into subsections for the type of metal being cleaned. A similar set of guidelines will be necessary for graphite core components in order to ensure reactor vendors can properly clean their components upon receiving them and confirm through appropriate records that the graphite material was properly cleaned and protected from contamination through the manufacturing process.

With respect to cleanliness, the following gaps have been identified:

- There is uncertainty in the effects of surface contamination on graphite core components in irradiated environments as well as coolant environments at the relevant temperatures.
- There is uncertainty in the effectiveness of different cleaning strategies on graphite billets and core components.
- The relationship between surface cleanliness and the different nuclear graphites available is not known. Different forming methods as well as the different microstructures of nuclear graphites will need to be researched with respect to their effect on surface cleanliness.

Closure of these gaps, and the creation of guidelines based on the findings, will give reactor vendors methods to appropriately clean their graphite core components. Guidelines on the records of cleanliness for graphite material will also ensure the reactor vendors that any parts received are adequately handled and processed to not introduce contaminants to the surface.

4.6 Structural

Designers of graphite components need to understand the structural aspects of graphite and graphite components. For metal components, the structural aspects and the properties connected to them have been studied extensively. The research and operating experience for metals led to ASME code specification and ASTM standards for the development and design as well as testing standards for metal components. Graphite has some ASME and ASTM guidance and standards for structural attributes, but the lack of research and operating experience has resulted in fewer ASME code specifications and ASTM standards for graphite compared to metals. The structural attributes discussed in this section do not describe particular microstructure impacts on the attributes. The microstructure issues of graphite are discussed in Section 4.10. These structural attributes (and their respective ASME or ASTM considerations where available) are as follows:

- Compressive strength, tensile strength, and flexural strength are not mentioned in the ASME BPV Code Section III, Division 5 for the design of graphite components as any sort of requirements. The compressive strength and tensile strength of graphite is mentioned in relation to weight loss from oxidation. These attributes of graphite are specified in the ASTM Standards, D7301-08 and D7219-08. Each of these standards specifies compressive, tensile, and flexural strength values for nuclear grade graphite [26, 27]. ASTM standards for compressive, tensile, and flexural strength tests exist as C695, C749, and C651 respectively. These methods are all destructive testing methods. As discussed in Section 4.3, the consistency of components cut from billets can be questionable, and destructive testing of proof components received with the real components may not provide confidence without further research on the consistency of graphite core components and the billets from which they are cut [6].
- Creep strength of graphite is a subject of current research. An irradiation induced creep coefficient is required in the materials data sheet of the ASME BPV Code, but no testing method is listed in the ASTM Standard C781 [11, 6]. Graphite creep strength in irradiated components is a current subject of study in the Advanced Reactor Technologies (ART) program through the Advanced Graphite Creep (AGC) program. The AGC seeks to understand the effects of irradiation on different grades of nuclear graphite in order to quantify the irradiation creep data for these grades [43]. Creep is to be considered in the analysis of irradiation effects according to the ASME BPV Code, but no testing is required by the manufacturer or upon receiving the materials.
- Fracture toughness in graphite describes the resistance to crack propagation. As a brittle material, graphite components can easily crack, and fracture toughness of graphite core components being received will need to be confirmed in order to determine if they are

defective. ASTM standard D7779 defines the process to determine the fracture toughness of graphite core components. D7779 involves adding forming a notch on the graphite component being tested and forcing crack propagation from that notch [6]. Performing destructive evaluations with graphite components can bring into questions consistency in the graphite components cut from the same billet as discussed in Section 4.3. These inconsistencies will need to be understood in order to purchasers to accurately test their materials and be able to trust their results.

To correctly define the attributes listed above, graphite material properties must be known. The material properties are determined through research of nuclear graphite grades, as well as test methods developed by the ASTM. The properties important for graphite core components are as follows:

- CTE is a very important property for graphite use in nuclear reactors. Low CTE allows for graphite components to retain their shape while at the extreme temperatures at which advanced reactors are intended to operate. The CTE of graphite can vary with direction, based on the microstructure. Tests to determine how isotropic a graphite component is use CTE from different directions of the component to determine a ratio. The ASTM standards for nuclear graphite grades require a CTE isotropy ratio of 1.15 or less for all microstructure types of nuclear graphite [26, 27]. This ratio is determined using the ASTM practice C781 [6].
- Poisson's ratio is another important property of isotropic graphite. Understanding how graphite will change dimensionally under stress is important for designers to generate tolerances. Isotropic graphite will change predictably, and Poisson's ratio can be determined through the ASTM test methods E132 and C747 [6]. Poisson's ratio is required in the ASME BPV material sheet in table HHA-II-2000-1. There is an ASTM testing practice to find Poisson's ratio in ASTM C781, but there is no requirement for determining Poisson's ratio upon receiving the component [11, 6].
- The elastic modulus of graphite is required in both the ASME BPV Code Section III, Division 5 as well as the ASTM standards for nuclear graphite, D7301-08 and D7219-08. The ASME Code requires designers to report the modulus of elasticity in the material data sheet for qualification, while the ASTM standards restrict the elastic modulus of nuclear graphite based on its class [26, 27]. The ASME also requires the elastic modulus be used for stress analysis, and consideration for internal stress due to irradiation and changes in properties due to irradiation be considered for the elastic modulus of graphite core components in subpart HHA-3100 [11]. The ASTM test methods can be carried out by manufacturers to ensure elastic modulus values for the designer and the material data sheet, as well as upon receipt inspection of the graphite core component by the vendor fabricator.

Currently the ASME BPV Code and the ASTM standards concerning nuclear graphite grades provide guidance and specifications for structural attributes for graphite, but do not always provide methods of measuring the necessary properties to define the structural attributes effectively. The ASME BPV Code does also require different graphite structural properties for

completion of the material data sheet, but there are not standards available for all of the tests needed to provide those physical properties. Additionally, there is concern with the consistency discussed in Section 4.3 that may cause some parts of a billet to pass inspections, and other parts of the same billet to fail inspections. This can result in graphite core components that have varied properties from one another, of varying properties across the volume, rendering sampling for testing unreliable as the property variance is not completely characterized. Currently, research at ORNL is concerned with both quantifying the creep response of graphite in irradiation environments, as well as the baseline characteristics of graphite. These baseline characteristics include the properties needed for determining the structural attributes of graphite (e.g., elastic modulus, Poisson's ratio, and CTE). The baseline graphite characterization (BGC) program is intended to provide property information on the various nuclear graphite grades available from different manufacturers [44]. ASTM tests may be available for these properties, but each graphite grade will behave differently and provide different results. The closure of the gaps in the consistency between the grades of graphite discussed in Section 4.3 will begin to close the gaps needed to effectively ensure the structural attributes and properties of graphite core components being delivered to reactor vendors.

For metals, structural attributes and properties can easily be determined through tests designed to be performed by manufacturers to ensure specifications, and by the material purchaser after receiving materials. Proof components can be generated alongside smaller components for destructive evaluations representative of the original component. Sufficient material samples from a given heat can provide qualification that the material and components are built to ASME code and ASTM standards. For graphite, the ASME code and ASTM standards are less specific. This does not provide reactor vendors enough information on what is needed after receiving a component to confirm the material properties of their parts. Additionally, issues with consistency within billets can lead to proof components not being representative of the billet, or even components cut out from the same region of the billet. More guidance, which will need to be based on research and experimentation of nuclear graphite grade materials, will allow for better control and confidence in the tests performed by reactor vendors and material manufacturers to determine the structural attributes and properties of graphite core components. Further research for this purpose will increase the confidence in determining the acceptability of graphite core components.

4.7 Surface Preparation

The surface condition of graphite, similar to metals, can have an effect on different properties of the graphite core components. Not only the properties of graphite but the potential lifespan of components can be decreased due to improper surface preparation before installation in the reactor. Graphite porosity is a major factor in the material properties of graphite components, and in some cases, improper surface conditions could affect those properties through increased oxygen diffusion to pores, molten salt intrusion, or fission product retention in pores. The surface of graphite core components will determine how the graphite core components will respond to different environments. Reactor vendors will need a method of testing and

examining the surface of graphite core components to ensure the proper manufacturing specifications were followed for the part. Some of the different phenomena that are affected by the surface condition of graphite are as follows:

- The wear and abrasion of graphite while in service is an issue that is being studied by ORNL currently. Graphite is a very brittle material that can easily be worn away through constant movement, and any reactor design that may incorporate movement between graphite on graphite, graphite on metal, or graphite interacting with coolant will need to consider wear and abrasion and how the surface condition affects these factors [32, 45]. As graphite is worn away, tolerances between individual components will be affected, and, potentially, the structural integrity of some components could be compromised.
- Molten salt intrusion is a concern for molten salt reactors which will use graphite components for moderation, reflection, and structural components for coolant channels and to potentially hold fuel. Molten salt is able to infiltrate the pores of graphite, and in the case of coarser grained graphite discussed in Section 4.10, the intrusion could penetrate deep into the graphite components [32]. This can cause internal stress, potential oxidation, and chemical interaction between the salt and graphite, as well as retention of fission products from fuel bearing salts. ORNL is currently studying the effect of environment on the salt wetting on nuclear graphite grades and will eventually explore how surface conditions affect the molten salt intrusion [45].
- NDE of graphite components, as discussed in Section 4.4, can be affected by the surface condition of graphite. Eddy current examination in particular requires a smooth surface for accurate, noise free, assessments of the near surface condition of graphite billets or components [36]. Other NDE examination methods may benefit from a reduction of noise with smoother surfaces, and work is needed to determine whether surface condition affects the popular NDE methods enough to warrant code requirements.
- Some designers have explored the use of coatings on graphite materials to protect integral components from potential salt intrusion, oxidation, or chemical reactions. These coatings are intended to cover the surface, as well as fill surface level pores in order to halt diffusion [46, 47]. Surface conditions of graphite are an integral factor in coating applications, and substrate condition will determine the lifetime of coatings and how they degrade over time.

Table 4-1 describes the ASME/ASTM specifications for graphite surface conditions with respect to the issues discussed above. Table 4-1 also describes the current research level of these issues. The research level is defined as:

- 1: Little to no research currently being conducted on the issue with respect to surface condition.
- 2: Some research is being performed, designers are interested in results regarding the issue with respect to surface condition.
- 3: There is a wealth of research available, databases are being formed using the information to inform designers on how to approach the issue with respect to surface condition.

Table 4-1. Surface condition issue research levels

Surface Condition Related Issues	ASME/ASTM Requirements or Specifications	Current Research Level
Wear and Abrasion	None	2
Molten Salt Intrusion	None	2
NDE techniques	None	1
Coatings	None	2

Currently the ASME BPV Code Section III, Division 5 only addresses the surface of graphite billets and components with respect to flaws from visual examination. The ASTM Standards, ASTM D7219-08, the Standard Specification for isotropic and near-isotropic nuclear graphites and ASTM D7301-08, Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose, only specify that graphite billets be brushed off after the graphitization furnace run to remove any loose material. Upon receiving graphite core components, surface preparation before installation is currently not discussed by the ASME BPVC or ASTM specifications. This lack of guidance and understanding of the surface condition of graphite and its effects means that reactor vendors will not have enough information to correctly assess the acceptability of graphite core components. Further research will provide more understanding on the effect of surface condition of graphite components on the material properties of graphite, and this in turn will drive the need for ASME and ASTM modification necessary for informed reactor designs and produce inspections upon receiving a completed graphite core component. These surface inspection methods will help ensure that the graphite components being received are suitable for installation within the reactor core.

4.8 Destructive Analysis

Destructive analysis allows for an understanding of a materials response during failure, as well as the response in the regimes leading to the failure of a material. Destructive analysis of metal components can be performed at different points in the manufacturing process. For example, the specifications for Alloy 690 steam generator tube destructive analysis allows for tube ends cut from tubing early in the manufacturing process [33]. The homogeneity and history of research on metal components allows for the destructive analysis to consist of samples such as tube ends or possibly just samples cut from the same billet as other kinds of metal components. This allows the reactor vendor to have representative analyses showing that their components have the properties specified. The potential heterogeneity inherent in graphite billets raises concerns with this approach. Graphite billets have varying properties throughout their volume, and cutting plans for graphite components require careful consideration in order to introduce as little heterogeneity into the graphite core component as possible. This complicates destructive analysis of graphite core components as proof components may not be representative of the part being installed in the reactor.

Destructive analysis for graphite core components is not considered by the ASME BPV Code Section III, Division 5. The only analysis considered is the NDE analysis of graphite core components during manufacturing and before installation. The ASTM standards, D7219-08, and

D7301-08 have requirements for compressive, tensile, and flexural strength of nuclear graphite. These tests are performed with a minimum sampling frequency of billets from the production lots described in Section 11 of ASTM D7219-08 and Section 10 of ASTM 7301-08. The requirements are in place along with recommendations that all graphite billets are nondestructively tested as well [26, 27].

The destructive testing required by the ASTM standards for nuclear graphite grades aid in completing the material data sheet required by the ASME BPV Code Section III, Division 5 Mandatory appendix HHA-II with the mechanical properties of the graphite billets. Inconsistencies within the production lots of graphite, discussed in Section 4.3, can cause uncertainty in the individual billets being produced, as well as the graphite core components cut from the billets. The lack of requirements for destructive testing of graphite core components leaves uncertainty in the failure limits, as well as failure modes, of graphite core components. The issue facing the lack of destructive testing requirements is the lack of information available to determine how representative graphite core components can be fabricated, as well as how billet to billet variability can be accounted for when performing destructive testing. Without appropriate requirements for destructive testing of graphite core components, reactor vendors are unable to ensure that the received parts are acceptable for installation, outside of the documentation provided on the billet destructive testing required by the ASTM standards. Billet testing alone will not provide enough confidence in the graphite core components being produced for reactor vendors. Closing gaps related to consistency in graphite material groups, discussed in Section 4.3, will allow for greater confidence in the destructive testing already required, as well as a better understanding of the destructive testing needed for acceptance of graphite core components being received.

4.9 Chemical

Graphite purity is of concern for advanced reactor manufacturers and designers. Impurities in graphite can cause issues both in changing the mechanical properties through large inclusions that affect the crystal structure of graphite and potential activation of certain elements due to the irradiation environment. The ASTM specifications for graphite, ASTM D7219-08 and ASTM D7301-08, each contain requirements for the chemical properties of nuclear graphites in Section 6 and Section 5 of the standards respectively [26, 27]. The ASME BPV Code also provides a list of controlled substances through the table HHA-4222-1, although these substances are in reference to machining facilities and do not discuss anything apart from a list of prohibited substances and a list of controlled substances [11]. Graphite can be affected in a few different ways at increased temperatures in irradiated environments, and the ASTM and ASME requirements serve to ensure the operation of the graphite core components as designed once received from the manufacturer.

Both ASTM specifications, D7219-08 and D7301-08, provide limitations set on boron within the graphite components Section 6 and Section 5 of the standards respectively [26, 27]. Boron is a neutron absorber and can be transmuted into lithium and helium atoms, putting stress on the graphite through helium generation. Helium generation can cause helium to diffuse to the pores of

graphite and due to the temperatures of advanced reactors, will create internal stresses, potentially causing failure. The ASTM specifications have maximum limitations for boron content in nuclear graphite material because of this. The maximum limitation is different for the different classes of nuclear graphite. These classes also contain a maximum limitation on the ash content available in material to further reduce chemical contamination. The ASTM specifications also provide a minimum list of elements to be measured for equivalent boron concentration (EBC) calculations in Section 6 and Section 5 of D7219-08 and D7301-08, respectively, which include boron, cadmium, dysprosium, europium, gadolinium, and samarium [26, 27]. Outside of this, chemical impurities are allowed by the ASTM specifications to be agreed upon between the supplier and the purchaser. As long as the ASTM requirements, and the ASME prohibited substances are followed, there are no other requirements. The ASTM specifications do list impurity categories for graphite in Table X1.1 of the two nuclear graphite standards, including neutron absorbing impurities, oxidation promoting catalysts, activation relevant impurities, metallic corrosion relevant impurities, and fissile/fissionable elements, as well as the elements within these categories.

The boron equivalent calculation is detailed in the ASTM practice C1233, determining equivalent boron content of nuclear materials, and can be used to determine the contents of multiple species within manufactured graphite, and ASTM C781 provides a method of determining boron content within nuclear grade graphite through calcination and inductively coupled plasma optical emission spectroscopy (ICP-OES) [6, 48]. The boron equivalent values found using ASTM C1233 can be used to compare with the minimum requirements provided by D7219-08 and D7301-08 in order to confirm chemical purity of the nuclear graphite material. The boron results from C1233 are reported by manufacturers, as they are required by ASTM D7219-08 and D7301-08 to perform EBC tests during the sampling described in Section 11 [26, 27]. The methods of boron calculation through ICP-OES can be performed as well to determine the boron found in the nuclear graphite, which could be performed by the manufacturer or the reactor vendor upon receiving the graphite material.

From the available specifications, it can be concluded that graphite is controlled in its chemical composition and purity. The ASTM specifications, ASTM D7219-08 and ASTM D7301-08, provide guidance and limitations on problematic elements that can be found within graphite components through processing. The ASME BPV Code provides in Table HHA-4222-1 a list of prohibited elements concerning graphite machining in order to maintain graphite purity. One part of graphite not considered for chemical purity is the petroleum coke and coal tar pitch being used as raw materials. The raw materials used for graphite production do have requirements for CTE and density, but do not have any chemical purity requirements. Any impurities found in the raw materials can be carried forward through the manufacturing process to the graphite core component. The ASTM sampling requirements for nuclear graphite grades are designed to identify these potential impurities and prevent them from affecting the final component, but additional specifications on the purity of the raw materials would help distribute the responsibility of chemical purity throughout the ASTM standards. This will help provide confidence in the acceptability of graphite core components being produced by graphite manufacturers.

4.10 Microstructure

The microstructure of graphite is an integral part of the material and its properties. Unlike some metals, such as steel, which can have multiple crystal structures associated with it, graphite as a material refers to a particular structure, sheets of hexagonally connected carbon atoms called graphene, stacked together with Van Der Waals forces holding the graphene sheets together. The microstructure of graphite is determined by not only the size of the molecular structures, but the pitch binder, any filler material used by the manufacturer, and the inherent porosity within the graphite. The driving factors determining graphite properties are the grain sizes and the amount of porosity in graphite. Manufactured graphite, including the nuclear graphite grades, have about 20% porosity within their microstructure. The porosity differs in pore size between the nuclear graphite grades based on the grain size of the grade. The microstructure and porosity of graphite is what defines the irradiation response of graphite as well. Pore closure and expansion are the driving factors affecting the changes in graphite properties when irradiated [32]. These factors are what drives the classes for nuclear graphite grades in terms of their microstructures.

The ASME BPV Code Section III, Division 5 does not directly address the microstructure of graphite. The code does address irradiation and oxidation of graphite in subsection HH, subpart A, which is affected by pore and grain size. These factors will determine how graphite core components respond to the irradiation environment, as well as what may happen if acute oxidation were to occur in the reactor [11]. The ASME BPV Code does refer to the ASTM standards for nuclear graphite, which do discuss microstructure of graphite for nuclear grades. Most microstructure variation comes from individual nuclear graphite grades produced by manufacturers. Each supplier grade will have differences in pore and grain size, which will affect the resulting properties differently. The ASTM standards for nuclear graphite, ASTM D7219-08 and ASTM D7301-08, each provide a table defining the nomenclature of nuclear graphite in relation to the grain size. These range from microfine grained graphite with a grain size of < 2 μm, to medium grained graphite with grain sizes less than 4 mm. There is a specification within these ASTM standards for the maximum particle size of the coke filler, which determines the grain size of the graphite after graphitization [26, 27].

Because the microstructure of graphite is very dependent on processing, more specifications may be necessary in addition to what is already provided by the ASTM standards. Processing methods may affect the microstructure of graphite through the grain sizes, pore sizes, or porosity of graphite materials. More research will need to be done on the graphitization process and the filler, binder, and impregnation of graphite in order to understand how these processes affect the final microstructure. For molten salt reactors, grain structure has been shown to affect the intrusion of molten salt into the graphite components. Larger the grains, and the resulting larger pores, will allow for more molten salt intrusion, in some cases well into the graphite component. This can cause internal stress as well as degradation of the graphite, and in the case of fuel bearing salts, potential hot spots within the graphite core components that could result in variations of irradiation levels not accounted for in the component design. A greater understanding of the effects of molten salt intrusion and the driving factors for molten salt intrusion is underway through ORNL molten salt research programs, but more will be

needed for designers to make informed decisions [32, 49]. Because the microstructure of graphite is heavily dependent on the processing methods and raw materials, the gaps in consistency discussed in Section 4.3 significantly affect the knowledge gaps regarding the microstructure of graphite. As greater understanding and more specific standards are created for the manufacture of graphite, the microstructure of nuclear graphite grades will start to become more controlled and uniform across individual grades.

Currently, although the ASTM has specifications for grain size and classifications for nuclear graphite based on grain size, there is no listing of a testing method for the grain size of graphite. This will leave the manufacturer and the graphite vendor on their own to come up with an acceptable method of grain size examination. The ASME requires a grain size to be reported in the material data sheet according to the maximum filler particle grain size from the mix calculations [11]. These requirements are not enough to ensure the reactor vendors that the parts they are requesting and receiving have the appropriate grain size. ASTM test standards for grain size measurements will need to be put in place to ensure vendors that the parts they are receiving are being made to the specifications they require. Additionally, it will allow for a standardized method to test the parts received to ensure the grain sizes are acceptable for use in the reactor core.

4.11 Defect Disposition

The ASME BPV Code Section III Division 5 provides guidance on defect disposition for graphite core components as well as graphite billets after graphitization. The material used by the manufacturer to develop graphite core components are to be examined in accordance with the material specifications according to HHA-2510. These material specifications are specifically referring to the ASTM standards for graphite, D7219-08 and D7301-08. The ASME BPV Code allows for repair in the cases of billets failing purification or having cracked or damaged sections. Repurification or re-graphitization is allowed for graphite failing purity or electrical resistivity defined in the material specifications, with documentation of the repair. Unaffected sections of damaged billets are allowed to be used for graphite core components as long as the other requirements of the material specifications are met. Receipt inspection is required in HHA-4223 in order to ensure that any material used is of the correct grade, particular to the graphite vendor, and that the material meets the requirements of the rest of the HHA-2000 requirements for graphite. This receipt inspection is done before machining of the graphite into the graphite core component [11].

Section HHA-4233 of the ASME BPV Code Section III, Division 5 describes the examination for material defects and damage with respect to graphite core components. Each component is to be examined, at least visually, for defects or damage caused by machining or handling. These include cracks/flaws or excessive voidage/porosity [11]. The brittle nature of graphite leads to cracks and chips easily formed during handling. The acceptance criteria is defined by the designer in the construction specifications for the graphite vendors. Because of the complexity of analysis for mechanical properties of graphite, a probabilistic failure model is allowed in HHA-3300 to handle the variability of graphite grades. The probabilistic failure model uses structural reliability classes (SRC) to differentiate the importance of the graphite core component's reliability in the reactor core. Different acceptance criteria for SRC-1, SRC-2, and

SRC-3, as well as different nuclear graphite grades if included, apply to the different graphite core components based on their role in the reactor core. Surface visible defects are subject to the designer's approval considering factors such as crack length, area of damage on the component, distance to other defects, etc. [11]. The limits defined by the designer will determine whether components are rejected or accepted for repair. The repair of surface cracks does have a hard limit in HHA-4233.4, which allows dressing to a depth not exceeding 0.079 inches; if the crack is still visible the component is rejected [11].

These requirements and specifications do provide the reactor vendors ordering graphite core components a degree of certainty that the part is able to be correctly dispositioned and either repaired or rejected as a result. The designer is required to determine whether any cracks visible on the graphite material purchased constitute a violation of the SRC for that particular component. A standardized method of defect disposition is not yet available for graphite core components and can limit the reactor vendors ability to determine if cracks in received graphite products are acceptable. The ASTM standards, D7219-08 and D7301-08, recommend screening all graphite billets for internal defects in Section 12, but determination of the specifics is left up to the designer and the manufacturer [26, 27]. Additionally, as discussed in Section 4.4, graphite NDE will need more research and demonstration before being used to make decisions on the viability of graphite billets and core components being produced. Defect disposition upon receiving graphite core components will also need more consideration by the ASME BPV Code as well as the ASTM standards outside of just visual inspection. Standardization of violating defects for individual nuclear graphite grades may be necessary in order to have a collective understanding of what constitutes a violating defect upon receiving a cracked component. Further research from programs such as the baseline graphite characterization program will provide this information through testing different billets of graphite grades, but this will take time before a complete database is usable as a reference for ASME and ASTM guidelines [44]. Once completed, this database can be used by reactor vendors to determine if a component with a defect is acceptable. This database could also give vendors a better idea of what examinations to request from graphite manufacturers during the manufacturing process to ensure component acceptability. Note that this discussion of pre-service defects is distinct from the consideration of the definition of component failure related to in service degradation discussed in Section 6.3.2.

4.12 Packing, Shipping, and Storage

The packaging, shipping, and storage of graphite core components requires care and attention. Graphite core components are required to be kept defect free and clean, but graphite is delicate and will require further consideration. Graphite is brittle and prone to cracking and chipping. These cracks and chips can affect the overall material properties of the part. Additionally, graphite can be contaminated and will need to be carefully wrapped and isolated in order to keep the graphite core component clean. Graphite core components, as well as graphite billets being handled between operations, will need special care to ensure that no defects, such as cracks, are introduced through transportation. Graphite, if improperly stored,

can become corroded from dampness, uncontrolled climates, and exposure to other materials that may be in storage or being manufactured or machined.

The ASME BPV Code does consider the packaging of graphite core components in HHA-4250 of Section III, Division 5. The code states that the procedures will be agreed upon by the designers during the specification of the construction of the graphite core component. Certain minimum considerations are required, such as large graphite core components will be packaged to prevent damage at all stages of use, machined components will be wrapped with an impermeable barrier to prevent contamination, and small components may be packaged together appropriately to prevent contamination and damage. Stacking of large components is allowed up to a safe level in order to prevent overloading of the bottom components. HHA-2600 also requires that packaging should prevent mechanical damage. Each package will require data reports and billet identification in order to maintain traceability of the graphite core components. The methods used to achieve these minimum requirements falls upon the designer and the graphite vendor [11].

Graphite has been transported for use in ORNL and INL tests in the last two decades multiple times. Nuclear graphite grades from multiple organizations have been transported to these labs for multiple projects including the ART programs, the molten salt intrusion experiments, and the next generation nuclear plant work [44, 50, 51]. The packaging, shipping, and storage methods for these graphites has been successful, as they did not report serious issues with the transportation of the test graphite. Storage of graphite has not been an issue as some experiments have been in progress over many years with billets of graphite acquired during the start of the experiment. The industry knowledge on the packaging, shipping, and storage of graphite seems sufficient for support of advanced reactor deployment.

5 AGING AND DEGRADATION

The aging and degradation of graphite can be considered using the following three general processes:

- Irradiation
- Oxidation
- Reaction with Molten Salts

These are discussed in the sections below. Fatigue of graphite is discussed in Section 5.4.

5.1 Irradiation Response

The irradiation response of nuclear graphite subjected to fast-neutron bombardment within graphite-moderated reactors is a fundamental atomic response and, on a macroscopic scale, processing dependent. Graphite has remained a candidate material for use as a moderator and structural material for current and future reactor designs due to its baseline properties. Specifically, suitable moderating materials should display the following attributes:

- It should efficiently thermalize fast neutrons with elastic atomic collisions.
- It does not capture or react with fast neutrons.
- It is compatible with other core materials.
- It is satisfying the structural design constraints of the reactor.

Synthetic polycrystalline nuclear graphites possess unique physical, thermal, and mechanical properties to tolerate the extreme environments of high temperature reactor (HTR) designs. One limiting factor for component lifetime for the future graphite moderated advanced reactor concepts will be irradiation-induced degradation of the graphite. This section addressed the following topics:

- Mechanism of radiation damage
- Resultant bulk property changes
- Irradiation creep

5.1.1 Radiation Damage in the Graphite Microstructure

The fundamental response of nuclear graphites to irradiation damage stems from the atomic displacements produced by fast neutron bombardment. On the atomic scale, all nuclear graphites have a hexagonal or rhombohedral crystal system characterized by the two space groups, $P6_{3/mmc,}$ and $R\bar{3}M$. Hexagonal graphite is composed of an ABAB stacking sequence, whereas rhombohedral has an ABCA sequence (Figure 5-1). The energy required to displace a carbon atom from its lattice position is characterized by the threshold for atomic displacement (E_d). The value of E_d ranges in literature from 12 eV to 60 eV [52, 53, 54, 55]. The variance in values is due to the

anisotropy of graphite's crystal structure. High values of E_d arise from incident irradiation perpendicular to the *c*-axis, whereas lower values correlate to incident irradiation parallel to the *c*-axis [53, 56]. The *c*-axis refers to the axis perpendicular to the basal planes, the flat planes of hexagonally ordered carbon atoms, as shown in Figure 5-3, while the *a*-axis refers to the axis parallel to the basal planes, also shown in Figure 5-3. However, considering synthetic polycrystalline nuclear graphite, an appropriate E_d is reported as 25 eV [53].

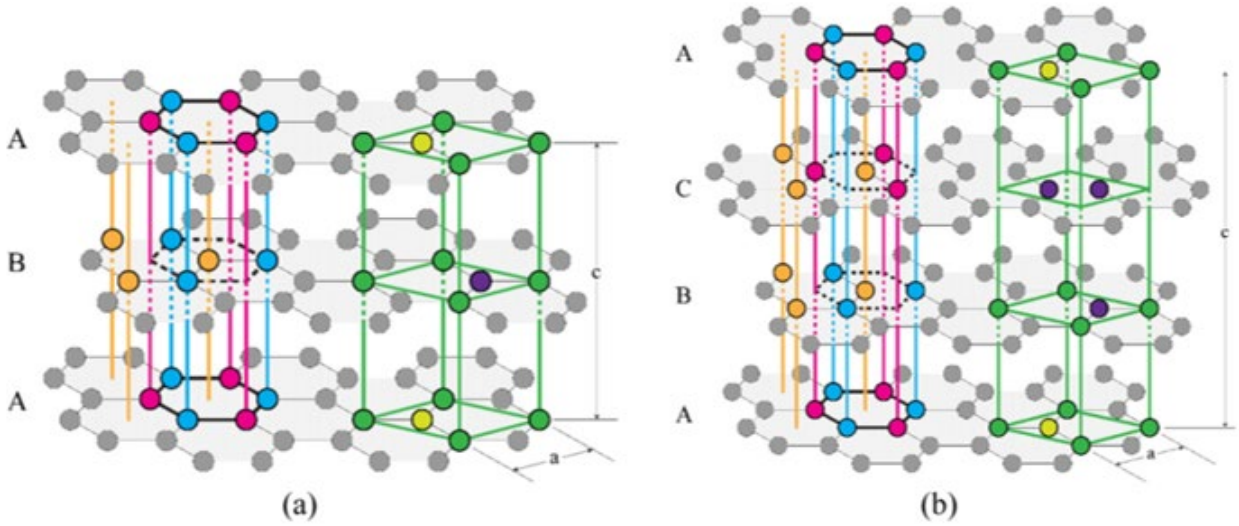


Figure 5-1. The (a) hexagonal and (b) rhombohedral crystal of graphite showing the ABAB and ABCABC stacking sequence of graphene planes in which the carbon atoms have 3-fold coordination [98]

Nuclear graphites have a complex microstructure composed of binder, filler particles, quinoline insoluble (QI) particles, chaotic arrangements of crystallites, and cracks and pores ranging from nanometers to millimeters [57, 58, 59]. Figure 5-2 shows transmission electron microscopy (TEM) images of nuclear graphite 2114. Figure 5-2(a) shows lenticular cracks which run parallel to the basal planes. These cracks are known as Mrozowski cracks and form during cooling from graphitization temperatures [30]. The cracking is due to the anisotropy of the coefficient of thermal expansions (CTE) and the weak van der Waals bonding between basal planes [30]. Figure 5-2(b) shows the binder phase with no long-range order to crystallites, amorphous phase, and turbostratic phases [58]. Figure 5-2(c) shows clusters of QI particles that form due to the presence of hydrocarbons before graphitization [58]. Figure 5-2(d) shows an example of other chaotic structures within the microstructure. All nuclear graphites, independent of grade, have the microstructural features shown in Figure 5-2; however, the size, distribution, and quantities of such features may vary [58, 59].

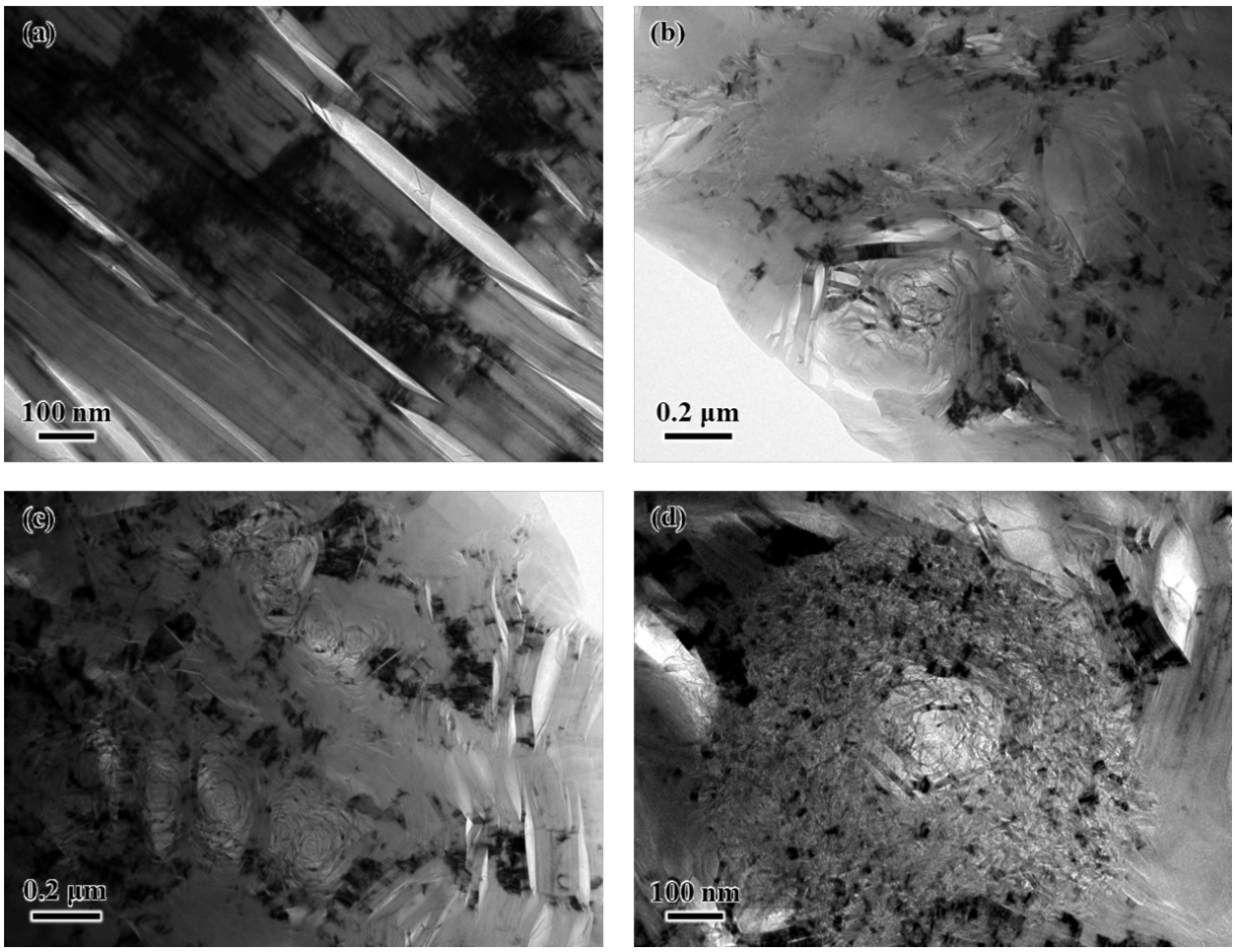


Figure 5-2. Bright-field TEM images of nuclear graphite 2114. (a) shows a filler particle with several Mrozowski cracks, (b) the binder phase, (c) a cluster of QI particles, and (d) a chaotic arrangement of crystallites

In general, on the atomic scale, displaced Carbon atoms will cause contraction along the *a/b*-axis, and expansion along the *c*-axis. This mechanism has been well documented experimentally in neutron-irradiation studies conducted on highly oriented pyrolytic graphite (HOPG) [60, 61, 62]. The formation of interstitial loops between basal planes is a historical model to depict the atomic mechanism governing *c*-axis expansion in graphite crystallites (Figure 5-3(a)) [64, 65, 66]. However, there is little to no experimental evidence of the formation of interstitial or vacancy loops in nuclear graphites. The current consensus is that interstitial loop formation is not the dominant defect mechanism [67, 68, 69, 70, 71, 72, 73, 74, 75, 76]. Alternative explanations exist for defect mechanisms that use theoretical models of basal plane defects. These theories of basal plane distortion arise from the presence of five- and seven-member rings [77, 78]. Perhaps most notable is the *buckle, ruck, and tuck* proposed by Heggie et al. and later confirmed experimentally by Johns et al. (Figure 5-3(b) and (c)) [68, 75].

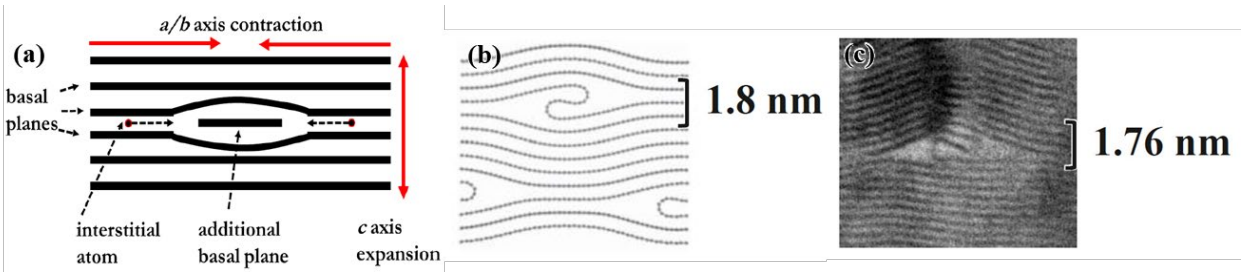


Figure 5-3. (a) Schematic representation of the historical model of defect evolution in graphite; (b) The theoretical model of the proposed *ruck and tuck* defect; (c) Experimental evidence of a *ruck and tuck* defect occurring in neutron-irradiated IG-110 [75]

The distortion of sp^2-bonded materials is known to occur in the presence of defect domains consisting of ordered arrangements of 5-7 ring members, in which the atomic arrangement of atoms departs from the typical *chicken wire* lattice structure [72]. Specific examples include the Stone-Wales or Dienes defect and the less common Haeckelite allotrope of carbon [79]. Basal plane defects are most often modeled two-dimensionally as graphene using density functional theory (DFT) and molecular dynamics (MD). Stable basal plane defects have been shown to cause strain with respect to pristine graphene and have been theorized to result in out-of-plane blistering [78]. MD studies show graphene ribbons of a critical size that contain five-member rings and temperatures between ~525-925°C; the edges will curl and meet, resulting in closure and the formation of open-ended hollow structures (i.e., nanotubes) [80].

With the rise of atomic resolution TEM, the characterization of irradiation-induced defects has improved dramatically relative to early studies conducted in the 1960s [64, 65]. However, neutron-irradiated specimens are scant, particularly when considering grades, doses, and irradiation temperatures of interest. Additionally, characterization often requires specialized radiological facilities. A suitable surrogate is in-situ electron irradiation, which is argued in the literature to be an acceptable approximation [52, 54, 74, 76, 81, 82, 83, 84, 85, 86, 87]. Relevant exposures (dpa) can be generated in minutes rather than years, and samples are not radioactive. Room temperature electron-irradiation TEM studies conducted by Koike [52] proposed a mechanism of fragmentation of basal planes to explain the extraordinary 300% expansion along the *c*-axis of a crystallite of HOPG. On the other hand, room-temperature electron-irradiation studies on graphitized carbon fibers showed homogenous swelling of the *c*-axis lattice spacing, thus, contradicting expansion due to fragmentation [87]. Yet, room temperature electron irradiation conducted on IG-110 by Karthik et al. proposed the formation of new basal planes via dislocation climb [83]. There is no consensus on a single dominant mechanism to describe irradiation damage in nuclear graphite.

While room-temperature electron-irradiation studies provide valuable insights, no graphite-moderated reactors operate at such temperatures, and studies conducted at elevated temperatures remain scant [54, 74, 75, 76, 81, 88]. Muto and Tanabe [88] inferred through electron diffraction patterns there exists a critical dose above approximately 125°C in which the mechanisms controlling irradiation damage change. Although, when the irradiation temperature was increased to 400°C, lattice spacing remained unchanged and indicated the graphite structure was not locally altered [88]. High-temperature in-situ electron irradiation conducted by

Johns et al. showed the formation of fullerene-like defects; however, only when irradiation temperatures exceeded 750°C [76]. In further studies, fullerene-like defects were found abundant in neutron-irradiated nuclear graphite at comparable dose and temperature to previous electron-irradiation studies [74]. Although, it was noted that such defects only occurred in disordered regions of the microstructure, such as the binder phase, while filler particles appeared to retain their structure [74]. The fullerene-like defects are shown in Figure 5-4; additionally, further experimental evidence of a *ruck and tuck* defect is in Figure 5-4(d) [74].

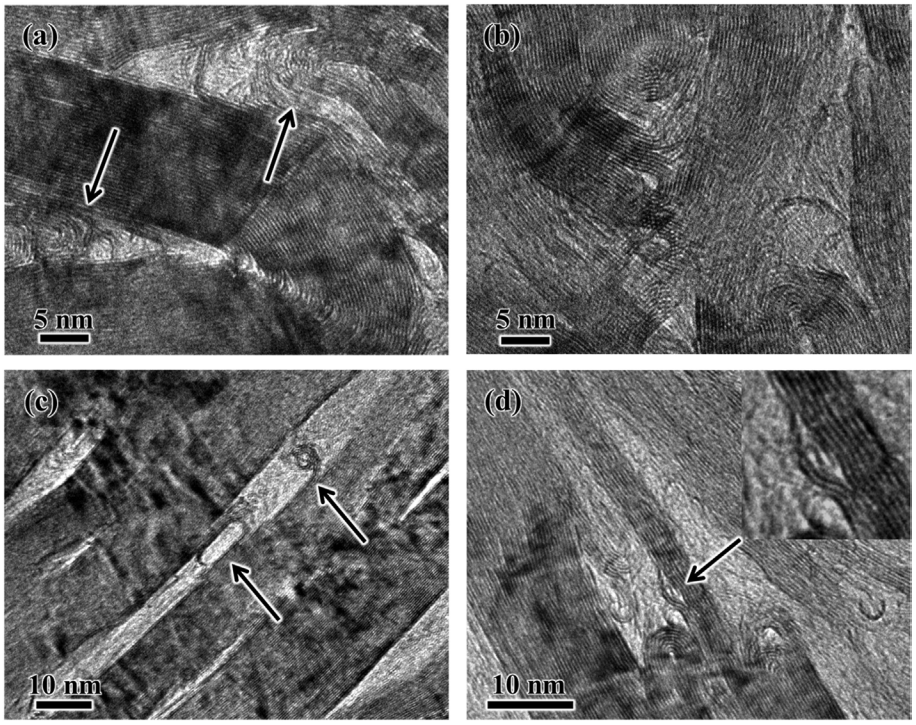


Figure 5-4. HRTEM images of high-temperature neutron-irradiated IG-110. (a) and (b) show the presence of fullerene-like defects occurring within disordered microstructure regions (813°C - 1.73 dpa). (c) and (d) show further evidence of fullerene-like defects occurring within the accommodating porosity in a sample irradiated at 817°C to 3.56 dpa; within a Mrozowski crack in (c) and additionally a 'ruck and tuck' defect in (d). Adapted from Ref. [74].

Defect formation and evolution in the graphite microstructure are not a result of one or a few dominating mechanisms. There exists an entire zoo of defects that arise from fast-neutron bombardment; additionally, the type of defects will change depending on the irradiation temperature [68, 74, 76]. A comprehensive understanding of defects that spans irradiation temperatures of HTR designs has yet to emerge. As a further complication, each microstructural feature (filler, binder, QI, Mrozowski crack, and pores) has a different response to irradiation. Therefore, even though nuclear graphites are all composed of one atomic element, given their complex microstructure, they must be treated similarly to a composite material *for each grade* (although it is also unclear what unit of production might constitute a single grade in this case). For these reasons, complete theoretical models that accurately describe irradiation-induced property change of nuclear graphites do not exist. Thus, predicting the in-service irradiation response for a chosen grade is largely empirical (if irradiation data exists). Indeed, the graphite

core lifetime assessments for the AGR and Magnox reactors of the UK are based on finite element analysis (FEA) using empirical irradiation data [89]. A comprehensive understanding of irradiation damage mechanisms is needed to extend the in-service lifetime of nuclear reactor graphite components.

5.1.2 Bulk Property Change

Due to the accumulation of irradiation damage in the graphite microstructure, the bulk material properties change significantly. Predicting the material properties response to irradiation is critical in determining component in-service lifetime and affects design constraints to maintain a safety envelope. The complex correlations among nuclear graphite property changes are still not fully understood. The current understanding of the irradiation-induced change of dimensional, mechanical, thermal, and electrical properties is discussed in the following sections. Example data sets are shown that are representative of the changes that occur but are not comprehensive for all the data that are currently available in the literature.

5.1.2.1 Dimensional and Volumetric Change

Dimensional change can be argued to be one of the essential properties in designing and predicting the in-service lifetimes of graphite-moderated reactors. On the nanoscale, crystallites will expand along the *c*-axis and contract in the *a/b*-axis. On the macroscale, this behavior occurs across the grains or the needle-like filler particles. Filler particles will contract in length and expand along the widths during irradiation. While nuclear graphites are manufactured to produce a near isotropic material, regardless of forming method, there remains an inherent preferential grain orientation within the bulk. Therefore, the irradiation-induced linear dimensional change is different with grain and against the grain directions of a graphite component [90, 91, 92]. Examples of this behavior are shown for the graphite grades ATR-2E [93] and G347A [91] at nominal temperatures of 400°C and 750°C (refer to references for actual temperatures) in Figure 5-5. Depending on the function and geometry of the graphite component, design considerations may need to incorporate anisotropy in linear dimensional change.

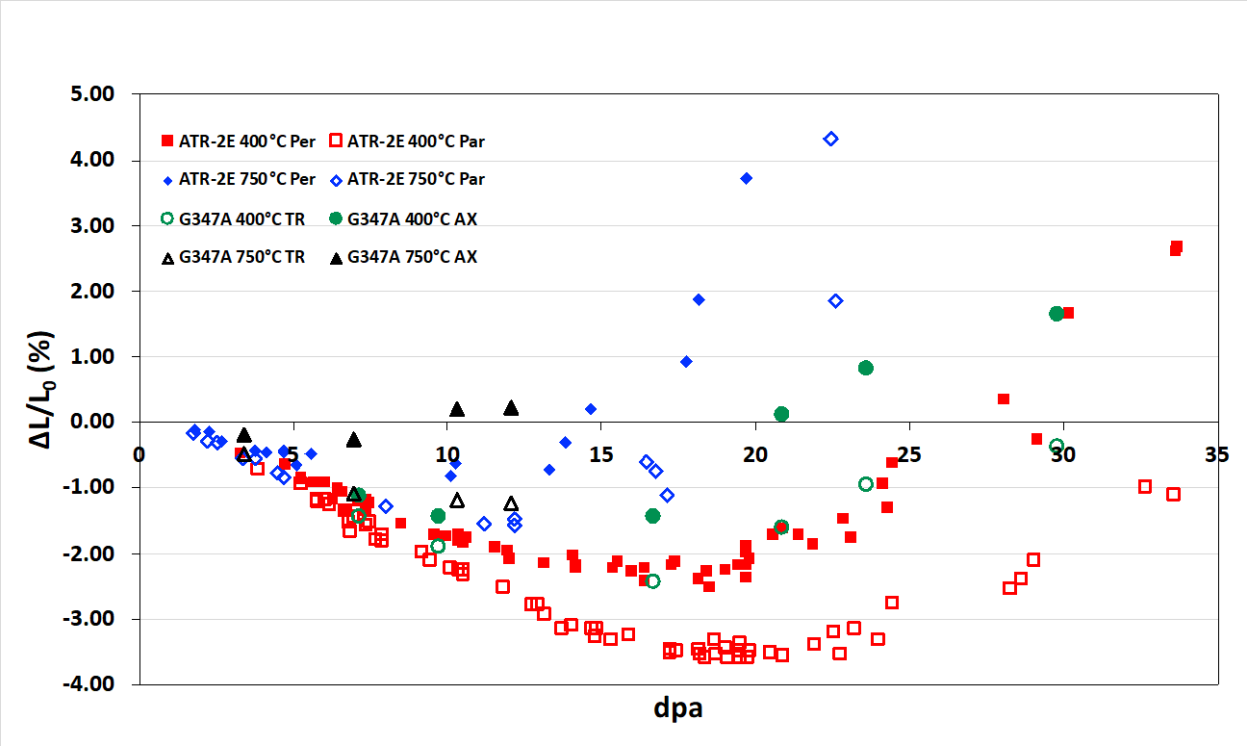


Figure 5-5. Plot of relative length change of ATR-2E [93] and G347A [91] at nominal temperatures of 400°C and 750°C, highlighting the anisotropic length change relative to billet orientation. ATR-2E data reported as "per" (perpendicular) and "par" (parallel) to billet extrusion direction. G347A data reported as axial (AX) and transverse (TR) relative to billet molding direction.

A more general approach to analyzing the irradiation-induced dimensional change is the analysis of the volumetric change, which normalizes component geometries and grain orientations. However, the amount of volumetric change varies amongst each grade of nuclear graphite due to processing techniques and the resulting microstructure [57, 89, 92]. An example of this grade-to-grade difference is shown in Figure 5-6 for nine different grades irradiated at a nominal temperature of 750°C. At the onset of irradiation, there is a high shrinkage rate. The current consensus for the mechanism which explains net volumetric shrinkage is the accommodating porosity and Mrozowski cracks [74, 89, 94]. Mrozowski cracks are formed during cooling from graphitization temperatures due to the Van der Waals bonding between basal planes and anisotropy in the CTE between the *a* and *c*-axis. Consequently, Mrozowski cracks have lengths on the *a/b*-axis and widths along the *c*-axis of crystallites, as shown in Figure 5-7, thus accommodating *c*-axis expansion due to irradiation. The net shrinkage is explained by the contraction along the *a/b*-axis.

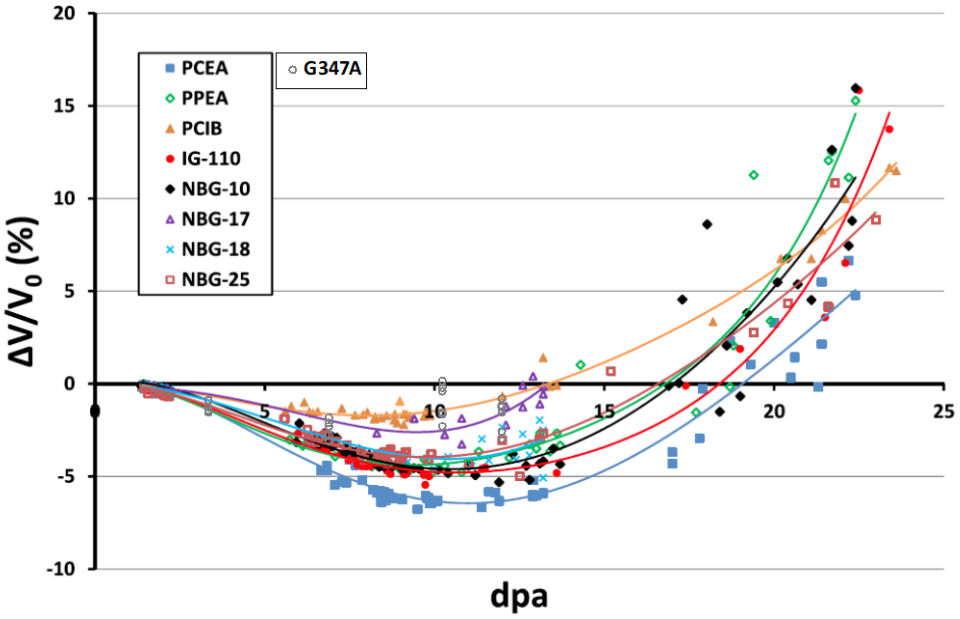


Figure 5-6. Volumetric change for multiple graphite grades, nominally irradiated at 750°C. Data for PCEA, PPEA, PCIB, IG-110, NBG-10, NBG-17, NBG-18, and NBG-25 from [95].

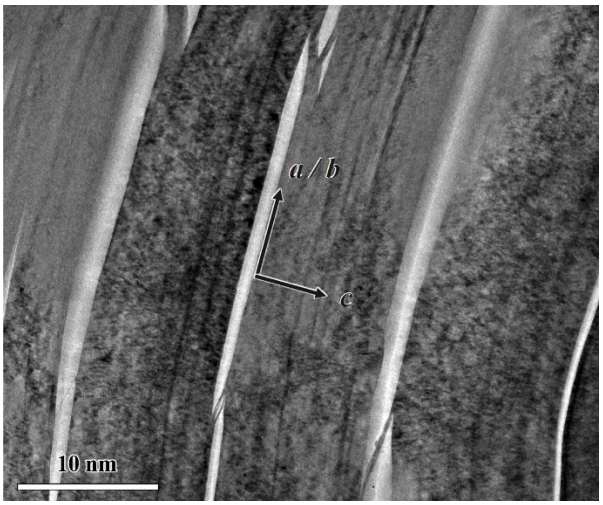


Figure 5-7. Mrozowski cracks in nuclear graphite IG-110. Indicated is the direction in which Mrozowski cracks form with respect to the crystallographic orientation of crystallites [Figure 1.4(a) from [216]].

When a significant amount of the accommodating porosity is decreased, the volumetric change is reversed, and the graphite component will expand (turnaround dose). Expansion of the graphite component continues until the component returns to its pre-irradiation volume (crossover). The turnaround dose is a critical aspect of the volumetric change as it can be a predictive measure of the in-service lifetime of the graphite component. Additionally, the turnaround dose signals when other properties, such as the elastic modulus and CTE, will start to degrade significantly [96 , 97]. The amount of volumetric change and the turnaround dose is a function of temperature. Figure 5-8 shows volumetric curves for G347A [91] as a function of

temperature. As irradiation temperature increases, the turnaround dose and the magnitude of volumetric change decrease. The atomic mechanisms governing turnaround behavior in nuclear graphite are still not well understood; thus, no theoretical models exist [74, 89, 94].

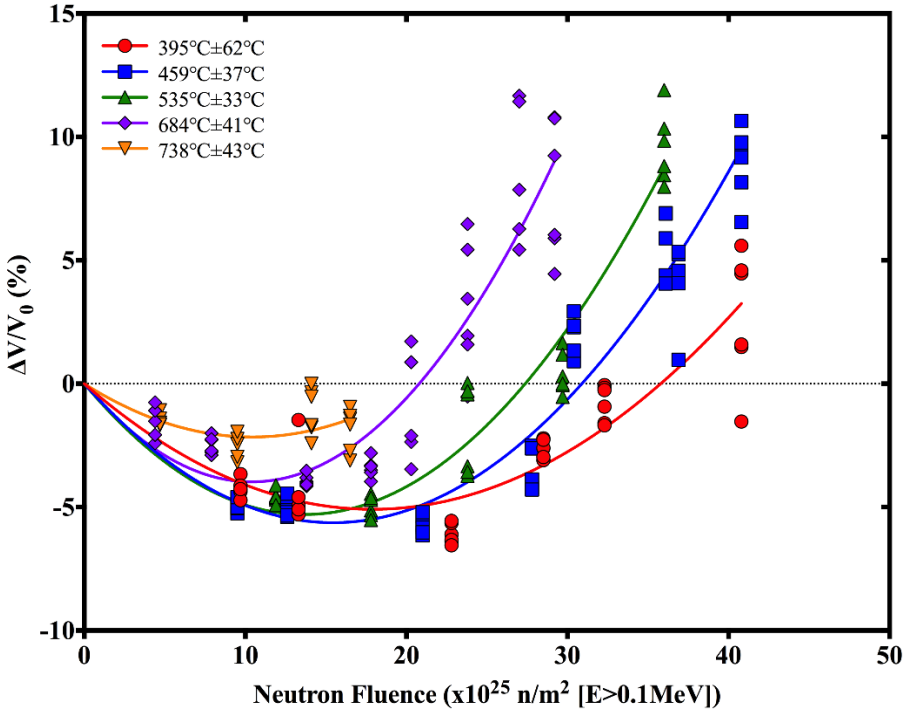


Figure 5-8. Volumetric change of G347A at various irradiation temperatures [91]

The irradiation data needed to empirically model and predict the irradiation behavior for most next-generation nuclear graphites does not exist. Encompassing all potential nuclear graphites for HTR reactor designs in the ASME material data sheets is impossible without new irradiation programs.

5.1.2.2 Mechanical Properties

The two primary mechanical properties of concern for graphite are the elastic modulus (i.e., Young's modulus) and strength. The effect of neutron irradiation on these properties is thought to be primarily driven by the changes to the bulk density. The effect of neutron irradiation on the Young's modulus for multiple graphite grades irradiated at a nominal temperature of 750°C is shown in Figure 5-9 from [91, 92]. The effect of different irradiation temperatures on the Young's modulus of a single grade of graphite is shown in Figure 5-10 from [91]. The long-held belief is that the change to the Young's modulus is a two-process function controlled by both dislocation pinning causing the low-dose increase, while higher dose changes are due to structural changes [98]. This two-process dependence of Young's modulus change was developed for grades that have medium grain grades (0.1 – 4 mm), while data from superfine grain grades (0.01 – 0.05 mm) do not show this two-process mechanism and instead appear to have a stronger dependence on only density changes [98]. In both cases, the maximum increase

in the Young's modulus occurs at the turn-around dose where the irradiated graphite has the highest density. An interesting phenomenon to note is that even when volume change returns to zero, the Young's modulus does not return to the pre-irradiation value.

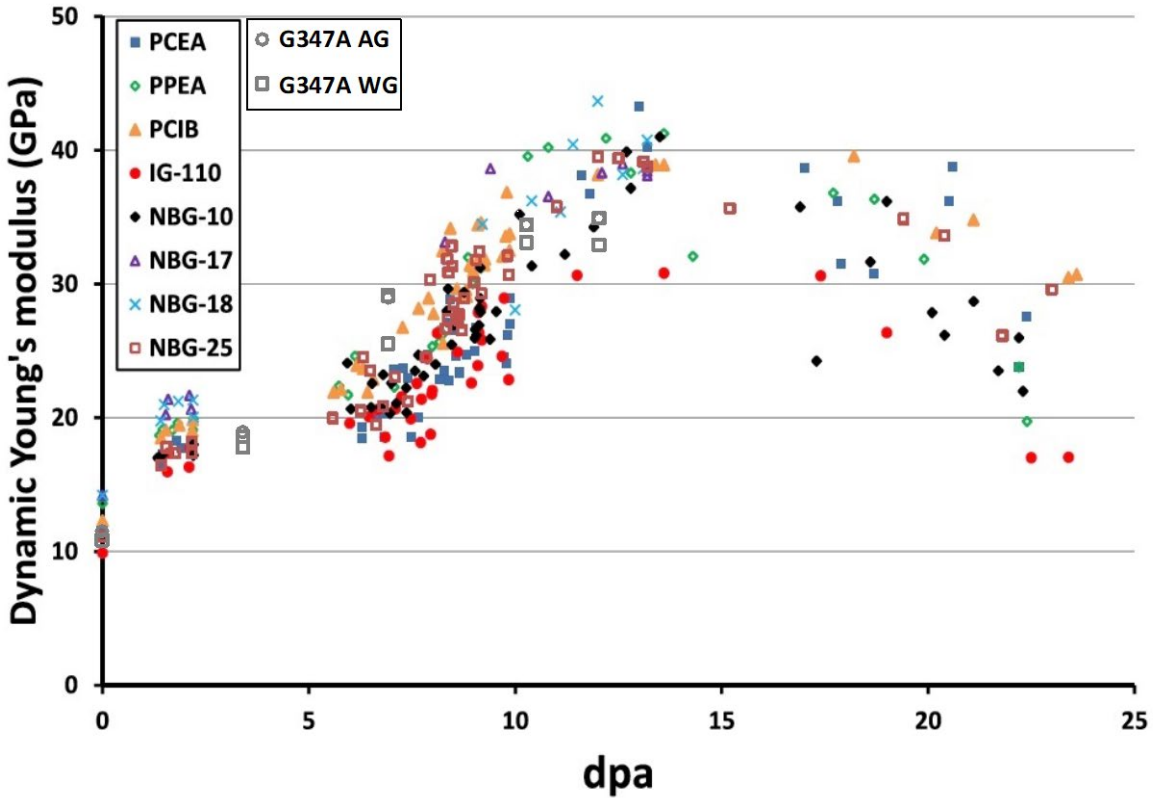


Figure 5-9. Young's modulus for multiple graphite grades, nominally irradiated at 750°C. Data for PCEA, PPEA, PCIB, IG-110, NBG-10, NBG-17, NBG-18, and NBG-25 from [92], and G347A from [91].

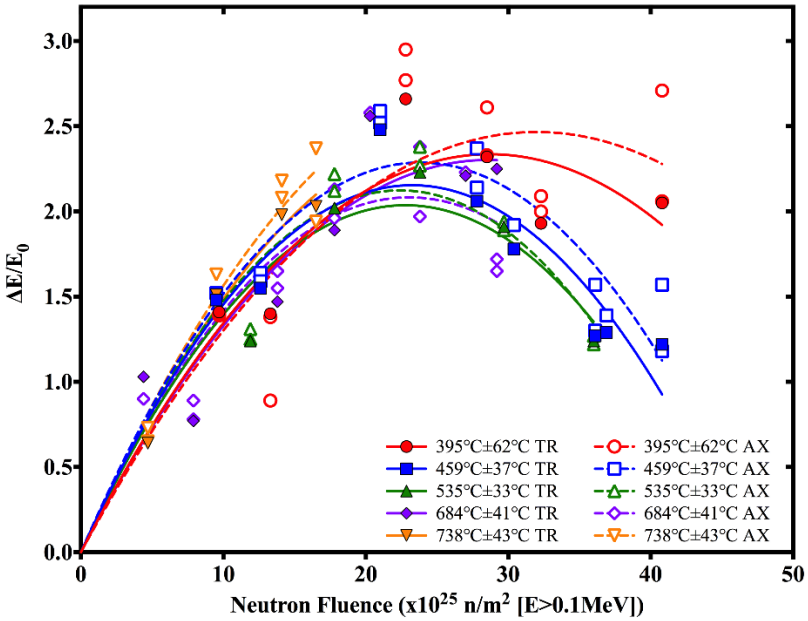


Figure 5-10. Young's modulus change of G347A at various irradiation temperatures [91]

The effect of neutron irradiation on the Young's modulus for multiple graphite grades irradiated at a nominal temperature of 750°C is shown in Figure 5-11 from [91, 92]. The effect of different irradiation temperatures on the Young's modulus of a single grade of graphite is shown in Figure 5-12 from [91]. The strength change for the grades from Reference [92] was measured via a modified split disk testing method to estimate the tensile strength, while the changes to G347A [91] were measured via an equibiaxial flexural testing method. The same two-phase dependence of Young's modulus on dose has been long held as the same processes driving the strength change [98]. There are not enough low-dose data for the newer graphite grades to comment on this two-phase process. An interesting phenomenon to note is that even when volume change returns to zero, the strength does not return to the pre-irradiation value, but the total hysteresis between pre- and post-irradiation values is smaller than that observed for the Young's modulus.

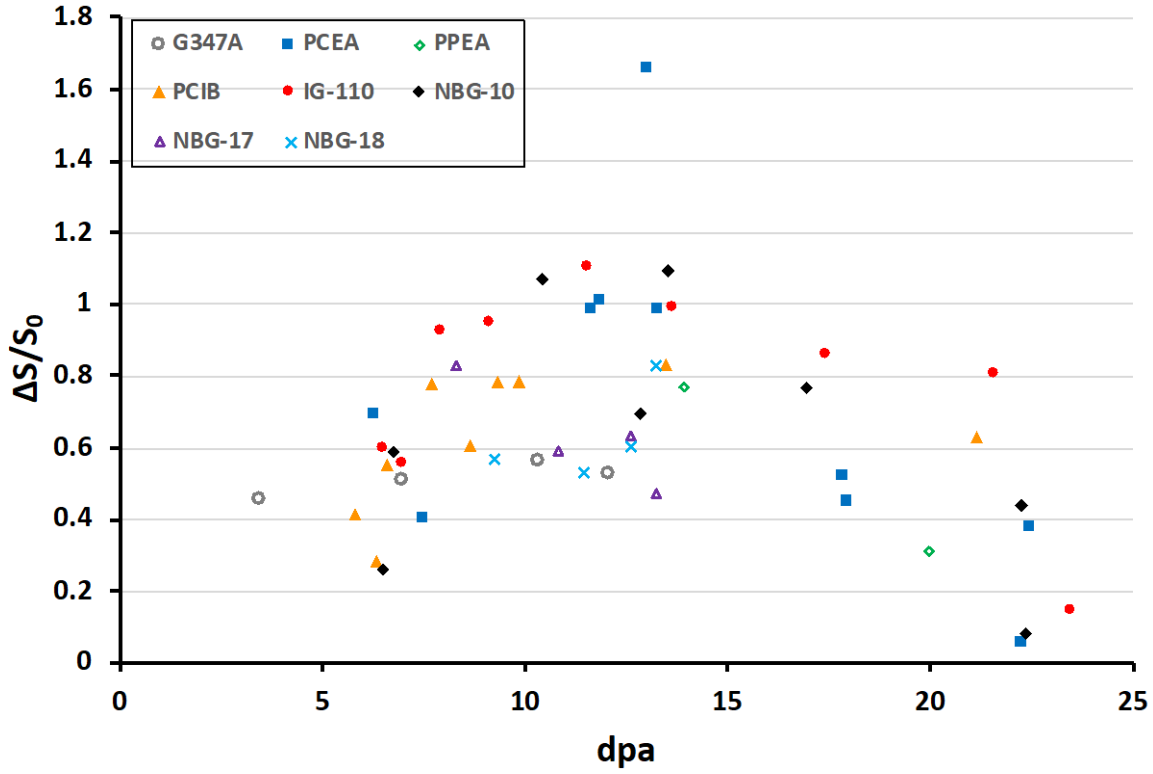


Figure 5-11. Strength change for multiple graphite grades, nominally irradiated at 750°C. Data for PCEA, PPEA, PCIB, IG-110, NBG-10, NBG-17, NBG-18, and NBG-25 is a result of split disk tensile strength from [92], and G347A is a result of equibiaxial strength from [91].

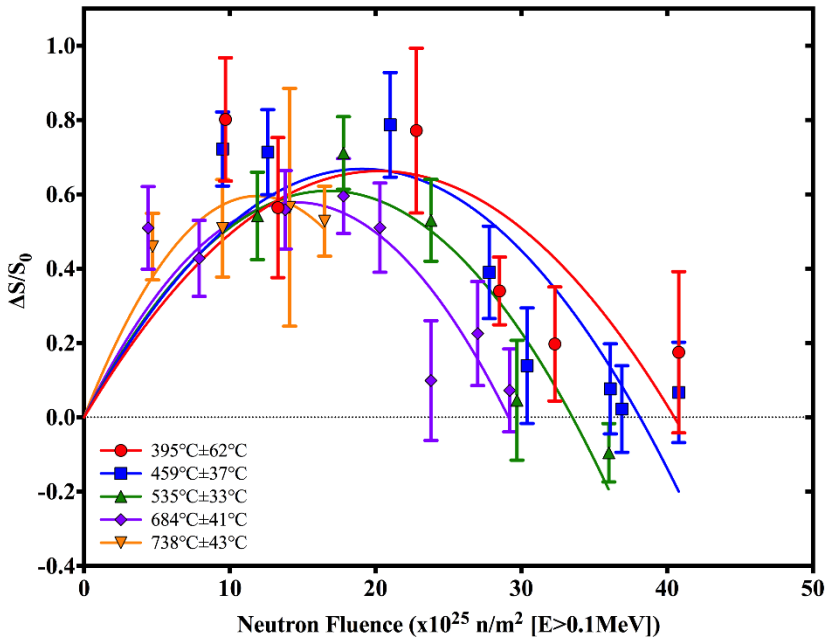


Figure 5-12. Strength change of G347A at various irradiation temperatures [91]

5.1.2.3 Thermal Properties

The coefficient of thermal expansion (CTE) and the thermal conductivity are both important thermal properties for core structural materials. The CTE of a material dictates the amount of swelling that occurs during reactor startup/shutdown. The CTE in bulk graphite is fundamentally different from the CTE for the two directions within the anisotropic crystal structure (Figure 5-1). The bond anisotropy results in a the c-axis thermal expansion being a factor of ~35 higher than that for the a-axis [98]. This large difference in crystal CTE is thought to be the source of the Mrozowski cracks [74, 89, 94]. The irradiation induced mean CTE for multiple graphite grades irradiated at a nominal temperature of 750°C are shown in Figure 5-13 from [91, 92]. The effect of irradiation temperature on the measured mean CTE and the net change of mean CTE is shown for graphite grade G347A in Figure 5-14 from [91]. The trends for these different grades and different temperatures are all similar, where there is an initial increase in mean CTE that peaks at roughly ½ the turn-around dose, then decreases to a value lower than pre-irradiation that ceases around the turn-around dose, and then the mean CTE remains nearly constant for further increasing fluence. Multiple authors have put forth a discussion about the source of this three-phase behavior [91, 93, 98, 99], which occurs as follows: 1) the initial increase in CTE is due to the irradiation induced closure of Mrozowski cracks due to c-axis swelling, thereby reducing the volume of these cracks available to accommodate thermal expansion, 2) the decrease is due to the formation of new accommodation porosity, and 3) the primary source of swelling after turn-around is due to the formation of porosity that does not provide new accommodation of thermal or irradiation c-axis expansion. An interesting point to note from Figure 5-13 and Figure 5-14a is that the mean CTE for all grades, and for a range of irradiation temperatures, is roughly the same once they reach saturation even though the pre-irradiation values have a much larger spread.

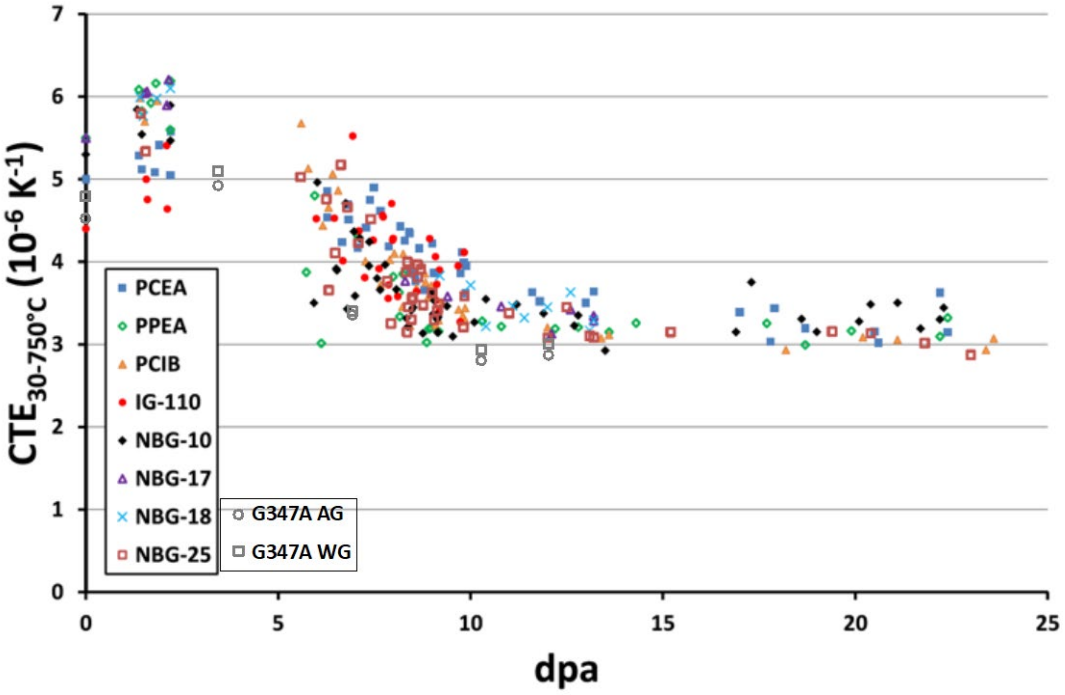
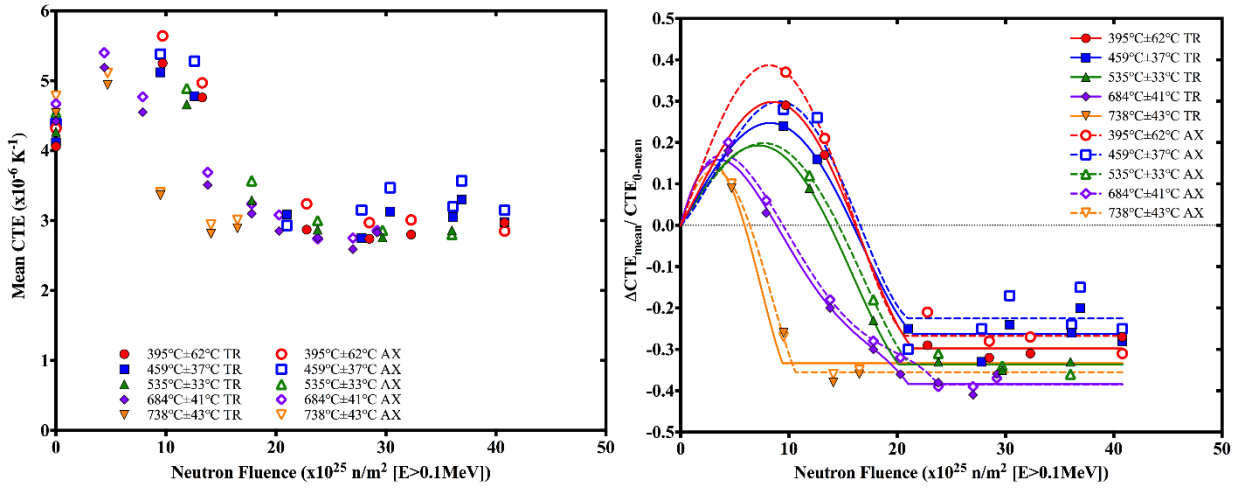


Figure 5-13. Mean CTE for multiple graphite grades, nominally irradiated at 750°C. Data for PCEA, PPEA, PCIB, IG-110, NBG-10, NBG-17, NBG-18, and NBG-25 from [92], and G347A from [91].



(a) (b)

Figure 5-14. (a) Mean CTE for G347A measured at irradiation temperature and (b) net change of CTE at irradiation temperature [91]

Thermal conductivities of materials are critical in nuclear reactors where structural materials are not just providing support but also heat transfer from the fuel to the reactor coolant. As manufactured graphite has a high starting thermal conductivity at low temperatures (>100 W/m-K below 100°C). The conductivity decreases with increasing temperature because heat

transfer in graphite is phonon driven, and higher temperatures increase phonon-phonon scattering (Umklapp) [46]. Figure 5-15 shows the thermal conductivity measured at 750°C of multiple graphite grades irradiated at 750°C from [91, 92]. Figure 5-16 highlights the effect of irradiation temperature on the (a) measured and (b) net change to the thermal conductivity measured at irradiation temperature [91]. The data in Figure 5-15 and Figure 5-16 show that irradiation damage causes a two-phase response in the changes of the thermal conductivity. The first phase is the initial reduction by 50-80% of the initial thermal conductivity and is thought to be due to the rapid saturation of point defects that perturb the phonon scattering. The second phase is the continued reduction with increasing fluence that is thought to be due to pore generation [91, 98].

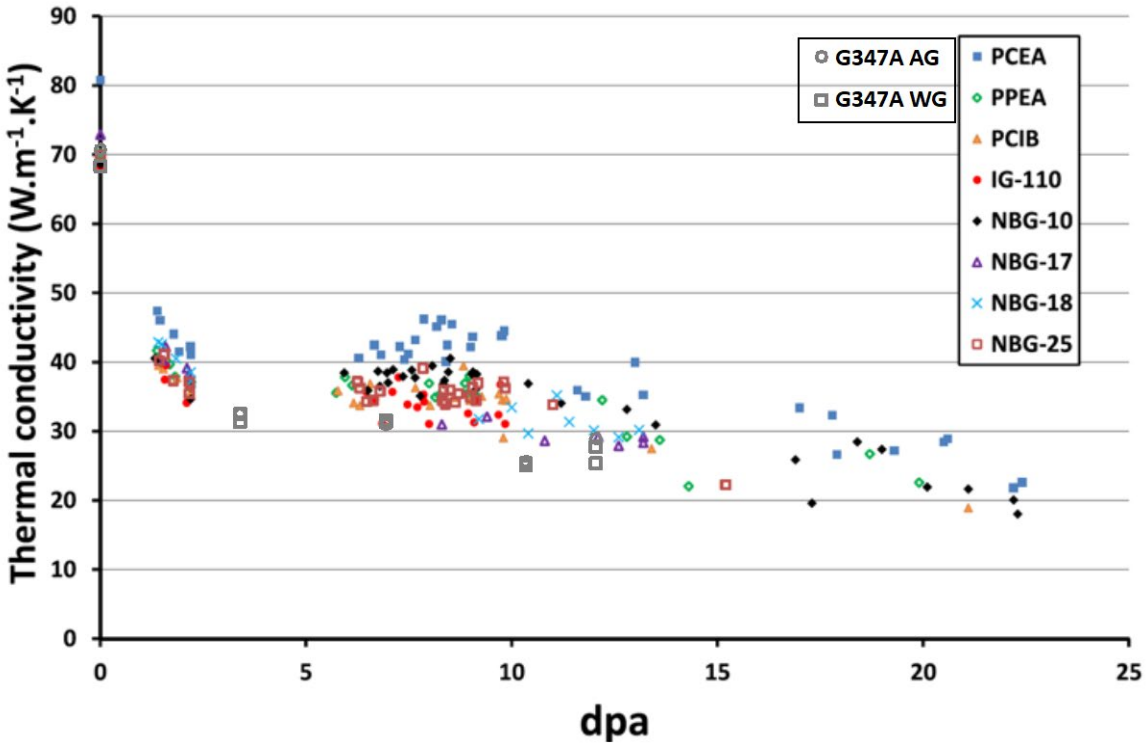
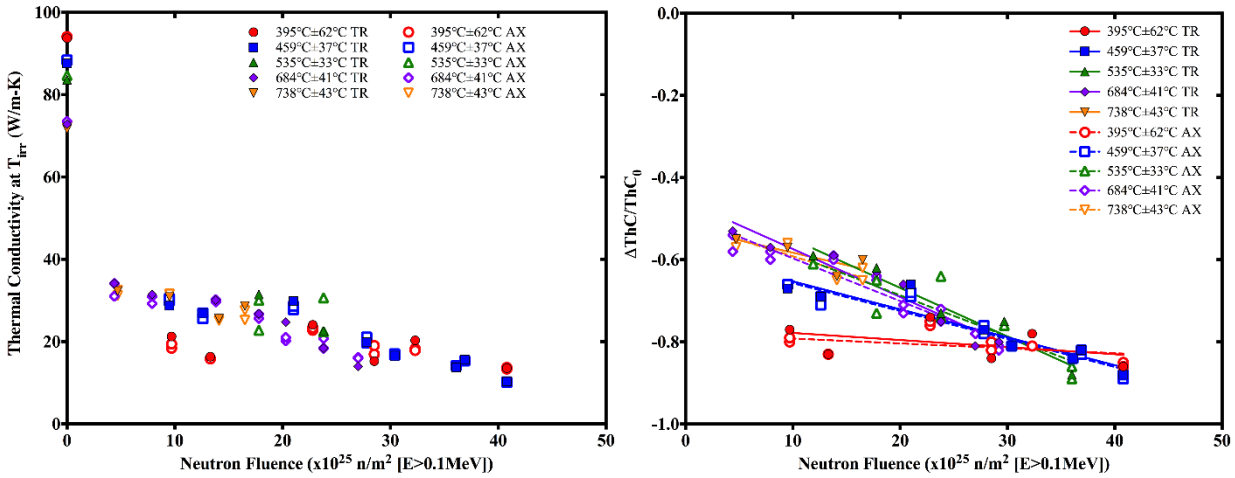


Figure 5-15. Thermal conductivity for multiple graphite grades, nominally irradiated at 750°C. Data for PCEA, PPEA, PCIB, IG-110, NBG-10, NBG-17, NBG-18, and NBG-25 from [92], and G347A from [91].



(a) (b)

Figure 5-16. (a) Thermal conductivity of G347A measured at irradiation temperature for different irradiation temperatures and (b) thermal conductivity change of G347A at various irradiation temperatures [91]

5.1.2.4 Electrical Properties

The electrical resistivity of graphite is not as actively studied as the other materials properties, mainly because heat transport in graphite is primarily driven by phonon scattering rather than electronic conduction. The comparison of the net change of electrical resistivity for samples ATR-2E [93] and G347A [91] for multiple irradiation temperatures is shown in Figure 5-17. Both graphite grades show an initial early rise in resistivity followed by a region of relatively no change and a later increase at high dose. The initial rise is postulated to be due to the shortening of the electron mean free path by crystal distortion and point defects, the plateau region is thought to be due to the increasing density of scattering sites and electron holes negating additional increases from the crystal distortion.

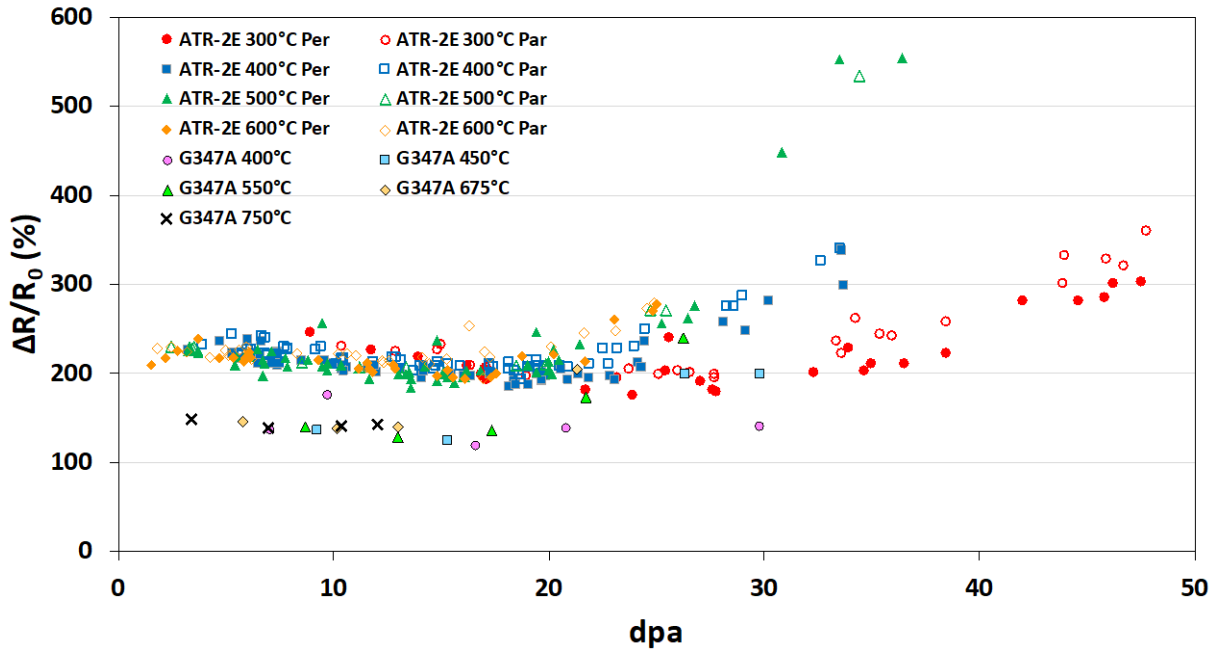


Figure 5-17. Electrical resistivity change for ATR-2E [93] and G347A [91]

5.1.2.5 Summary Bulk Property Changes

The changes that occur in the materials properties of graphite are quite complex. To date multiple mechanisms that describe these changes have been proposed, but large data sets to support or disprove these mechanisms are not available. The other limiting factor related to understanding these changes is that until recently very few characterization techniques existed that could capture irradiation-induced changes in the graphite microstructure. The lack of characterization techniques was due to multiple challenges, but newer techniques and advanced equipment are now able to support the studies necessary to quantify these changes.

5.1.3 Irradiation Creep

Thermally activated creep in materials generally occurs at temperatures that are higher than 30%-50% of the melting temperature, but when exposed to irradiation creep can occur at much lower temperatures because the production of defects that drive creep is no longer thermally driven but instead produced via irradiation-induced displacements. In most metallic systems creep is generally regarded as a life-limiting behavior because the plastic deformation results in the components deforming in a way that limits or ends its intended function [100]. In graphite, irradiation creep is a beneficial phenomenon because it is a mechanism through which stress relaxation can occur that extends graphite component lifetime [98].

During reactor operation, graphite blocks will experience both temperature and flux gradients resulting in different regions within the block having different localized dimensional/volume change rates like those shown in Figure 5-5 and Figure 5-8. Tsang and Marsden [101] developed a finite element model of a graphite moderator block to better understand the stress states that arise in annular graphite bricks due to irradiation. A simplified discussion of this behavior is

shown in Figure 5-18. The picture in Figure 5-18a is a radial cross section of a hypothetical annular graphite block. The rainbow gradient shows the possible neutron flux or temperature gradient that such a block may experience during normal reactor operation. In this example, center of the annular block is hollow and would generally contain fuel, hence the inner surface of the block will experience the highest flux and temperature. In the simplest case, the block is at a constant temperature with a flux gradient, which causes the localized dimensional change within the block to proceed in a manner shown in Figure 5-18b. In this case each region within the block undergoes the same maximum dimensional shrinkage at the same neutron fluence, but the time where this occurs for each region is different because each has a different flux. In a more realistic case, the block has both temperature and flux gradients, which causes the localized dimensional change within the block to proceed in a manner shown in Figure 5-18c. In this case the hotter region near the central hole undergoes less dimensional shrinkage and reaches turn-around much faster than the outer edge of the block.

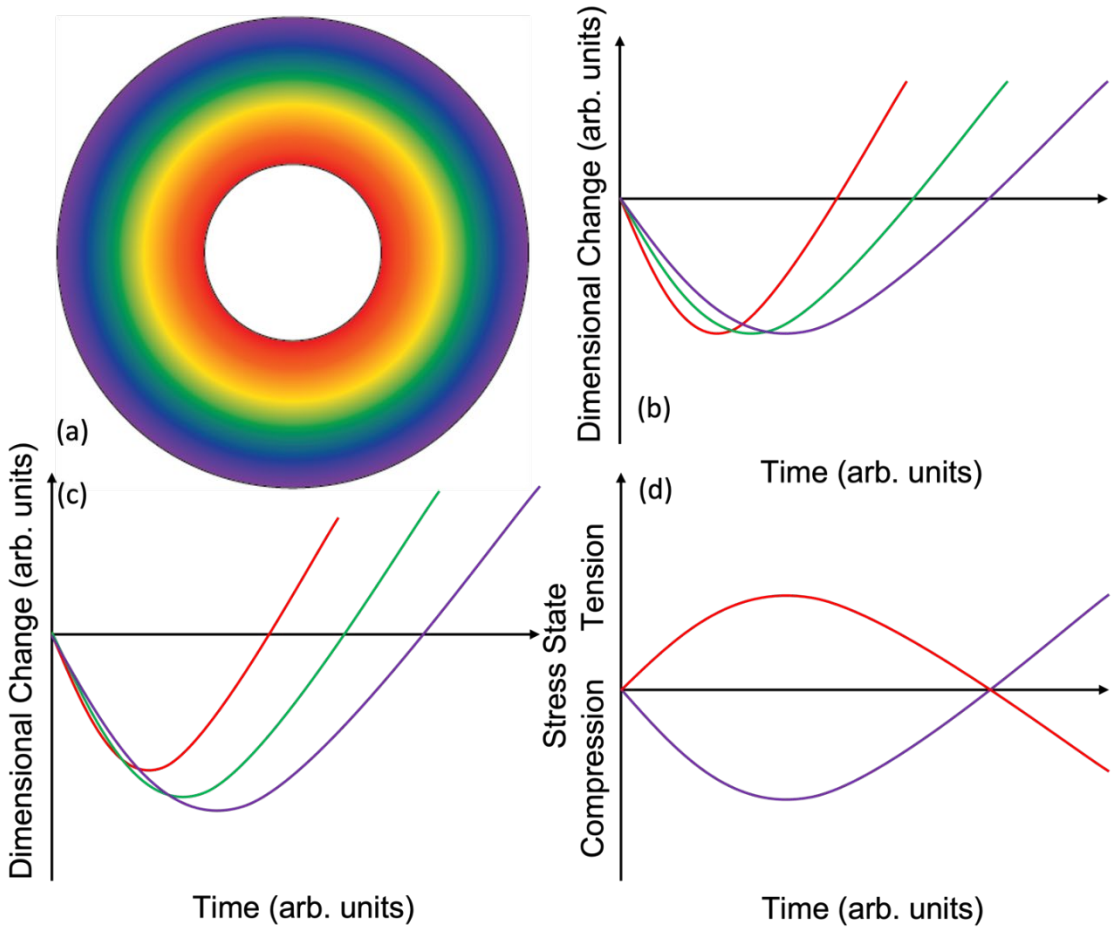


Figure 5-18. (a) Hypothetical neutron flux or temperature gradient across the radius a simplified annular graphite block (center is higher value and outer is lower). (b) Hypothetical dimensional change within different regions of block in (a) with a constant temperature across the radius of the block. (c) Hypothetical dimensional change within different regions of block in (a) with temperature and flux gradient across the radius of the block. (d) Stress state changes in the inner (red) and outer (purple) regions of the hypothetical block in (a). (Example based on results published in [101]).

Once all the dimensional changes are accounted for, the localized stress within each region can be estimated, which is shown in Figure 5-18d. For the central region of the block with the highest temperature and flux, the material experiences faster dimensional shrinkage than the regions around it, putting this region initially into a tensile stress state (red line in Figure 5-18d), while the outer edge is contracting slower and is put into a compressive stress state (purple line in Figure 5-18d). As this behavior starts to change and the inner region starts to swell the center region begins to expand while neighboring regions are still contracting or expanding at a slower rate, which changes the stress state in the inner region to compressive. At the same time the outermost region of the block switches from compressive to tensile stress. In the calculations by Tsang [101], the maximum tensile stress for the inner region was predicted to be ~8 MPa, which in many graphite materials is approaching ~50% the tensile strength. It is during these times of high compressive or tensile stresses that irradiation creep as a stress relaxation mechanism assists with extending the lifetime of the blocks, especially late in component lifetime when the outer region of the block starts to experience the build-up of tensile stresses that will eventually lead to cracking of the block.

It is worth noting that the example case given in Figure 5-18 for an annular block configuration is most likely a worst-case scenario. This is due to the block having a flux and temperature gradient that radiates out from the center of the block (due to fuel loading in the block center) and the annular shape of the block restraining volumetric changes. Newer reactor designs like the prismatic or pebble bed gas-cooled reactors and the molten salt reactors will have less concerns due to these issues. In prismatic gas-cooled reactors the fueled graphite blocks will have an array of >100 holes for fuel, meaning that the flux and temperature across the blocks will be more uniform. The reflectors for these various designs will have a more uniform gradient across the blocks and limited restraints for dimensional change.

5.1.3.1 Creep Behavior

Irradiation-induced creep in graphite is observed to occur in three stages (much like metals): primary, secondary, and tertiary. This three-stage behavior is observed in the 500-550°C compressive and tensile creep results shown in Figure 5-19. The data at 300°C in Figure 5-19 does not appear to have yet reached the tertiary creep behavior, which is expected because the creep and dimensional change of graphite occurs over a longer dose/time at lower irradiation temperatures.

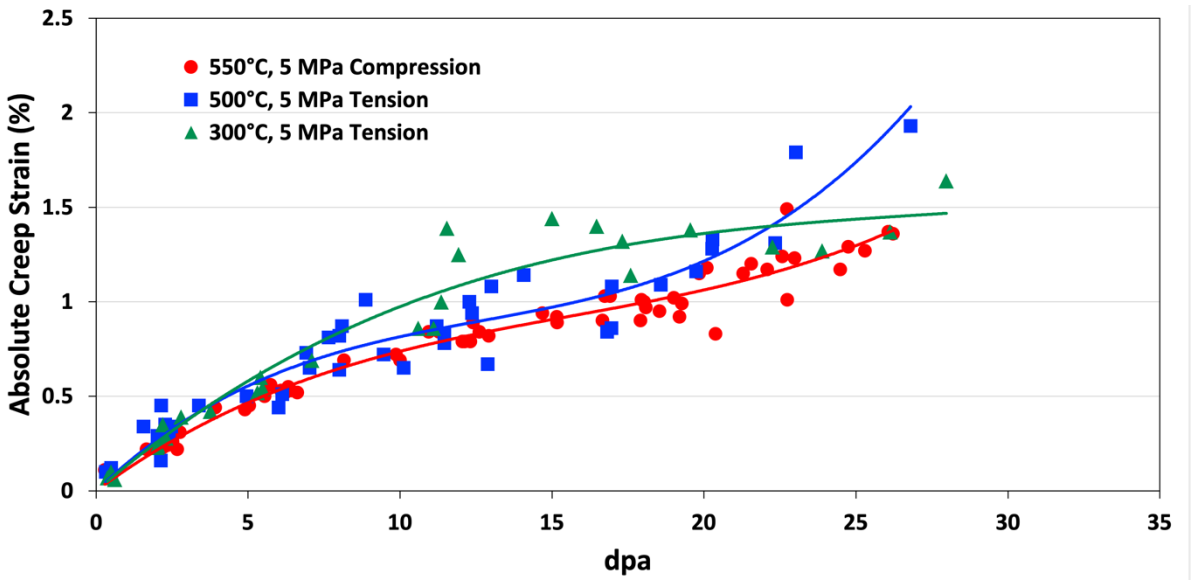


Figure 5-19. Absolute creep strain of ATR-2E irradiated in tension at two temperatures, and at two different stress states (data from [93]). The line fits are third-order polynomials with a y-intercept set to zero and are purely for guiding the eye to the behavior of the data.

Multiple conditions have effects on the creep behavior of graphite. The three main environmental conditions are temperature, neutron flux, and applied stress. The primary creep strain has been shown to be linearly dependent on applied stress [102, 103, 104, 105]. Most of the assumptions have been that the primary creep saturates around a value equal to the applied stress divided by the pre-irradiation Young's modulus (called an Elastic Strain Unit or ESU) [102, 103, 104], but more recently it was shown that this saturation value is not always equal to one [53] and is actually temperature dependent [105]. The effect of experimental conditions on the steady state creep rate are as follows:

- The effect of temperature on the steady-state creep coefficient is roughly linear for temperatures greater than 600°C because the activation energy being quite small (~0.3-0.4 eV) [98, 105, 106, 107, 108, 100].
- Within the 300-600°C range the creep coefficient is roughly constant.
- At temperatures below 300°C the creep coefficient increases with decreasing temperature.

An example of this temperature dependence is shown in Figure 5-20 for multiple graphite grades irradiated in the Oak Ridge Research Reactor [105, 107]. The effect of neutron flux on the steady state creep rate has been shown to be linearly dependent on flux for constant stress experiments [105, 108], while restrained shrinkage experiments [106] suggested an inverse dependence on flux. The steady state creep rate has a linear dependence on applied stress [103, 105, 108, 109, 110]. There is not sufficient experimental data at high doses to provide any commentary about the environmental effects on tertiary creep.

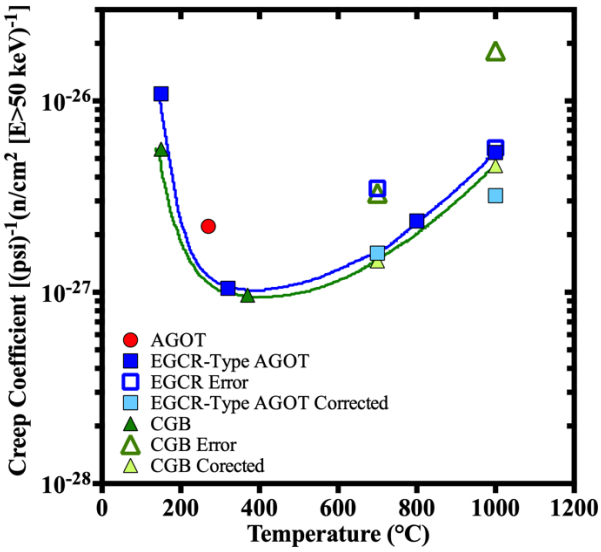


Figure 5-20. Plot of creep coefficient versus irradiation temperature for multiple graphite grades from [105, 107]

The mechanism of irradiation creep in graphite is still not agreed upon. Historically a mechanism based on dislocation glide (called the pinning-unpinning mechanism [111]) that occurs within graphite crystals has been accepted. Recently published work from the 1960s [105, 107], and newer experimental [112] and modeling efforts [113], have proposed that the mechanism of irradiation creep is actually due to the climb of dislocations rather than glide.

5.1.3.2 Creep Models

Initially irradiation creep of graphite was described by the linear visco-elastic creep model [104, 105, 114, 115, 116, 117, 118], where the total creep strain is a sum of the transient and steady state defined by:

$$\varepsilon_c = \frac{a\sigma}{E_0}[1 - \exp(-b\gamma)] + k\sigma\gamma \qquad [5\text{-}1]$$

where ε_c is total creep strain, σ is applied stress, E_0 is pre-irradiation Young's Modulus, γ is fast neutron fluence, a and b are constants ($a \cong 1$), and k is the steady state creep coefficient in units of reciprocal neutron dose and reciprocal stress.

When it was determined that structural changes within graphite were affecting the creep rate, the UK creep model [114, 119, 120] was proposed, which utilized changes to the Young's modulus to quantify the structural changes. The creep strain (ε_c) from the UK model is given by:

$$\varepsilon_c = \frac{\sigma}{E_0} + \left(\frac{d\varepsilon_c}{d\gamma}\right)_0 \sigma \int_0^{\gamma} \frac{E_p}{E_\gamma}(\gamma) \cdot d\gamma \qquad [5\text{-}2]$$

where σ is applied stress, $(d\varepsilon_c/d\gamma)_0$ is the initial secondary creep rate, γ is fast neutron fluence, E_p is the Young's Modulus after the initial increase due to dislocation pinning, and E_γ is the Young's Modulus at dose γ. This model does not account for the fact that the stress state (compressive versus tensile) alters the structural changes.

Shortly after the UK model was proposed, Kennedy [121] replaced the Young's modulus based structural term based on the Young's modulus with a term that is dependent on the volume change. The creep strain (ε_c) is given by:

$$\varepsilon_c = \frac{\sigma}{E_0} + \sigma\gamma k_0 \int_0^\gamma \left[1 - \mu\left(\frac{{}^{\Delta V}/_{V_0}}{\left({}^{\Delta V}/_{V_0}\right)_{max}}\right)\right] d\gamma \qquad [5\text{-}3]$$

where σ is applied stress, E_0 is pre-irradiation Young's Modulus, γ is fast neutron fluence, μ is an empirical constant equal to 0.75, k_0 is the steady state creep coefficient established from low dose creep experiments, $(\Delta V/V_0)$ is the volume change at fluence γ, and $(\Delta V/V_0)_{max}$ is the maximum volumetric contraction for an unstressed sample. This model predicted high dose tensile creep well but not compression, which is likely due to the lack of acknowledgement of the stress state effects on structural changes. Furthermore, this model is not useful for low temperature creep since the there is no volumetric contraction at low temperatures [122].

After the Kennedy model was found to be lacking, the Kelly and Burchell [122, 123] model was proposed, which utilizes changes to the thermal expansion and crystallite dimensions to model the creep strain (ε_c) is given by:

$$\varepsilon_c = \left(\frac{\sigma}{E_0} + k\sigma\gamma\right) + \int_0^\gamma \left[\left(\frac{\alpha_x' - \alpha_x}{\alpha_c - \alpha_a}\right)\left(\frac{1}{X_c}\cdot\frac{dX_c}{d\gamma} - \frac{1}{X_a}\cdot\frac{dX_a}{d\gamma}\right)\right]\cdot d\gamma \qquad [5\text{-}4]$$

where σ is applied stress, E_0 is pre-irradiation Young's Modulus, γ is fast neutron fluence, k is the steady state creep coefficient in units of reciprocal neutron dose and reciprocal stress, α_x is the thermal expansion coefficient in the x-direction, α_c and α_a are the thermal expansion coefficients of the graphite crystal parallel and perpendicular to the hexagonal axis (<c>-axis), respectively, over the same temperature range, and $(1/Xc)(dXc/d\gamma)$ and $(1/Xa)(dXa/d\gamma)$ are the rates of change of graphite crystallite dimensions parallel and perpendicular to the hexagonal axis. This model was shown to reasonably predict the compressive creep behavior of grade H-451 at 600°C and 900°C up to ~3.4 dpa, but did not predict the measured creep for tensile creep above this dose at 900°C as shown in Reference [123].

The newest proposed empirical creep model is the M^2 model from Davies and Bradford [124, 125] (both named Mark hence the M^2 designation). This model includes three terms that include primary creep, secondary creep, and recoverable creep. In this model creep strain (ε_c) is given by:

$$\varepsilon_c = \frac{\alpha k_1}{E_0}\exp(-k_1\gamma^1)\int_0^{\gamma^1}\left[\frac{\sigma}{SW}\exp(k_1\gamma)\right]\cdot d\gamma + \frac{\omega k_2}{E_0}\exp(-k_2\gamma^1)\int_0^{\gamma^1}\left[\frac{\sigma}{SW}\exp(k_2\gamma)\right]\cdot d\gamma + \frac{\beta}{E_0}\int_0^{\gamma^1}\left[\frac{\sigma}{SW}\right]\cdot d\gamma \quad [5\text{-}5]$$

where σ is the applied stress; α, ω, and β are empirical fitting parameters; k_1 and k_2 are doses where primary and recoverable creep saturate respectively; and S and W capture the changes to Young's modulus due to structural and radiolytic oxidation, respectively. The first term in the equation is primary creep, the second term is recoverable creep, and the third is secondary creep. This model was shown to predict the creep strain for the tensile creep at 500°C and the compressive creep at 550°C data in Figure 5-19 up to ~20 dpa, along with the low-dose compression data for grade H-451 from Reference [123] that only went to ~3.5 dpa. The M^2 predicts that, in the 350-600°C temperature range, primary creep strain saturates around 1 ESU, while recoverable creep strain saturates around 3 ESU, with the secondary creep rate was constant over this temperature range.

5.1.3.3 Summary of Irradiation Creep

Irradiation-induced creep in graphite is a complex phenomenon that is still not fully understood. Multiple mechanisms to describe the in-crystal creep are still be discussed as there does not exist a sufficiently large data set to support or disprove these hypotheses, but the component of bulk structural changes also needs to be determined. The main cause for this is that irradiation creep experiments are complex to design and operate and still only result in a limited data set. Multiple empirical models to describe irradiation creep have been developed, and the M^2 model is the newest and appears to predict creep strain well for the conditions tested. However, this model was only evaluated over a limited temperature range and required several additional property values.

5.2 Oxidation

Much attention has been paid to the rate of graphite oxidation because of the corresponding effect on material performance, the deterioration of the physical and thermal properties of the graphite essential to safe reactor operation. Oxidizing species (including molecular oxygen, steam, and carbon dioxide) may be present at contaminant levels throughout routine operation or at high concentrations following an off-normal event. Oxidation of graphite converts the solid atomic matrix of carbon (via reaction at active sites at edges and defects in the stacked graphene planes) into gaseous products (primarily carbon dioxide and carbon monoxide, depending on the local conditions and oxidizing species) [126, 127].

The oxidation rate is commonly inferred from the corresponding change in measured weight. This loss of solid material is initially limited by the accessible active sites but accelerates with the initial extent of reaction as pores, increasing in size and connectedness, allow for more efficient gas exchange and exposure of more edges and defects making them accessible for reaction (rate varies with extent of reaction). Depending on the (temperature dependent) oxygen penetration depth, the rate stabilizes at a quasi-steady state where diffusion conditions and active site availability produce a nearly linear observed reaction rate (conditions approached during 5-10% mass loss of typical test specimens). If oxidation continues beyond quasi-steady state, the rate slows as the reaction sites become limited by the quantity of graphite available. For oxidation of an as-machined nuclear graphite specimen to ash, the graph of weight with time produces a (nominally) sigmoidal-shaped curve as exhibited in Figure 5-21 [128].

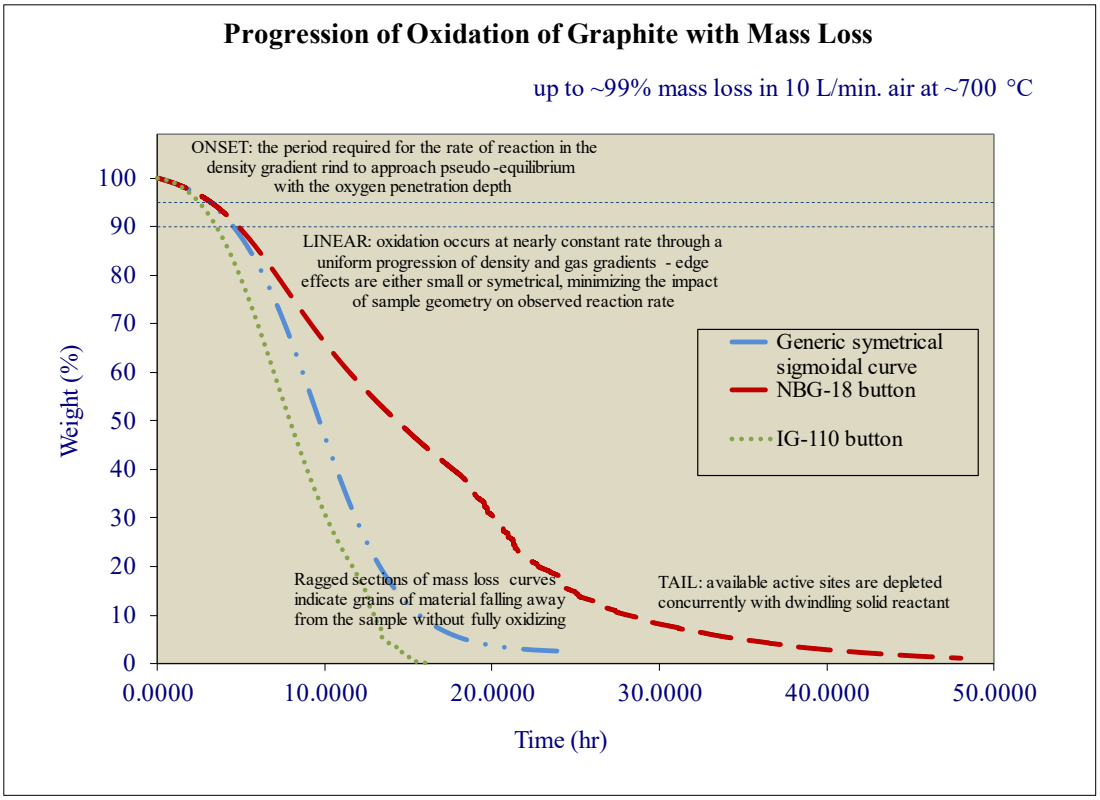


Figure 5-21. General progression of oxidation where weight is plotted over time producing a sigmoid-shaped curve [128]

Early testing indicated that oxidative mass loss of 10% resulted in loss of more than half the original strength of the material [129, 130, 131]. Subsequent development of the ASTM D7542 Standard focused on evaluation of rate over the 5% to 10% mass loss range to provide a conservative rate analysis relevant for comparison of graphite grades for use as structural nuclear reactor materials. Oxidation rate testing is seldom pursued beyond 15% mass loss where catastrophic strength loss is a foregone conclusion.

While oxidative mass loss degrades the graphite, many variables complicate performance modeling. Empirical evaluation supports an Arrhenius oxidation rate relationship with temperature, T, Equation [5-6].

$$rate = \mathrm{A}e^{-\frac{E_A}{RT}} \quad [5\text{-}6]$$

where *A* is a pre-exponential factor (accounting for combined effects associated with oxidant concentration and reaction order), E_A is an activation energy, and *R* is the gas constant. A semi-log graphical analysis enables calculation of apparent parameters from experimental data collected under conditions of consistent gas supply.

Approximated as three linear segments with slight bends joining them into a continuous curve, chemical kinetics determine low temperature rate behavior (Regime I), diffusion determines high temperature rate behavior (Regime III), and intermediate-temperature rate behavior is influenced by both chemical kinetics and diffusion over the transition (Regime II) [132]. This theory has been expanded to address parallel behavioral trends for oxygen penetration depth and for the density profile (envisioned as a cross section of an infinite slab in Figure 5-22) that develop (as colloquially described as an oxidation front) depending on both the environmental conditions and the graphite microstructure – the extrinsic and intrinsic factors in oxidation [128].

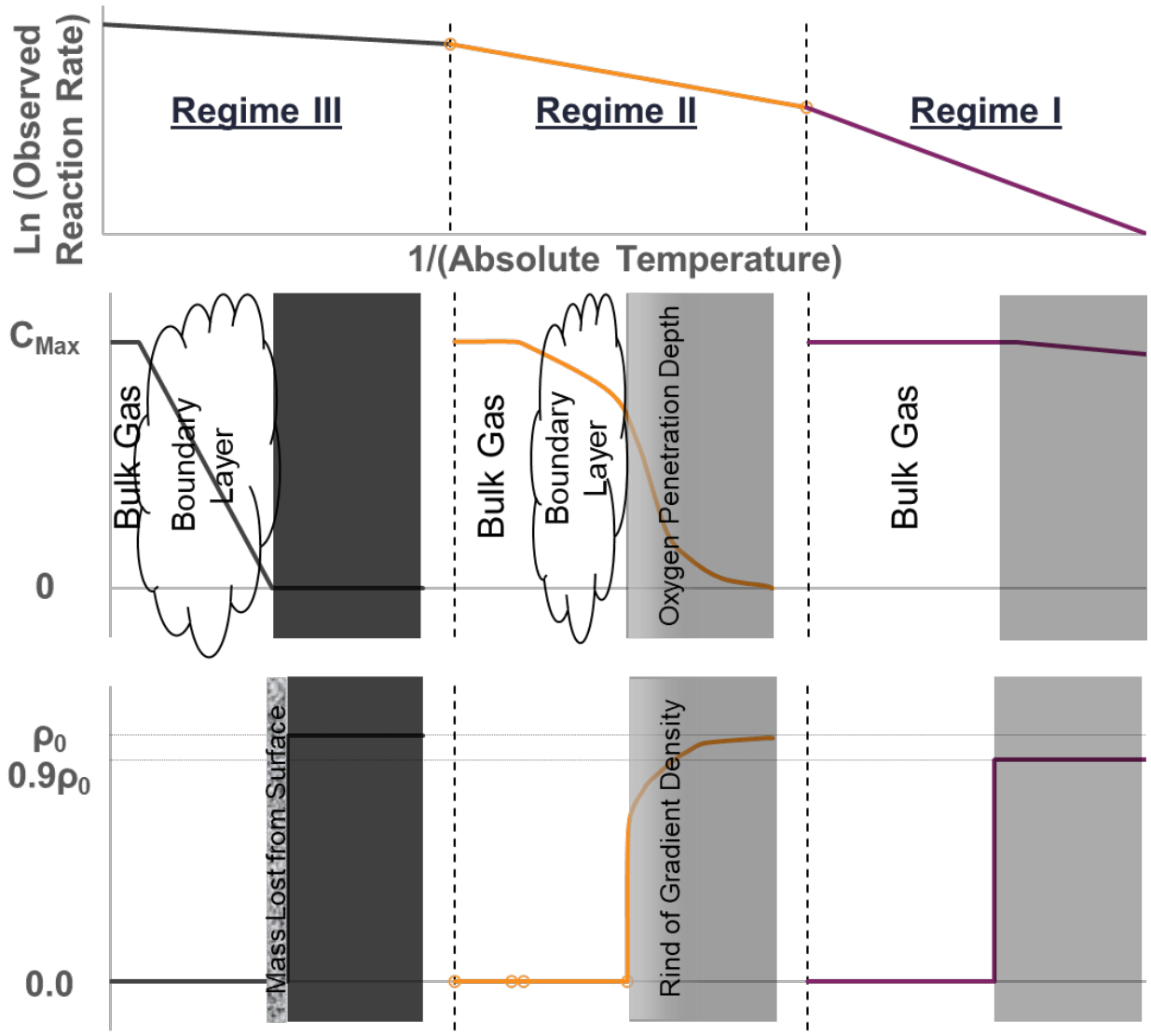


Figure 5-22. Arrhenius temperature dependence (top), oxygen penetration and diffusion (middle), and density profile (bottom) as correlate with the three-regime theory of graphite oxidation [128]

While observed rate behaviors generally fit this three-regime theory, the transitions between the regimes are sensitive to graphite microstructure and are therefore grade-dependent. More importantly, observed density profiles demonstrate the practical limitations on experimentally testing and modeling property changes with oxidation. Test specimens, having finite dimensions, may corrode nonuniformly demonstrating edge or corner effects specific to the locally evolving microstructure. Post-oxidation property testing produces inconsistent results for specimens exhibiting a pronounced density gradient (as expected of Regime II). High temperature oxidation (approximating Regime III) causes oxidation primarily from the specimen surface producing a geometrically smaller specimen with limited property changes to the remaining bulk material – the steep density gradient makes interrogation of changes in properties with mass loss difficult to correlate to local density or even to detect depending on the grade (Figure 5-23). However, specimens tested at low temperatures (approximating

Regime I) take a long time to attain the requisite measurable mass loss. And even impractically long tests (at 500°C) still produce specimens with detectable density gradients. Nonetheless, oxidation of test specimens at low temperature is the only way to correlate oxidative mass loss to degradation in material properties – creating test specimens with *relatively* flat post-oxidation density profiles enables correlation of property performance to that fraction of oxidative mass loss (as represented by the lower specimen density).

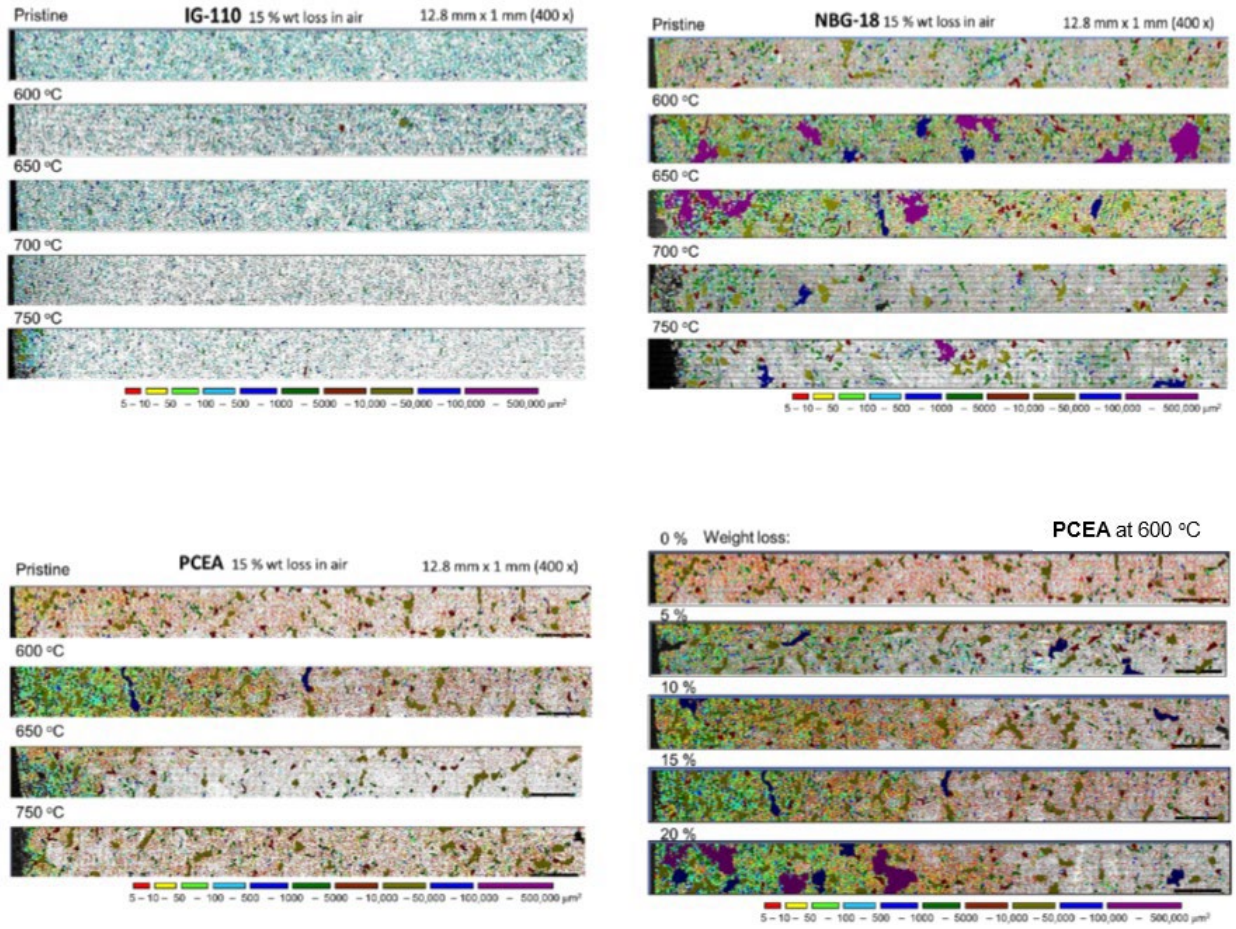


Figure 5-23. Optical microscopy images of subsurface porosity: IG-110, NBG-18, and PCEA graphite grades, pristine and oxidized in air to 15% weight loss at 600°C, 650°C and 750°C, and PCEA at 0%, 5%, 10%, 15%, and 20% weight loss in air at 600°C [217]

5.3 Molten Salt Issues

The new high-temperature reactor (HTR) designs being considered for future advanced reactor deployment include designs using molten salt as the primary coolant. These MSR designs provide several technological and safety advantages over the gas-cooled HTR designs, including lower operational pressure, higher thermodynamic efficiency, and smaller pressure vessel designs. However, there is minimal operational experience using molten salt in these nuclear applications, and the understanding of the potential material issues between the molten salt and graphite components is limited [11].

These molten salt–cooled graphite core designs pose new material compatibility challenges that are not considered within the gas-cooled HTR designs that have been previously built and operated. For example, initial results from the 1960s indicated that the fueled FLiBe used in the MSRE was chemically inert to the graphite internal core components [4, 133, 134]. However, more recent results revealed that some chemical interactions may occur [135, 136, 137]. Specifically, molten salt intrusion into the open pore structure of nuclear graphite grades can cause additional internal stresses within the microstructure, exacerbating the stress accumulation from irradiation-induced dimensional change. Additionally, designs using a molten salt–containing liquid fuel could produce hot spots within graphite structural components, causing local thermal stresses. Abrasion and erosion concerns are magnified with molten salt because of their extremely high density (some salts have higher densities than the structural graphite components). Finally, the graphite–graphite and fuel pebble–graphite tribological behavior are distinctly different within the molten salt from the inert gas environments and must be investigated.

This section describes graphite's properties and outlines the current molten salt–graphite material issues and ongoing research efforts to address these issues.

5.3.1 Nuclear Graphite Structure That Affects Salt Interactions

Graphite is an excellent neutron moderator, with intended applications in MSRs and fluoride salt–cooled HTRs (FHRs). Nuclear grade graphite is a manufactured composite material, fabricated from coke filler particles, a pitch-based binder phase, and a complex network of pores (Figure 5-24). The shape, size, distribution, and connectivity of the pores are functions of the manufacturing process. Most manufactured graphite has a density between 1.7 and 1.8 g/cm^3, and therefore a total porosity of about 20% [138].

Depending on the size of the grains or filler particles (which greatly affects the pore size), ASTM D8075 [139] classifies nuclear graphite into the following categories: coarse, medium-coarse, medium, medium-fine, fine, superfine, ultrafine, and microfine. Similarly, depending on the molding process, graphite properties may exhibit various levels of anisotropy, as defined in ASTM D7219 [140].

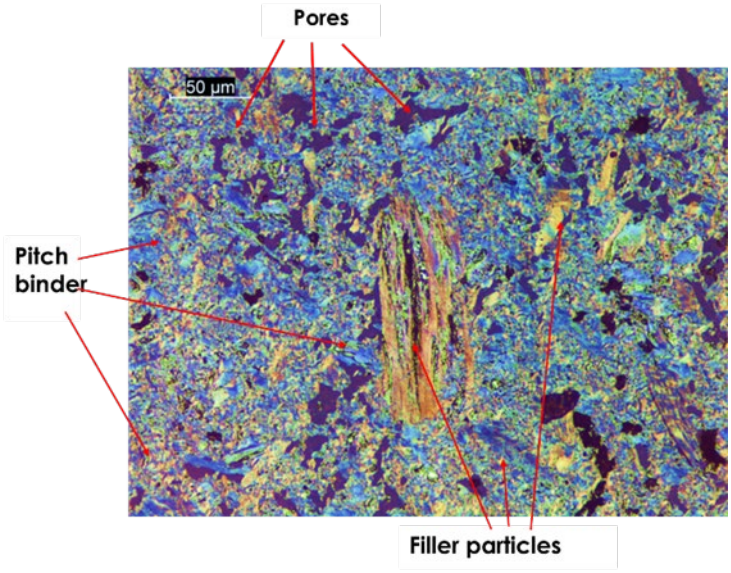


Figure 5-24. Optical microscope image showing the presence of filler particles, binder and porosity in nuclear graphite [49]

Graphite porosity plays a critical role in defining its neutron irradiation–induced material behavior (i.e., strength, stiffness, dimensional change). However, graphite porosity also provides channels for penetration of oxidant species and fission products, with undesired effects on mechanical, thermal, and neutronic properties. When exposed to molten salts (either clean or fueled), partial salt infiltration is expected to occur, causing (still not fully elucidated) additional complicated effects on graphite's structure and properties.

Nuclear graphite grades exhibit a wide range of porous structures. For example, Figure 5-25 shows the mercury intrusion and pore size distribution of two graphite grades with significantly different structures, pore size, and pore size distribution: PCEA (extruded, medium-fine grain) and IG-110 (isostatically pressed, superfine grain). The differences between graphite grades translate into different behaviors when exposed to molten salts.

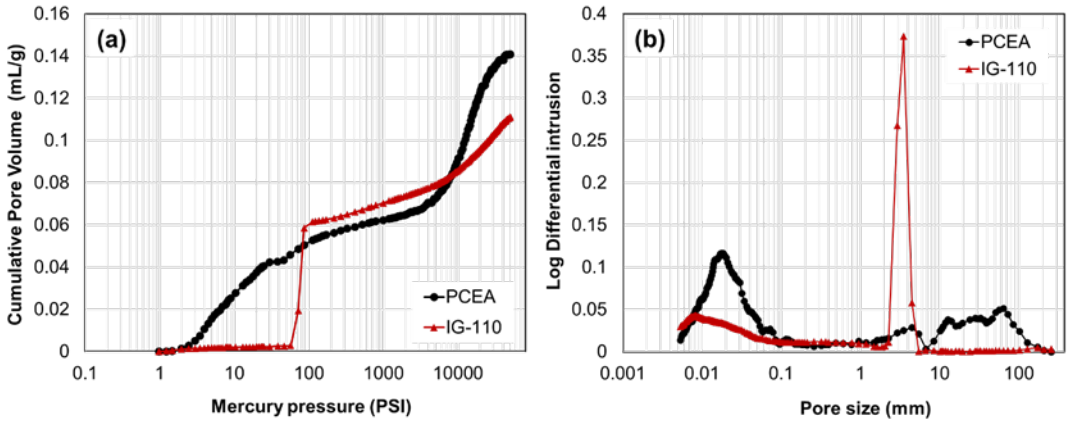


Figure 5-25. (a) Cumulative pore volume of mercury intrusion vs. mercury pressure for graphite grades PCEA and IG-110; (b) Log differential pore volume vs. pore size [141]

5.3.2 Graphite–Salt Interactions

When graphite is exposed to a molten salt at high temperatures and modest pressures, physical as well as chemical interactions may occur. Physical interactions may include salt intrusion, abrasion, erosion, and wear. Chemical interactions may include fluorination [139, 140] or tritium retention [45]. The effects of these interactions on the mechanical or thermal properties may affect graphite's performance in a reactor environment.

5.3.2.1 Salt Intrusion into Graphite and its Effects

Ongoing DOE-funded research focuses on quantifying and understanding molten salt intrusion into graphite's porous structure as a function of pressure, temperature, time, and graphite grade. Preliminary results have been documented in several publications [45, 142]. When it comes to salt intrusion into the graphite pore structure, the parameters *D0* and *Dt* as defined by the ASTM D8091 [142] do not provide a complete picture of the intrusion process. Therefore, understanding the effect of salt intrusion on graphite properties requires understanding salt penetration depth and salt distribution across the graphite component [49].

Recent efforts at ORNL have focused on using neutron imaging techniques for this purpose and have reported some preliminary results from the analysis of FLiNaK-exposed graphite samples that relate the infiltration behavior of several graphite grades with different microstructure [141]. Preliminary neutron imaging results for two graphite samples exposed to FLiNaK at 750°C and 5 bar pressure for 12 h are shown in Figure 5-26. The pore structures of the two graphite samples are shown in Figure 5-25. As shown in Figure 5-26, these two different porous structures lead to dramatically different salt distribution patterns between graphite grades PCEA and IG-110, although both samples were exposed to the same conditions of pressure, temperature, and time [141].

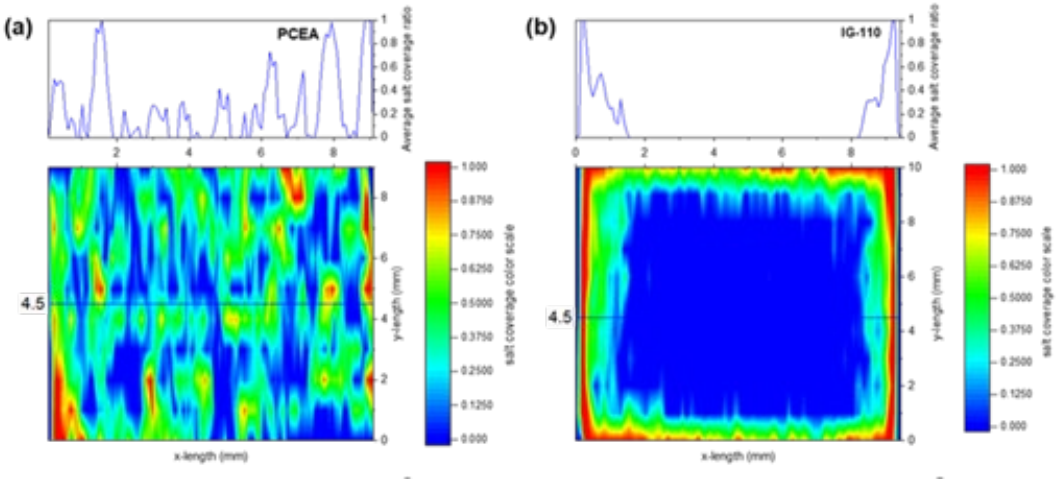


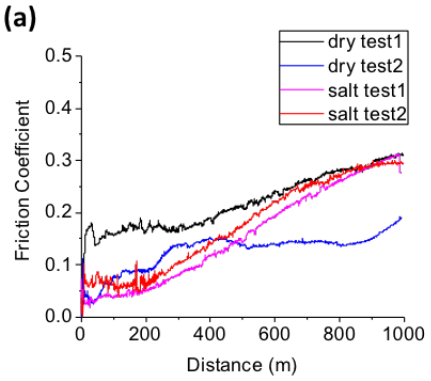
Figure 5-26. Preliminary neutron imaging results showing salt coverage comparison between graphite samples (a) PCEA; (b) IG-110 exposed to molten FLiNaK at 750°C and 5 bar for 12 h [141]

Molten salt intrusion into the open pore structure of nuclear graphite grades could generate additional internal stresses within the microstructure if subjected to cooling-heating cycles, exacerbating the stress buildup from irradiation-induced dimensional change. Understanding the salt intrusion behavior (penetration depth) as a function of intrusion conditions and graphite grade is a first step. However, the analysis of the effect of salt intrusion in graphite properties possess some challenges, such as determining whether testing should be done at room temperature (where salt is solid) or at higher temperatures (where salt is liquid) or whether the salt should be removed after exposure and before testing. An analysis of these challenges has been summarized elsewhere [49].

5.3.2.2 Graphite Wear, Erosion, and Abrasion in Molten Salt

In addition to salt intrusion and its effects on graphite properties, wear, erosion, abrasion, and degradation caused by physical and chemical interactions must be fully understood. The unique physical and thermal considerations from the salt coolants may affect the safe operation of an MSR design. Furthermore, the graphite–graphite and fuel pebble–graphite tribological behavior are distinctly different within the molten salt and inert gas environments and must be investigated.

Preliminary results shown in Figure 5-27 indicate a significant difference between the tribological behavior of a graphite pin against a stainless-steel flat surface when immersed in molten FLiNaK versus similar materials in an inert environment (dry conditions) [45]. The dry sliding test results are used as a baseline to understand the chemical (i.e., corrosion) and mechanical (i.e., lubrication) effects of the molten salt. In dry sliding tests, the graphite pin experienced wear loss (i.e., abrasion), whereas the stainless-steel square showed material deposition on the contact area. The deposit was later identified as carbon-based, likely material transfer from the graphite pin and accumulation of graphite wear debris.



(b)

Wear volume (mm^3)	Pin	Disk
Dry test 1	0.32	(0.154)
Dry test 2	0.14	(0.113)
Average	$0.23^{\pm 0.09}$	$(0.134^{\pm 0.21})$
Salt test 1	0.29	0.057
Salt test 2	0.53	0.041
Average	$0.41^{\pm 0.12}$	$0.049^{\pm 0.008}$

** "( )" represents deposit*

Figure 5-27. (a) Friction coefficient traces of the graphite pin against 316L Stainless Steel in dry and fluoride salt at 650°C; (b) wear volume on the pin and disc after cleaning [45]

In the case of sliding in the molten salt, the initial value of friction coefficient was lower than that measured in dry sliding, likely because the liquid salt at the pin-disc interface reduced adhesion. Unlike in dry sliding, the stainless-steel square had wear loss in the molten salt sliding possibly

caused by a combination of abrasion and corrosion. The graphite pin wear in molten salt was more extensive than that in dry sliding, possibly because the wear-roughened stainless-steel surface became more abrasive and/or the graphite pin's mechanical properties degraded in molten salt. Additionally, reactor designs using a molten salt-containing liquid fuel could produce hot spots within graphite structural components causing local thermal stresses. The abrasion and erosion concerns are therefore magnified with molten salt because of their extremely high density (some salts have higher densities than the structural graphite components).

5.3.3 Summary

The new molten salt–cooled HTR designs being considered for future advanced reactor deployment pose new material compatibility challenges. Although historical results from the MSRE demonstrated minimal chemical degradation between graphite and molten salts, several novel physical, chemical, and thermal issues are considered significant and are currently under investigation by the nuclear graphite community. Many of the phenomena being investigated stem from nuclear graphite's large pore defect microstructure, which can provide opportunities for salt intrusion into the interior microstructure. Unfueled (clean) molten salt intrusion may increase the internal stress distribution, whereas fueled salts may induce localized hot spots. Chemical interactions may include fluorination, intercalation, or tritium absorption. Finally, because of the high density exhibited in molten salts, issues surrounding abrasion, erosion, and wear in core components are concerns.

5.4 Other Issues

Mechanical fatigue is an additional degradation mode in nuclear graphite. The crack growth rate in graphites under low and high loading rates has been measured using various test specimen geometries in previous studies [143, 144, 145, 146, 147]. Although, of these studies, the cyclic loading rate considered *low* was a cycle every ca. 50 s [148]. Shaw and Bacon found a decrease in crack propagation rate with reducing loading rate [148]. Additionally, fatigue conditions in the cited literature do not include tests on irradiated nuclear graphites with higher modulus values [92]. It should be noted that loading cycles far exceed any mechanical fatigue conditions which are anticipated to occur in HTR designs; therefore, fatigue studies on nuclear graphite have been limited over the past decade.

With the progress in designing the next-generation HTRs, the question of what effect thermal fatigue has on graphite has arisen. Depending on the design, the next-generation HTRs are anticipated to operate at high temperatures ranging from 400°C to 1200°C. Additionally, large temperature gradients may exist within graphite components themselves. Periodically shutting the reactor off for refueling, maintenance, etc., creates the scenario of thermal fatigue. Over time, thermal fatigue could generate stresses within the graphite component. The effect thermal fatigue may have on graphite's properties and possible design constraints remains an open question.

6 ASME GRAPHITE CODE RULES

6.1 Status of Applicable Codes and Standards

6.1.1 Code of Federal Regulations

While the Code of Federal Regulations (CFR) is not discussed in this document, the following documents are required for operating nuclear power plants:

- 10 CFR Part 50 – Reporting of Defects and Noncompliance
- 10 CFR Part 21 – Quality Assurance Criteria for Nuclear Power Plants and Fuel Reprocessing Plants

These documents do not specifically mention graphite, but a reactor employing graphite core components will need to consider these documents when considering NRC required reporting (10CFR21) and quality assurance requirements (10CFR50).

6.1.2 Nuclear Regulatory Commission

- RG 1.87 Rev 2 – Acceptability of ASME Code, Section III, Division 5, "High Temperature Reactors"

Regulatory guide 1.87 is currently the only guidance from the NRC on the use of graphite core components in high temperature reactors. This guide provides the applicability of the different sections and articles of the ASME BPV Code Section III, Division 5 as reviewed by the NRC to ensure the integrity of components operating in high temperature reactors. This regulatory guide includes the applicability of the ASME BPV Code sections that provide guidance on graphite core components.

6.1.3 ASME

The ASME Boiler and Pressure Vessel Code Section III, Division 5 provides the following subparts concerning graphite use in nuclear reactor cores:

- HAB – General Requirements, Subpart B – Graphite and Composite Materials
- HHA – Class SN Nonmetallic Core Components, Subpart A – Graphite Materials

These rules discuss the design and construction of graphite core components and reactor cores containing graphite core components. These subparts are currently under discussion by ASME Code Committees in order make the qualification process of graphite core components more streamlined and accessible.

The ASME Code rules aim to be comprehensive in their rules for graphite core component consideration in nuclear reactors. These rules include classification of components, QA program requirements, certificates and data reports, materials, design, machining, examination, testing, and installation.

The ASME Boiler and Pressure Vessel Code Section XI, Division 2 is currently being discussed among the ASME Code Committees in order to include RIM programs for graphite core components. Issues related to RIM of graphite core components is discussed in Section 8.4.

6.1.4 ASTM

- D7219-08 – Standard Specification for Isotropic and Near-isotropic Nuclear Graphites
- D7301-08 – Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose
- D8093 – Standard Guide for Nondestructive Evaluation of Nuclear Grade Graphite
- C781-20 – Standard Practice for Testing Graphite Materials for Gas-Cooled Nuclear Reactor Components

These standards, and other practices and test methods referenced within them, are the ASTM specifications for nuclear grade graphite. Standard specifications D7219-08 and D7301-08 are specifically reference in the ASME BPV Code Section III, Division 5 as the 2008 versions, and are listed here as such. These specifications are discussed throughout this document. Note that in some cases, the user should refer to the more recent versions of these standards, which include clarification of some requirements (e.g., some requirements are given without units in the 2008 versions but have been updated to include units, with the same values, in more recent versions).

Standard C781-20 provides a list of standards and practices that provide materials properties for graphite components. Table 6-1 provides the standards pertinent to nuclear grade graphite manufacturing as well as properties measured and the applicable temperature range.

Table 6-1. ASTM Standards concerning as manufactured graphite listed in C781-20

Title of Standard	ASTM Code	Property Measured	Temperature Range (°C)
Standard Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance	C747-16	Stiffness - sonic resonance	-75 to 2,500
Standard Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining an Approximate Value of Young's Modulus	C769-15	Stiffness – sonic velocity	Not defined
Standard Test Method for Poisson's Ratio at Room Temperature	E132-17	Stiffness – Poisson's ratio	RT
Standard Test Method for Tensile Stress-Strain of Carbon and Graphite	C749-15	Tensile Strength	≤2,400
Standard Test Methods for Tension Testing of Carbon and Graphite Mechanical Materials	C565-15	Tensile Strength	Not defined
Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature	C651-15	Four point bending test	RT
Standard Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature	D7972-14	Three point bending test	RT
Standard Test Method for Compressive Strength of Carbon and Graphite	C695-21	Compressive strength	RT
Standard Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite	D8289-19	Diametral compressive strength (disc splitting)	RT
Standard Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature	D7779-20	Fracture toughness	RT
Standard Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air	C1179-15	Oxidation mass loss	371 to 677
Standard Test Method for Air Oxidation of Carbon and Graphite in the Kinetic Regime	D7542-15	Oxidation Kinetics	500 to 750

6.1.5 International Considerations

The discussion in this report is primarily focused on the US. With respect to technical issues, all of the gaps identified herein are, of course, applicable in other jurisdictions. However, this report also addresses code and regulatory issues. With respect to other jurisdictions, the following items are noted:

- In many jurisdictions, the regulatory body requires designers to follow the requirements of the country in which the designer is based. For example, a designer primarily based and holding an existing design license in the U.S. would be required to follow U.S. practices in constructing and operating a plant based in such a jurisdiction. This usually applies to jurisdictions with relatively limited investment in nuclear energy.
- In the UK, there is extensive experience with operating reactors with graphite internals. This situation is discussed in Section 6.1.6.
- Several jurisdictions participate directly in the work of BPVC committees and working groups. For example, the Task Group on Non-metallic Component Degradation and Failure Monitoring (which supports consideration of RIM for graphite) includes active members from the UK. Similarly, the Working Group on Non-metallic Design and Materials (which supports ASME BPVC Section III activities) includes members from Canada, the UK, China, Korea, and South Africa.
- Some jurisdictions have a domestically-developed independent code that provides similar content to the ASME BPVC. In many instances, the exact requirements of these other codes are different from the ASME BPVC even though the ultimate objective (safe operation) is the same. In some cases, these codes align very closely with the ASME BPVC (e.g., the JSME code in Japan), while in others the code has been developed essentially independently of the ASME BPVC (e.g., the French RCC-M code).

Comparisons among various codes [149, 150] have been made, but these have mostly focused on rules for metallic components. No comprehensive comparison has been made for graphite.

6.1.6 EDF Energy, UK

The UK Atomic Energy Authority (UKAEA) developed gas-cooled reactors with graphite internals starting in the 1940s. Currently, the Advanced Gas-cooled Reactor (AGR) fleet in the UK is own and operated by EDF Energy. The graphite components in these reactors are currently operating with significant degradation requiring substantial monitoring and evaluation efforts to maintain confidence that the reactors are operating safely. The situation is analogous to the status of PWR steam generator tubing in the 1980s, PWR Alloy 600 pressure boundary materials, or BWR sensitized stainless steel piping, i.e., safe operating is maintained through the implementation of an examination and evaluation program requiring periodic inspections. This is essentially another example of a RIM program (see Section 8.1). The situation may be considered particularly analogous to PWR steam generator tubes in that the components of interest is important to long-term economical and safe operation but is not actually part of the pressure boundary.

The UK experience is being broadly leveraged in the development of the ASME BPVC for graphite, primarily through the participation of AGR graphite experts on various committees and task groups of the ASME BPVC.

One important difference between the implementation of RIM in the UK for AGR graphite components and the implementation of RIM for PWRs and BWRs in the U.S. is that a review by the UK regulator of outage inspection results and analysis is required before restart of the reactor. Such a restriction has not been the standard in the US.

6.2 HHA Description and Basis Documents

Similar to the rules for metallic components, BPVC Section III, Division 5, "High Temperature Reactors" is structured to provide a central location for all aspects of construction for high-temperature reactors, including nonmetallic components or, more specifically, Class SN components, which include graphite component and assemblies. According to ASME code terminology, *construction* includes all aspects of the following:

- Materials (HHA-2000)
- Design (HHA-3000)
- Machining, Examination and Testing (HHA-4000)
- Installation and Examination (HHA-5000)
- Nameplates (HHA-8000).

The rules stipulate details for material specifications (HHA-I), the requirements for preparing a material data sheet (HHA-II), and the requirements for generating design data for different graphite grades (HHA-III). It also gives reference guidance for consideration of factors such as graphite as a structural material (HHA-A) and the environmental and oxidation effects in graphite (HHA-B).

The general requirements under BPVC Subsection HAB complement the technical rules, as Subsection HAB details the rules for classifying graphite core components (HAB-2000), the responsibilities and duties during the design and construction process (HAB-3000), the quality assurance aspects (HAB-4000), the Authorized Inspection requirements (HAB-5000), the applicable standards (HAB-7000), and the required certificates and reports (HAB-8000). It also provides a glossary of highlighted terms (HAB-9000). HAB-1130 specifically states the limitations of the rules to include consideration of deterioration that may occur because of radiation effects and oxidation.

Despite the similarities in code terminology, it was from the outset clear that the differences between nuclear graphite and ferrous metals would influence the processes required for qualification and design.

A background document, titled “Background Information for Addressing Adequacy or Optimization of ASME Section III, Division 5 (2017 edition) Rules for Nonmetallic Core Components” was published by ASME in 2021 [151]. The purpose of this document was to provide background information on the scope, development, and verification of elevated-temperature design and construction rules as defined in ASME BPVC Section III, Division 5 Subsection HH, Class A Nonmetallic Core Support Structures, Subpart A Graphite Materials, 2017 edition. This document together with a study performed by Hindley [152] was used to provide the basis of the methodology and verification of the full assessment that was implemented in Subsection HHA. Additional work was performed to define the process zone volume parameter together with the stress range parameter to accommodate fine grain graphite (not considered in the original development of the code). This work remains a hot topic in the graphite design community as the changes introduced some additional conservatism for medium grain graphite while addressing the concerns with ultra-conservatism affecting fine and super fine grain graphite.

6.3 Code Issues Being Addressed

The following sections discuss ongoing work by Code committees to address unresolved issues. These issues can be roughly divided into the following categories:

- Design
- Definition of Component Failure
- Irradiation
- Oxidation
- Molten Salt and Abrasion
- Other Degradation Issues

6.3.1 Design

6.3.1.1 Background

Graphite core components can be qualified using one of two probabilistic approaches outlined in HHA-3000, or through design by test. Per the “spirit of the Code”, the simplified assessment is meant to be simpler to use and more conservative than the full. There are several differences between the full and simplified assessments, one being that the material strength distribution in the simplified assessment is described using a 2-parameter Weibull distribution (0-threshold) and the full assessment is described using a 3-parameter Weibull distribution (non-zero threshold). The difference in strength distributions in the two assessments is one aspect that contributes to the difference in relative conservatism, since all stresses lower than the threshold are assigned a 0-probability of failure. Several issues pertaining to the Code interpretation and methodology have been identified and lack of background documentation describing why current Code rules are in place have made the process of addressing the issues

more complicated. Part of the effort is to provide the background documentation for why the Code works the way it does and the safety implications. While substantial progress has been made on several of these topics, results have not yet been published and therefore, this section describes the issues that are being addressed, not the solutions. In this respect, all of these issues may be considered gaps.

6.3.1.2 Data

Three data sources are primarily being used in the review of the HHA-3000 design sections:

- The Baseline Graphite Characterization (BGC) Program data come from the graphite unirradiated material property testing program of the DOE-ART Graphite R&D Program [153]. The Baseline Program provides a large database of material property data for NBG-18, NBG-17, PCEA, 2114, and IG-110 graphite grades with four simple geometries: dogbone (rod, no notches), cylinder, bending bar, and splitting disks. A summary of baseline program data can be found in Reference [154] and in other documents cited in Reference [155].
- Hindley's [156] original NBG-18 dogbone and sixteen variations of the bending bar with notches.
- Kanse, et. al [157] fine graphite grade (unspecified) dogbone, cylinder, and bending bar geometries with various volumes.

The advantages of these three combined data sources are: diverse graphite grades and various component volumes. The limitations are that there are only geometries with more complex stress states for one grade, NBG-18, with only summary experimental data.

6.3.1.3 Simplified Assessment

Issues related to the simplified assessment are centered around inconsistent and unclear language in the Code and concern that the simplified assessment could be less conservative than the full assessment in some cases.

The simplified assessment requires two checks, a membrane stress check and a peak stress (or peak equivalent stress, subject to interpretation) check. The Code is inconsistent in that it defines the membrane stress as both the average of the normal stress along a ligament and the average value across the thickness of a ligament or other section of the combined stress, prior to the determination of the equivalent stress. The Code is also inconsistent, in that the *peak stress* is also defined as the *peak equivalent stress*, or the highest equivalent stress computed from the total stress, which is the sum of the stress contributions from the normal stress. The peak equivalent stress would be derived from the maximum deformation energy (MDE). For more details, see Reference [158]. The simplified assessment is intended to be easier to perform than the full assessment, which will only happen if MDE is used versus the normal stress.

The two checks in the simplified assessment permit two different allowable limits. The membrane stress check is the 10^{-4} lower bound allowable tensile stress limit. The peak stress check is applied to the ratio of the mean flexural strength to the mean tensile strength (R_{tf})

times the allowable stress. This effectively raises the limit to being a 10^{-4} lower bound allowable flexural stress limit [159]. If the peak equivalent stress based on MDE was applied to a 10^{-4} lower bound allowable tensile stress limit, the simplified assessment would always be more conservative than the full assessment. There is concern that there may exist cases where the full assessment is more conservative than the simplified with the inclusion of the R_{tf} factor.

To address these issues, an ASME BPVC task group is evaluating the relative conservatism of the use of the normal stress versus the MDE. Once terminology is agreed upon, the limits and their conservatism relative to the full assessment can be evaluated.

6.3.1.4 Full Assessment

The following subsections discuss elements of the full assessment that are being evaluated.

6.3.1.4.1 Distribution Choice

The distribution for the full assessment has not yet been determined.

6.3.1.4.2 Threshold Reduction

The threshold is first determined using maximum likelihood estimation (MLE). There is a step in HHA-3217 that prescribes that the threshold be reduced by a factor of the maximum equivalent stress (*max(es)*) divided by the lower bound of the characteristic stress $\left(\hat{S}_{c_{0.05}}\right)$, when *max(es)* < $\hat{S}_{c_{0.05}}$. The following two issues pertaining to the threshold reduction are being addressed:

- There is no documentation as to why the threshold is reduced, though several hypotheses exist. *Threshold reduction* is not a statistical, nor a typical Weibull analysis step. It seems to be an *engineered step* specific to the full assessment, with no theoretical basis. Hypotheses for the basis of the reduction include the following:
  - It is documented [35K sleeve bursts] that it takes many samples to accurately characterize the left tail of a graphite strength distribution and that, though the Weibull distribution is the best bi-modal distribution to do so, it may be under-conservative at low stresses. If too much of the component is at a low stress, the threshold must be reduced to assign more low stresses non-zero probabilities of failure (POFs).
  - The shape and characteristic stress lower bounds are used in an attempt to be conservative. The Code does not prescribe use of a lower bound on the threshold parameter, though this is statistically easy to obtain, so it would make sense to use a threshold lower bound versus an arbitrary reduction.
  - The shape parameter depends on the threshold parameter. The Code prescribes that the shape parameter not be updated when the threshold is reduced. The effect is that the left tail of the reduced-threshold Weibull distribution is more conservative than even the 2-parameter Weibull distribution, again giving low stresses an even higher POF.

- With respect to the relationship between the shape parameter and the threshold parameter discussed above, the Code does not prescribe updating the shape parameter when the threshold is reduced. This poses one main issue:
  - When the shape is not updated, the resulting 3-parameter Weibull distribution is more conservative in the left tail then even the 2-parameter Weibull distribution, which can make the simplified assessment more conservative than the full assessment. This is documented in Reference [156]. Work presented at the May 2023 ASME Design Task Group meeting displayed the added conservativism with the acceptance of the 2021 Vm grouping rules for NBG-18 [160].
  - Designers can go back to the original data to re-estimate the Weibull shape parameter using MLE. Two other articles have been identified which offer alternative equations to re-estimate the Weibull shape parameter without going back to the original data. However, the most accurate and consistent method is to use MLE. [161, 162]

6.3.1.4.3 Tuning the Minimum Link Volume (V_m) and the Relative Stress Range (Δ) Parameters

V_m sets a minimum link volume requirement for groups of finite elements (FEs) and $\Delta = \frac{\max(\sigma_{vi}) - \min(\sigma_{vi})}{\min(\sigma_{vi})}$ sets a minimum stress range requirement for groups. The original HHA-3217 Code rules through 2019 (including what was endorsed by the NRC in the 2017 Code version) contained grouping requirements of: $V_m = (10 * grain\ size)^3$ and $\Delta = 0.07$. The basis for using a minimum of 10*grain size is found in Reference [163]. In 2021, the grouping requirements were changed to $V_m = \left[\frac{\pi}{2} * \left(\frac{K_{IC}}{\sigma_m}\right)^2\right]^3$ and $\Delta = 0.2$. Several questions and concerns have arisen and are being addressed related to the grouping criterion.

- There are several reasons V_m makes sense as a requirement, a few of which are:
  - Computational: If V_m did not exist, each element would be its own group, drastically over-estimating the probability of failure (POF) of a component.
  - FE mesh refinement: Likewise, mesh refinement would have a large effect on POF if elements were not grouped according to volume.
  - Graphite material properties: Graphite strength begins to increase with volume after a certain minimum volume, and then begins to level off [164]. Additional work related to the volume-strength effect of graphite components is found in [165]. Volumetric strength changes in uniform tension stress needs to be examined to see whether that aligns with weakest link theory [166, 167, 168, 169, 170].
- Hindley [157] performed the original tuning of V_m. That work was only performed for NBG-18, a medium grain graphite, but the V_m Code guidelines, based on that work, were developed for all grain sizes. This made the original V_m overly conservative (too small) for fine grain graphites. Work presented in Reference [171], suggested a change from a grain-size based minimum link volume to a process zone/fracture toughness minimum link

volume would solve this problem. The task group discovered the wrong equation was used (for correct equation see Reference [172]). However, the corrected equation did not solve this problem.

- Furthermore, while the (wrong) 2021 V_m equation made resulting POFs less conservative for fine grain graphites, it added margin for the medium grain graphites, which was undesirable for some vendors.

The task group's approach to solving these issues was to replicate the work by Hindley to tune V_m for NBG-18 using most of the geometries in his original work [156] and the BGC data for NBG-18 to replicate his work. Following this, the methodology was expanded for other graphite grades to develop trends. Initial results are promising and are consistent with the weakest link theory but have not been published yet. There is no documentation as to why Δ exists, and that is one item that is also under investigation.

6.3.1.4.4 Mesh Refinement

There were concerns that mesh refinement could affect the simplified and full assessment acceptance decisions. While interpretation of the simplified assessment is still not agreed upon, results in Reference [173] demonstrate that mesh refinement did not affect full assessment results and that mesh refinement could affect simplified assessment results but did not in the study at hand. The study at hand is limiting in that it only uses simple geometries, and it suggests repeating the study when Code rules are agreed upon for geometries in more complex stress states.

6.3.1.4.5 Location

Currently, the grouping criterion of the full assessment is such that non-proximal elements may form the same group. Elements are ordered by decreasing stress, and are grouped that way, making all the high stress elements form one group. This is appropriate for the weakest link theory, since it is the one group with the highest stress that causes failure. However, alternative frameworks being explored (consideration of multiple groupings with similar stress) are not compatible with the current criterion. For this next step, it would be more appropriate to have multiple groups with high stressed regions that all have a high probability of leading to fracture.

6.3.1.4.6 Sample Number Requirements

Currently HHA-III-4100 requires sampling for isomolded materials from at least three lots, with four billets within each lot. Samples are taken from slices from the top, middle, bottom of the billet. In each slice, samples are taken from the center and the periphery. Two slices are with and two against the grain sampled in each quadrant to create specimens which form the tensile strength distribution (3x4x3x2x4 = 288 total specimens). While this seems to be a fairly representative sampling requirement, the question was asked as to whether these are enough samples to achieve a converged variance on the Weibull parameters and which source (lot, billet, location within billet) is contributing to the most tensile strength variation. Current work has not been published yet but was based on density-issue PCEA billets as a worst-case

scenario. Current work is limiting in that it does not contain specimens from the required three lots. Effort has been made to reach out to manufacturers to obtain more data but has not progressed further.

6.3.1.4.7 Margin

The full and simplified assessments are to provide conservative design allowable loads. However, there has been no work to meaningfully quantify (in terms of load), how conservative the assessments are as they stand, nor relative to each other, for various graphite grades. The DOE plans to publish a paper on this in the near term.

6.3.2 Definition of Component Failure

Subsection HHA of Section III Division 5 of the ASME code rules addresses the design of graphite components and assemblies in High Temperature Reactors (HTRs). The code provides assessment methodologies based upon design targets for components categorized in terms of probabilities of failure. Failure here refers to a component passing or failing the assessment, not whether the component has failed in terms of functionality. The use of the term *failure* must be used with extreme care. In practice, a component is deemed to have *failed* if it no longer meets its design function. Design function or functionality relates to the nuclear safety requirements for that component and the level of damage tolerance in the design of the assembly. The text of this subsection of the BPVC does not provide sufficient explanation of this terminology. Assessment methodologies used in the code compare evaluated stresses with Weibull strength distributions for graphite based upon mechanical tensile strength tests where the stress is uniform within the test specimen and where the stress during the test is increased until the specimen truly fails. In the case of an actual reactor graphite component, there will be non-uniform distributions of stress and geometries may be complex. The precursor to failure in this sense is crack initiation. Crack initiation in such a case may lead to crack propagation and component fragmentation, but cracks will only propagate if there is a stress driver within the component; cracks may arrest after formation. The tolerance of a component or assembly of components to crack initiation will depend upon the specific design of the plant and crack propagation dynamics. Some designs will not be tolerant to the initiation and development of cracking. However, there is plentiful data from operating plants that show that other designs are tolerant to extensive cracking within a component and to the presence of large numbers of cracked components within an assembly.

The simple and full assessment methods described in the ASME BPVC are fit for purpose as long as it is recognized that they address design constraints rather than representative behavior. Assessment outputs will be conservative and provide no information on component functionality or damage tolerance. Furthermore, if the design of components was based upon a crack initiation criterion rather than a design criterion, this may be perfectly acceptable but could also be highly conservative, thereby unnecessarily limiting the operational lifetime of the plant. To address these issues, text changes have been proposed that highlight these concepts and qualify the use of the term *failure*. The subtleties around the use of this term in the code require further discussion and clarification before a final text is submitted for ASME approval.

A supporting white paper has been published that illustrates the relationship between damage tolerance and design, based upon the UK experience with Gen I Magnox reactors and Gen II Advanced Gas-Cooled reactors (AGRs) [174].

Section XI Division of the ASME BPVC provides requirements for Reliability and Integrity Management (RIM) Programs for nuclear reactor facilities. Designers using Section III Division 5 of the ASME code need to be aware of these requirements as they could influence the plant design. Such RIM requirements allow plant operators to monitor the performance and behavior of components in the context of design targets discussed above. Currently, RIM requirements are specified only for metallic components in light water reactors (LWRs) and high temperature gas reactors (HTGRs). The omission of non-metallics has been recognized by both Section III and Section XI, and, to address this, a Section XI Task Group has been set up entitled *Non-Metallic Component Degradation and Failure Monitoring Task Group*. To provide RIM requirements for non-metallic components across a range of nuclear plant types is a challenging task. The task group, comprising participants from national laboratories, designers, vendors, and the U.S. regulator have selected graphite components in HTGRs as the first stage of a long and complex process. RIM requirements for specific reactor types are contained as supplements to an appendix to the Section XI document. It is important to note that Section XI requirements do not make any judgements on what may be observed. The supplements briefly describe degradation mechanisms and their potential effects on components. The requirements are not prescriptive but identify RIM options based upon degradation effects. Currently the first stage graphite supplement has been compiled and submitted for initial ballot and comment. Recognizing that a final fully-approved supplement may take several years to achieve and noting the need for designers to be alerted to RIM requirements, a parallel Code Case is planned that should progress through the ASME approval process in a more timely way.

6.3.3 Irradiation

Irradiation effects on graphite are considered by the ASME code in the following ways:

- Irradiated material properties.
- Irradiation effects.
- Stress calculations.

The material properties of irradiated graphite core components are required to be reported in the material data sheet for compliance with the HHA-3200 subpart. The material properties discussed are:

- Dimensional change
- Creep coefficient
- Coefficient of thermal expansion
- Strength
- Thermal conductivity
- Elastic modulus

These material properties are required to be report based on the irradiation design envelope for the qualification.

Irradiation effects discussed in the ASME Code pertain to the design envelopes of the graphite core components being qualified. The Code considers the fluence limits, the stored Wigner energy, the internal stresses due to irradiation, and graphite cohesive life limits. These are all considered in the context of the design of graphite core components.

Stress calculations involving the irradiation of graphite core components are discussed in the ASME BPV Code as well. This includes internal stress, combined membrane stress, and peak equivalent stress.

The effects of irradiation on graphite core components are discussed in Section 5.1.

6.3.4 Oxidation

ASME code rules pertaining to oxidation have been recently updated along the following four topic areas:

- Oxidants
- Types of oxidation
- Measurement of oxidation
- Effects of oxidation (degradation of component performance)

First, oxidants have been specifically defined to include molecular oxygen, water, and carbon dioxide, and to exclude hydrogen (which is not an oxidant and has been shown to impede oxidation by water) and to exclude radiolysis of carbon dioxide (a separate concern for reactors operated with carbon dioxide coolant, not relevant to HTR designs).

Second, the following two types of oxidation are explained in context:

- Chronic oxidation attributable to H_2O or CO_2 impurities in the coolant
- Acute oxidation resulting from O_2 (as from air ingress) or H_2O (as from steam ingress) displacing the helium coolant

Third, measurement of oxidation is expressed according to weight loss, which correlates directly with density and inversely with porosity. Weight loss is also understood as a local property with a density profile based on proximity to the exposed surface.

Finally, the degradation of performance is determined via weight loss in uniformly oxidized specimens. Degradation in graphite structures (property deterioration and geometry reduction) corresponds to scenarios that define the qualification envelope.

Additionally, ASME BPVC Section II Div. 5 Subsection HA offers two patently different guidelines for the stress evaluation of oxidized graphite components. Strength decreases as a function of weight loss, and the stress evaluation shall be made accordingly. The region where strength decreases to less than 50% shall not be credited. And the region where weight loss exceeds 30% shall be regarded as absent from the structure for both oxidation and strength calculations.

A review of source data and other literature indicates that weight loss of 10% in general corresponds to 50% residual strength (or less). These empirical strength data follow the Knudsen relationship, further substantiating that a region where weight loss exceeds 10% should be regarded as absent. The 30% weight loss value is not adequately supported and correlated by data, and therefore should be replaced by 10% for consistency and conservatism [126].

6.3.5 Molten Salt and Abrasion

The ASME Boiler Pressure and Vessel Code (BPVC) Section III Division 5 for HTRs addresses the rules for graphite core components in the general requirements [11]. The technical design and material requirements are addressed under subsection HA Subpart B (HAB) and Subsection HH Subpart A (HHA), respectively. The code was originally written for HTR gas reactors and did not consider the environmental effects of MSRs. However, according to Article HAB-1000, the graphite and composite rules are different from the metallics code in that it considers the deterioration from environmental effects that may occur when in service [175].

Therefore, the technical rules on design and material under Subsection HHA must address the in-service considerations relevant to MSRs. This requirement is partially fulfilled: the code considers the irradiation effects of graphite, but it circumvents any discussion related to graphite deterioration caused by coolant salt–graphite interaction. It dictates conditions for abrasion and erosion excessive to MSRs and does not accommodate salt coolant conditions (with or without fuel). Nonmandatory Appendix HHA-B provides some information on expected environmental effects and briefly mentions salt coolant–graphite interactions. It states that for salt–graphite interactions, salt intrusion into the graphite, porosity, buildup of tritium gas, and property changes should be considered. Moreover, additional consideration should be given to salt coolant that contains fuel because it has the potential to create hot spots in the graphite.

Despite being referenced in the nonmandatory appendix, the mandatory rules do not provide for any property changes or design consideration because of the salt–coolant interactions [175].

Ultimately, MSR designs are intended for commercial application. Therefore, licensing and applicable code rules must be considered. Unfortunately, specific code rules for these molten salt coolant designs are absent from the ASME BPV Code Section 5. A special Molten Salt Task Group has been formed within the ASME Working Group for Nonmetallic Design and Materials to establish any rules necessary to ensure the safe operation of these molten salt designs. All material issues discussed within Section 5.3 are being considered, and potential modifications to the code will be presented and discussed at future ASME meetings.

6.4 BPVC 2023 Code Rules

Since the NRC review on the 2017 edition of the ASME Section III Division 5, code development effort was spent on addressing the limitations that were called out in the Regulatory Guide [176]. Although not all changes have yet been implemented through the Section III Standards Committee, some are included in the revised 2023 edition These code changes as it relates to graphite, include:

- *Oxidation rule revisions*: The NRC did not endorse the provisions of HHA-3141(c) (2017 edition) that set the weight loss limit as 30% for geometry reduction in the oxidation analysis (see additional discussion of this issue in Section 6.3.4). It was also recommended that Figures HHA-3141-1 and HHA-3141-2 be reviewed for applicability in the context of reactor operating conditions and the dependence of strength on weight loss in uniformly oxidized graphite classes [177].

  As a result, an in-depth literature study was performed on graphite oxidation to validate the rules and several whitepapers were produced and published. The resulting effect was that several changes were implemented such as the Figures in HHA-3141-1 were removed and replaced in the nonmandatory appendix HHA-B-3000. It was also made clear that oxidative weight loss should be considered in uniformly oxidized graphite.
- *Simple assessment Weibull notation changes and replacement of Figures HHA-II-3100-1 and HHA-II-3100-2*: This was not specifically a topic raised by the NRC, but it was recommended to revise the original figures and include a table with properties as it was affecting calculation of the material properties in the data sheet. As a result, the notation changes were incorporated to address the Weibull calculations of the simplified assessment stipulated in HHA-3000 as well as HHA-II-3000 and the figures were replaced with revised figures supported by tables that will enable the designer to determine the pivotal quantities for the normalized upper bounds on the shape and characteristic stress.
- A concerned raised by the graphite community was the expense and the lack of standards to perform elevated temperature strength testing with graphite as part of the qualification process. It was not clear from the Material Data Sheet in HHA-II-2000 that high temperature strength testing was only required if the designer wants to take account of the strength increase because of temperature increase. Due to the expense and effort involve, this is usually not the designer's desire, but this rule was hidden in HHA-III-3000. To further clarify this point, it was also mentioned in the Notes of HHA-II-2000-1.

Not all changes mentioned were approved for the 2023 edition, but it is anticipated that the challenges mentioned by the NRC reporting will be addressed for the most part in the 2025 edition.

7 MARKET ISSUES

Assessment of deployment readiness requires an understanding of the current market status and future needs for nuclear graphite. Figure 7-1 shows a range of projections for new nuclear generating capacity in the U.S. Several changes have occurred in the energy industry since the projections in Figure 7-1, most notably the following:

- The Inflation Reduction Act will provide a $25/MWh tax credit for new nuclear for the first ten years of operation [178].
- The projections in Figure 7-1 do not account for electrification, i.e., the expansion of the total energy delivered through the power grid through phasing out other modes of energy transfer. This includes, for example, changing from internal combustion engine vehicles to electric vehicles or changing from natural gas to electric for residential heating. Electrification is expected to significantly expand the need for electricity. Some utilities are projecting an increase to two to four times their current production by 2050.

Based on the modeling results in Figure 7-1 and the above notes, new nuclear generating capacity for the U.S. is assumed herein to be 100 GW in a low-nuclear scenario or 1000 GW in a high-nuclear scenario.

Numerous advance reactor vendors are developing reactor designs. It is considered unlikely that all of these vendors will successfully bring their design to market. For the assessments performed herein, it is assumed that success would include capturing 2% of the projected market. Therefore, the amount of graphite needed to supply a particular vendor's design, if successful, is taken as the amount needed for enough units to produce either 2 GW (low-nuclear scenario) or 20 GW (high-nuclear scenario).

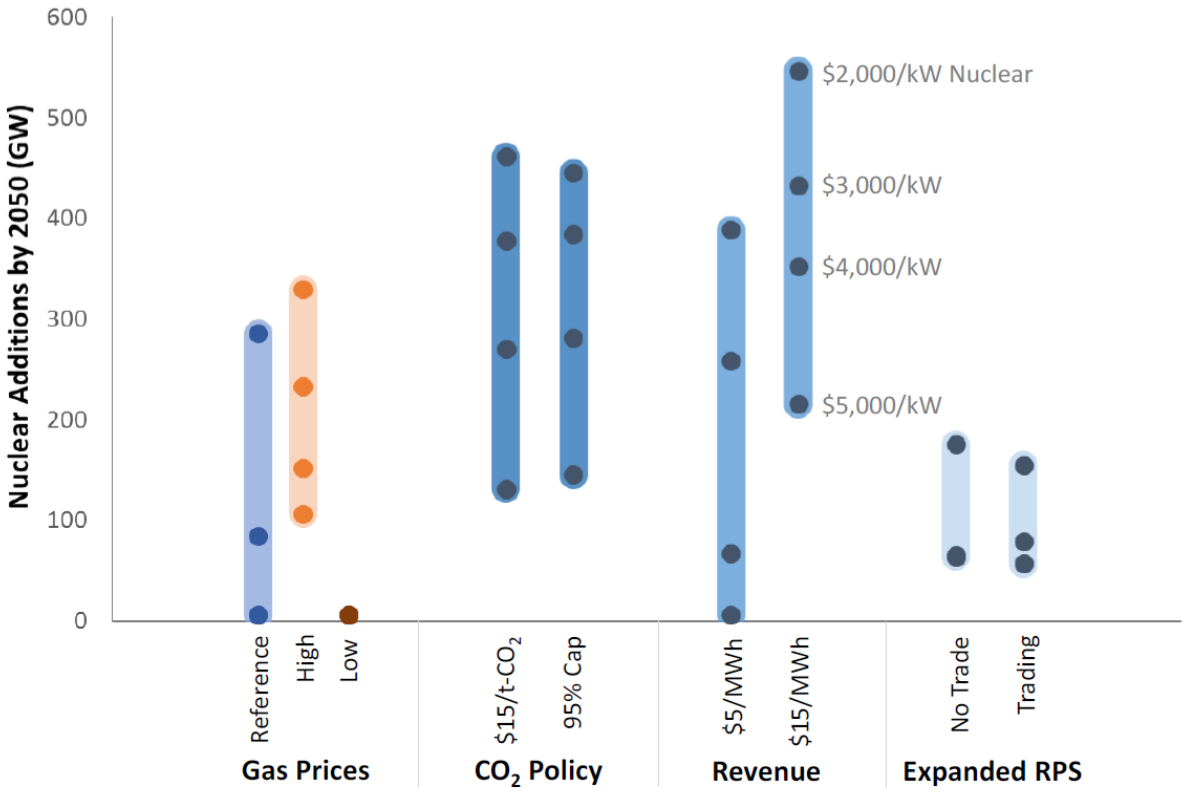


Figure 7-1. Projected new nuclear builds in the United States [179][1]

7.1 Producers (Graphite Suppliers)

Graphite is used in many different industries for manufacturing. Electrode manufacturing, battery manufacturing, refractory materials, and chemical manufacturing are examples of industries using graphite materials. With the current lack of impact from by nuclear on the graphite industry, few graphite manufacturers produce nuclear grade graphite as specified by the ASME BPV Code and the ASTM standards. As discussed in section 4 and section 6, the ASME and ASTM specifications for graphite are not as specific as that of a material like steel, which has more historic use in pressure vessels and nuclear reactors. Due to this, graphite manufacturers that produce nuclear graphite lack consistency between their grades, leading to differences in grain size, structural properties, chemical purity, and irradiation response among the different products offered by different suppliers (see extended discussion in Section 4.2). Additionally, graphite manufacturers are spread across the globe and acquisitions have led to a confusing connection among the different companies and the histories of various products.

[1] Note that the expanded RPS cases refer to the expansion of state renewable portfolio standards (RPSs), i.e., the inclusion of state-level incentives for nuclear power production through the classification of nuclear as a renewable energy source.

The following is a description of the different manufacturers located in the US:

- SGL Carbon has its U.S. headquarters in North Carolina, as well as multiple headquarters across Europe. SGL Carbon primarily provides graphite for the automotive, aerospace, energy storage, and semiconductor industries [180]. The American division of SGL originally started as Great Lakes Carbon, which developed the nuclear graphite grade H-451, which was considered a second-generation nuclear graphite for use in high temperature gas reactors (HTGR) in 1977 [181, 182]. This extruded, isotropic graphite was used in the Fort St. Vrain reactor and is no longer available [150]. SGL currently produces the nuclear grade graphites NGB-17, NGB-18, and NBG-25.
- Amsted Graphite Materials, located in West Virginia, produces nuclear grade graphite that was originally produced by Union Carbide Corporation and GrafTech International [183]. The nuclear grade graphite provided by Amsted Graphite Materials is PCEA, which is a historical grade of graphite that is being characterized by the BGC program at INL [35].
- Entegris Materials, located in Massachusetts, produces many products across many industries, as well as providing research and development solutions. Of these, graphite components are included with the grades POCO ZXF-5Q, POCO AXF-5Q, and POCO TM. These graphite grades are listed as industrial grade, but they are being tested in molten salt intrusion systems at ORNL for consideration in molten salt reactors [184, 32].

The following is a discussion of the different international graphite manufacturers:

- Toyo Tanso, based in Japan, produces graphite materials for a wide range of industries including semiconductors and industrial machinery. Toyo Tanso also provides the nuclear grade graphites IG-110 and IG-430, which are used in the high temperature test reactor (HTTR), the high temperature gas reactor-10 (HTR-10), and the high temperature gas-cooled reactor – pebble bed module (HTR-PM), which are test reactors for advanced HTGRs in Japan and China [185].
- Mersen is a company based in France that provides graphite products for the energy, electronics, and transportation sectors of manufacturing. The Mersen graphite grade 2114 has been used in the ORNL salt infiltration study, but it has not been used for much outside the semiconductor industry [45, 186].
- Tokai Cobex is a company based in Germany that produces graphite electrodes and cathodes for metallurgy and aluminum smelting respectively and specialty graphite advertised for the chemical industry [187]. The graphite grade G347A has been used in irradiation experiments in 2016 at ORNL to understand how it responds to possible advanced reactor environments [188].
- Nippon Graphite, based in Japan, does not directly support the nuclear industry, but the graphite grade IGS743NH has been used in ASTM testing to determine applicability and limitations of small graphite specimen testing. This testing was performed for understanding of the development and qualification of nuclear materials [189].

- Ibiden, a company based in Japan, produces products for the electronics, ceramics, and graphite industries. Their nuclear grade graphite ETU-10 has been noted by Kairos Power in their NRC submittal KR-TP-014-NP as a candidate for the KP-FHR design [190]. This grade has also been irradiation tested along with the IG-110 and NBG-25 graphite grades at the high flux isotope reactor at ORNL [191].

Of the grades discussed from the manufacturers above, all but IGS743NH from Nippon Graphite and G347A from Tokai Cobex are being considered in the Baseline Graphite Characterization program. This program, as discussed in earlier sections, intends to provide a database of information on the available nuclear graphite grades, including mechanical properties, irradiation response, and variability within graphite components, billets, and lots [44]. This program will help designers determine which graphite manufacturer is best suited for their reactor design.

7.2 Size of the Current Market

Currently the graphite industry is dominated by the production of electrodes and semiconductors. Advanced reactor vendors are not currently ordering graphite for reactor concepts, and as a result, the future size of the nuclear graphite market is difficult to determine. However, availability of graphite material can be estimated with statistics from the entire graphite industry. As nuclear-grade graphite is primarily synthetic, this estimation can be narrowed down to remove natural graphite from consideration. In 2021, the European Carbon and Graphite Association (ECGA) reported that the global consumption of synthetic graphite was 2.26 million tons [192].

Regarding the division of major market sizes by continent or region, it is once again difficult to determine. The Canadian government provides a breakdown of global synthetic consumption [193]. Asia dominates at 81%, followed by 9% in Europe, 8% in Southern America, 1% in North America, and 1% other. The massive Asian consumption can be attributed to the wide-scale manufacture of electronics such as electrodes, batteries, and semiconductors in this region that require graphite components and may not necessarily point towards a dominance in nuclear graphite. Another factor to consider is the location of the large suppliers of nuclear graphite, mentioned in Section 7.1, and where their clients are located. Additionally, the raw materials needed to manufacture synthetic graphite such as coke, pitch, and oil also influence the supply chain. In summary, there is a definite gap in information and data for the nuclear graphite market as a whole with more study required to estimate the true size and shape of the current market. This gap is exacerbated by the lack of concept reactor production by advanced reactor vendors. The primary reason nuclear grade graphite is being purchased currently is for research purposes and some graphite manufacturers discussed in Section 7.1 do not advertise their nuclear grades [35]. This leads to a lack of understanding of both the supply and the demand of nuclear grade graphite.

7.3 Industry Component Needs (Example Components)

Despite varying designs for different reactors, the typical anticipated graphite components fall into three categories: fuel, reflector, and support elements. Fuel elements are designed to be removable, with certain features engineered specifically to be handled by replacement machines. The primary purpose of fuel elements is to hold fissile material [24]. In most advanced reactor designs, TRISO fuel particles are used in either prismatic fuel blocks or spherical fuel matrices. These elements may also possess coolant and cool rod channels, structural alignment pins, shutdown material, and instrumentation. The main functions of graphite fuel elements, aside from containing the fuel and subsequent decay material, are moderating neutrons from fast to thermal spectrums to maintain the nuclear reaction and conduct the generated heat to the coolant [6]. Prismatic fuel element blocks also provide vertical and lateral mechanical support for fuel cannisters in the graphite elements. These fuel elements are exposed to the largest doses of heat and radiation as they are nearest to fuel material and will therefore experience the most deformation.

Graphite reflector elements are responsible for moderating and reflecting neutrons back toward the active core region [194]. They differ from fuel elements by the absence of fuel and coolant channels. These reflector elements can be further divided into smaller categories based on their location and lifetime, but all share the same main functions of moderation and reflection. Typical reflector types include the following:

- Removable reflector elements surround the active core region and are designed to be replaceable. These components usually have a lifetime of four to ten years before peak radiation damage occurs [6], and their dimensions are similar to those of fuel elements. However, some advanced reactor designs will not replace many components and instead use permanent reflectors.
- Permanent side reflector elements surround both removable reflector elements, if present, and fuel elements. Due to being permanently installed, they are designed to last the lifetime of the plant. If removable side reflectors are present, the permanent side reflectors receive lower irradiation dosage and suffer from less irradiation and heat damage over their lifetime.
- Some designs also incorporate a central reflector in the center of the core. This element can be either permanent or replaceable, and while also reflecting and moderating, primarily provides mechanical support.
- Pebble reflectors are a more niche reflector element. These are essentially removable side reflectors for high temperature, pebble bed reactor designs and are removable spherical graphite elements placed closest to the core region (e.g., intermixed with similarly sized fuel spheres).

Aside from the pebble reflectors, the other reflector elements are usually designed as free-standing bricks. This design minimizes gapping caused by thermal and irradiation expansion [195]. Additionally, the stress due to dimensional changes will possibly cause permanent distortions to all reflector elements and could require replacement sooner than anticipated.

Specifically for high temperature gas reactors, the permanent graphite elements should ideally have a lifetime of greater than 50 effective full power years (EFPY) but could have a more expected lifetime of 15-20 EFPY before needing replacement or decommissioning due to irreversible damage caused by irradiation and heat [13].

Permanent support elements, such as pedestals and columns, also function to moderate neutrons, but their main purpose is to provide vertical and lateral mechanical support for the core [14]. These elements also displace coolant, lowering the overall required coolant volume [6]. While the other graphite components can be considered supportive and load bearing, these elements have higher stress limits designed specifically to reinforce the core. They are also designed to last the lifetime of the plant, like permanent side reflectors.

7.4 Nuclear Industry (Total Mass) Needs (Reactor Types, Time to First Order, Number of Reactors)

The graphite needs of the nuclear industry are growing as advanced reactor designs incorporate many graphite core components. The advanced reactor designs utilizing such components include high temperature gas reactors, small modular reactors, and molten salt reactors. The specific components found in these reactors are described in Section 1.2 and Section 7.3. Since most of these designs are still in the early stages of development, their actual technical specifications regarding graphite are difficult to determine with most design criteria still being proprietary. Three designs available give some idea of graphite mass needed:

- Kairos Power is developing a 236 MWth pebble bed, fluoride salt-cooled, high-temperature reactor. The design calls for 43,310 kg in outer fixed radial reflectors, a 5,940 kg central reflector, 11,560 kg in fuel pebbles, and 5,362 kg in reflector pebbles [15]. This yields a total of roughly 60,000 kg graphite. Hermes, a smaller experimental reactor is expected to be completed in 2026. In a recent announcement, Kairos is partnering with Google to deploy the first FHR by 2030.
- The U-battery was a small modular high-temperature gas-cooled reactor, which was designed for 10 MWth and 20 MWth capacities. The reflector masses specified are 8.1 kg and 70 kg, respectively [18]. Both designs also called for graphite fuel blocks but the mass of these are not specified. The U-battery was a design that is no longer being pursued and is being described here as a reference point for a reactor of its size.
- A 2225 MWth, two-fluid, liquid fluoride thorium reactor has been designed by ORNL, with a total graphite mass of around 255,000 kg [24]. There are currently no plans for this reactor's construction.

Aside from three described above, there are at least seven other graphite-moderated reactor designs being developed by several companies. While there is not much available information on these seven other reactors, the designs described above demonstrate the large graphite needs for next generation of nuclear power plants and a sufficient idea of the growing demand as these designs are constructed in the coming years.

Using the masses for the three examples above and the market assumptions discussed in the introduction to this section, industry needs for graphite can be estimated as follows:

- Kairos or comparable design: 60,000–600,000 kg/yr
- U-battery or comparable design: 800–8,000 kg/yr
- ORNL or comparable design: 25,000–250,000 kg/yr

These estimates are a small fraction of the ~2 million tons ($2x10^9$ kg) produced each year by graphite suppliers. On the one hand, this indicates that supply quantity is not likely to be an issue. On the other hand, there has been ample experience in the LWR industry that being a small part of a large market can lead to difficulties with special requests (e.g., additional quality assurance measures or additional specifications).

7.5 Definition of Domestic Manufacture

While there is a concern for graphite use in advanced reactors on an international level, this section discusses domestic manufacturing of graphite from a U.S. perspective. Identification of domestic manufacturing is a concern in the U.S. due to government incentives for manufacturing. Similar concerns are expected to be a factor in other jurisdictions as well.

For a structural material like steel, a domestic manufacturer is defined as a manufacturer that smelts and mines its materials from the U.S. [196]. Such a definition does not currently exist for graphite materials. Currently there are no major graphite mines in the United States. While there are graphite deposits in the U.S. according to the United States Geological Survey (USGS), particularly in Alaska, there has not been any graphite production in the U.S. since the 1950s [197, 198]. However, these observations are related to natural graphite, i.e., mined resources, which are not expected to be a significant factor in nuclear graphite, which is generally a synthetic product.

For synthetic graphite, it is unclear what stage of production would be used as a marker for the country of origin. Each of the following could be a factor:

- Raw material (coke, binder, filler) origin
- Billet production location (forming, graphitization, or densification location)
- Component machining location

At this time, there is no indication which of these points in manufacturing would be determinative of origin (or partial origin if the country of manufacture is considered to include multiple locations). Currently the lack of graphite production in the U.S. is due to the lack of raw material production. There are still companies in the U.S. that produce billets and machine graphite, but there are no graphite mines currently located in the U.S. This means that if raw materials are the marker for the country of origin, then domestic graphite cannot be made. Billet production and graphitization may be considered the “origin” of the graphite being produced, as it isn’t considered graphite until after the graphitization step. Companies do perform graphitization in the U.S. and domestic suppliers would exist if graphitization is considered the stage where the graphite “originates.” This is similar to component machining, which is also performed in the U.S.

7.6 Definition of Nuclear Grade Graphite

Nuclear grade graphite is currently defined only loosely in ASTM D7219-08 and ASTM D7301-08 as well as the requirements of the ASME BPVC. However, these definitions do not provide enough detail for adequate procurement of components. For example, issues of quality assurance are not addressed. Likewise, a clear definition of representative material is not available (e.g., for determining how many components of an order must be destructively examined to provide demonstration of adequacy, the use of proof components for demonstration of adequacy, the extent to which different groups of material can be considered a single material type for qualification, etc.). The issue of material grouping is discussed more extensively in Section 4.2.

8 AGING ISSUES

8.1 Frameworks for Aging Management of Nuclear Components

In the U.S. there are two frameworks for establishing aging management programs for nuclear components. ASME Section XI, Division 2 was established in 2019 and has not generally been applied at this time. NEI 03-08 was established in 2003 and has been universally applied at the operating LWRs in the U.S. (with parts of the program being adopted in other jurisdictions as best practices rather than requirements). These frameworks are briefly described in Sections 8.1.1 and 8.1.2, respectively. The relationship between the two programs is summarized in Section 8.1.3. Currently identified gaps in these frameworks as they might apply to graphite are discussed in Section 0.

8.1.1 ASME Reliability and Integrity Management (RIM)

ASME Boiler and Pressure Vessel Code (the Code) was developed in response to industry events. The following historical note is taken from the ASME website:

> Two boiler explosions in Massachusetts shoe factories, (Brockton, 1905 and Lynn, 1906) motivated the Governor to include in his inaugural address a demand for prompt action for improved public safety. One outcome of this mandate was the creation of a new Massachusetts law, "An Act Relating to the Operation and Inspection of Steam Boilers" (1909). This motivated another state, Ohio, to draft their own laws in 1911. At the same time, as both states were developing laws, ASME was looking to the future in a way that would change the boiler industry and its future evolution, industry-wide standardization. From ASME's actions, the first edition of the Boiler and Pressure Vessel Code (BPVC) was issued in 1914 and published in 1915 [199].

In the 2019 edition of the ASME BPVC, Section XI, Division 2 was issued with the title *Requirements for Reliability and Integrity Management (RIM) Programs for Nuclear Power Plants* [200] (Previously, Division 2 had covered high-temperature gas-cooled reactors but was discontinued in 1995 because the only such reactor in the U.S. was shutdown.). Section XI, Division 2 lays out requirements for a risk-informed approach to managing degradation in subsystems and components. A high-level overview of the general RIM program process is given in Figure 8-1.

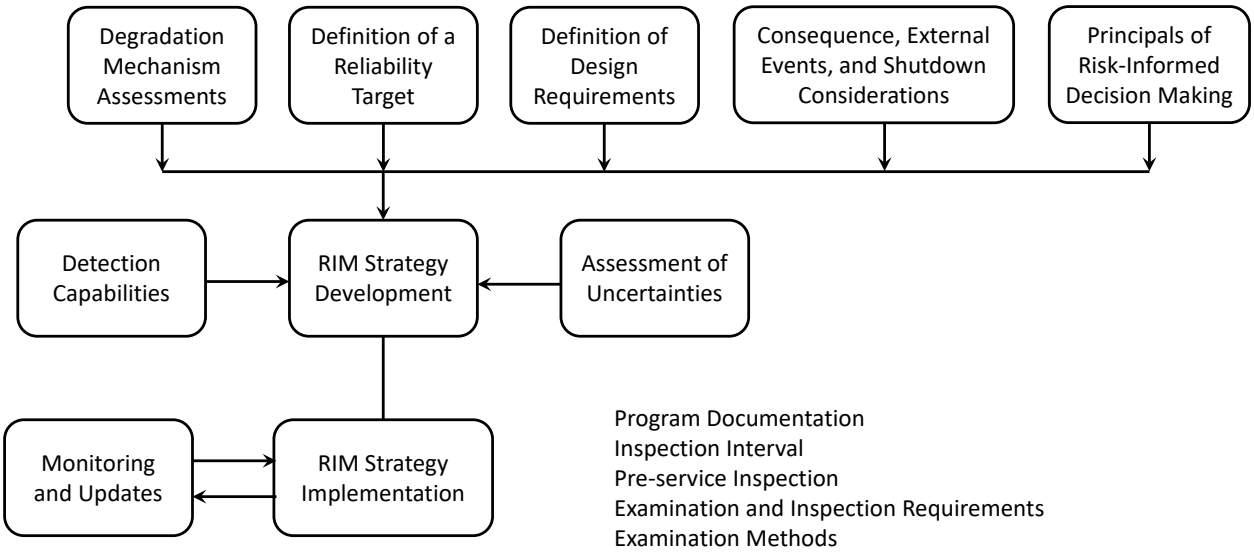


Figure 8-1. Overview of ASME RIM process

8.1.2 NEI 03-08 Framework

In 2003, the Nuclear Energy Institute issued NEI 03-08, Revision 0. This document established an industry commitment in the U.S. to a framework for managing material degradation issues in operating nuclear power plants. Since its initial issue, NEI 03-08 has been periodically revised. The latest revision is Revision 4 [201], issued in October 2020.

NEI 03-08 was issued in response to a corrosion event at Davis-Besse that nearly led to a loss of coolant accident. Although it made new commitments to the U.S. NRC on behalf of the U.S. utilities, it leveraged existing programs at EPRI, INPO, and the PWR Owner's Group (PWROG). It explicitly defined the relationships among the various entities involved in the management of materials issues. The overall framework structure is illustrated in Figure 8-2. The roles of the different organizations are as follows:

- NEI established and maintains the structure of the framework through NEI 03-08. NEI also represents the utilities in discussion of commitments to the NRC.
- EPRI (and to a lesser extent the PWROG) independently organizes and oversees technical tasks required to support the industry's commitments. This includes oversight of research activities and the development of guidance. EPRI's responsibilities (as well as those of the PWROG) are divided among specific issue programs (IPs), identified in NEI 03-08. The current IPs are as follows:
  - EPRI BWR Vessel and Internals Project (BWRVIP)
  - EPRI Materials Reliability Program (MRP)
  - EPRI Steam Generator Management Program (SGMP)
  - EPRI Non-Destructive Evaluation Program (NDE)

  - EPRI Water Chemistry Program
  - EPRI Welding and Repair Technology Center (WRTC)
  - PWROG Materials Committee (PWROG MSC)
- INPO is responsible for performing on-site reviews and evaluations of the implementation of the guidelines and standards developed by EPRI.
- Utilities are responsible for implementing the guidance developed through the NEI 03-08 process.

Note that since utilities are intimately involved in the organization and administration of NEI, EPRI, and INPO, there is significant feedback.

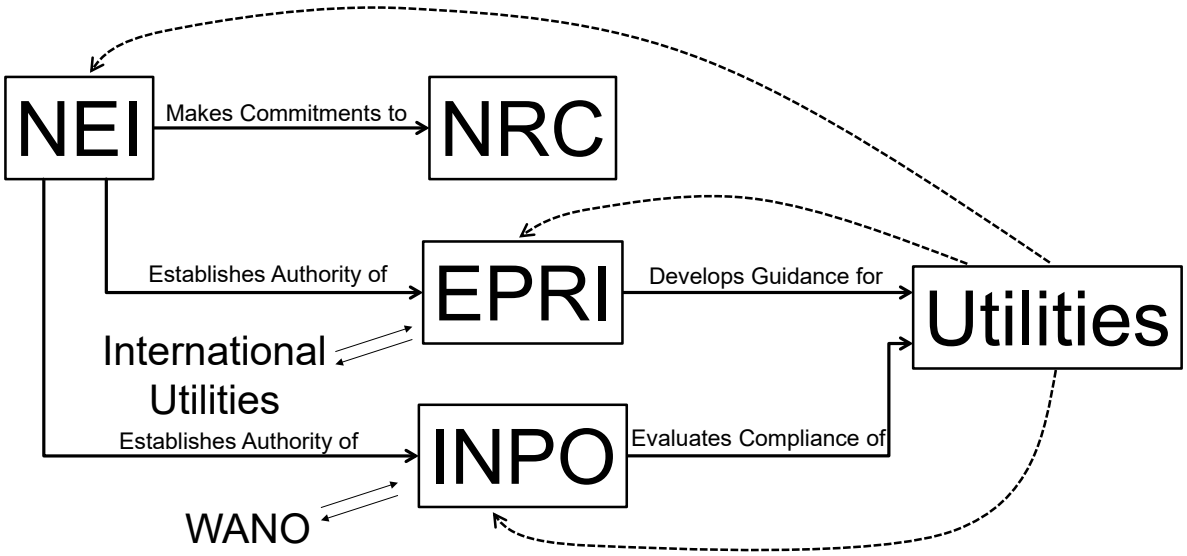


Figure 8-2. NEI 03-08 framework structure

A key feature of the NEI 03-08 framework is the fact that the NRC does not provide direct oversight of materials issues. In this sense, materials management by U.S. utilities is self-regulated. This extends to all of the issues covered by NEI 03-08, for example, chemistry programs, steam generator inspection programs, etc. In other jurisdictions, the regulatory body provides direct oversight of these issues, which can be a significant burden to utilities.

8.1.3 Relationship Between RIM and NEI 03-08

It should be noted that, like the NEI 03-08 framework, the Section XI, Division 2 framework is not new. Many RIM programs are already implemented in management of materials issues in the operating LWR fleet. For example, Figure 8-3 shows the steam generator degradation management process described in the *EPRI Steam Generator Integrity Assessment Guidelines* (SGIAGL) [202]. Figure 8-3 illustrates the details of the two lowermost blocks in Figure 8-1 (implementation and updates). The other blocks in Figure 8-1 have been addressed in the development of the SGIAGL over more than two decades.

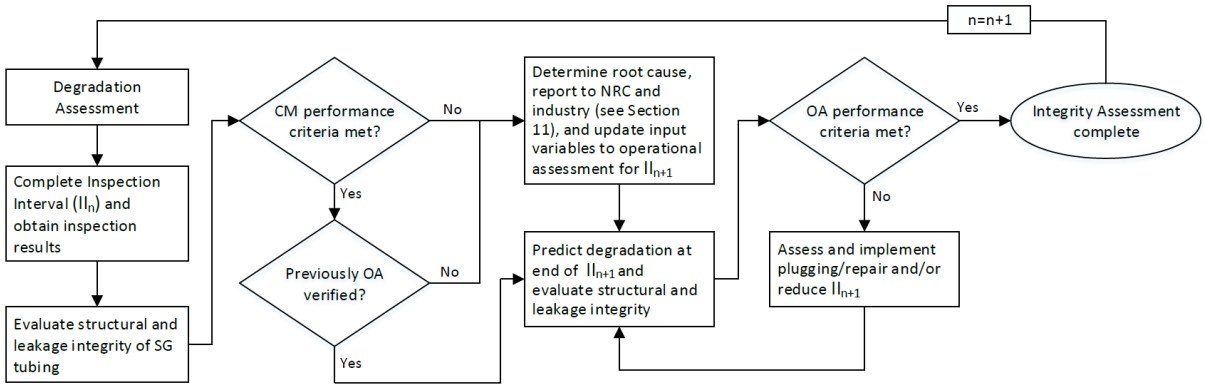


Figure 8-3. EPRI SGMP integrity assessment process [202]

The position of RIM programs within the overall framework of materials management at operating reactors is illustrated in Figure 8-4. As discussed in Section 8.1.2, NEI 03-08 establishes (or confirms) the role of EPRI Issue Programs in generating industry guidance and the role of INPO in auditing utility compliance with that guidance. This process has generally led to NRC acceptance of the self-regulated status of materials degradation management.

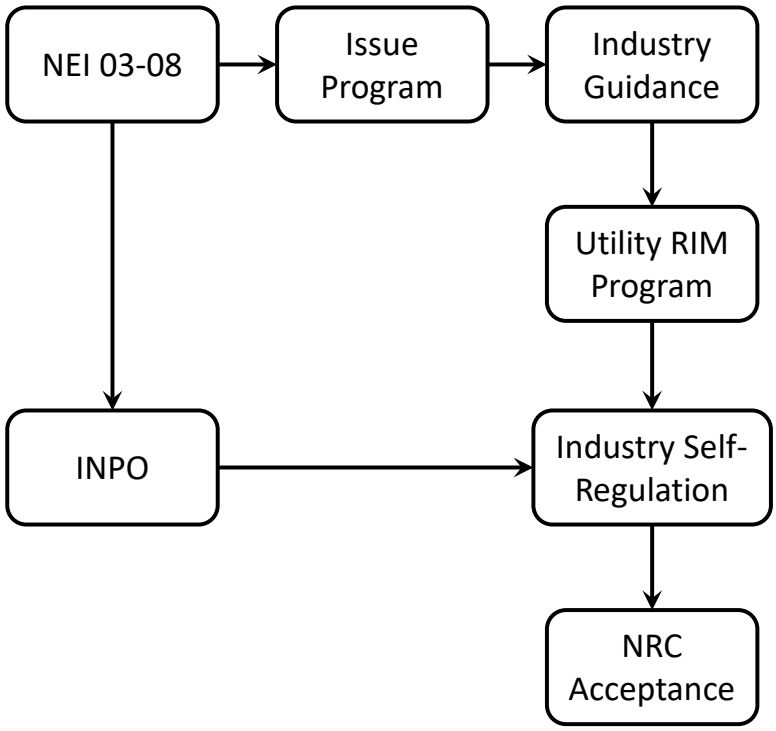


Figure 8-4. Position of RIM programs in current framework

8.1.4 Aging Management Framework Gaps

When considered within the experience of NEI 03-08 programs, the RIM process is robust and well proven. Most existing technical gaps are already identified elsewhere, as follows:

- Degradation mechanisms are addressed in Section 5.
- Detection of flaws is discussed in Section 4.11 and also in Section 8.4.1.

Implementation gaps may exist but have not been specifically identified. In large part, this is due to lack of a publicly available definition of the performance requirements for an actual graphite component, which would allow an assessment of the effects of various degradation modes on that performance. One possible way to identify any existing gaps would be to perform an example RIM assessment, i.e., to complete steps in Figure 8-1 up to *RIM Strategy Development*. Completion of such an example assessment would provide a template for similar assessments for other components. It would also help clarify the extent to which additional information on degradation and inspection are needed.

8.2 Tertiary Creep and End of Life Determination

Irradiation creep in graphite, as in other reactor materials, occurs in three phases: primary, secondary, and tertiary [98, 100, 203]. The primary creep occurs rapidly at very low dose, secondary creep occurs at intermediate doses, and tertiary occurs at high doses. It is relatively well understood that secondary creep occurs at least until the turn-around fluence, but during the secondary creep phase, the creep rate is continuously decreasing with increasing fluence [93, 204]. Limited high-dose studies [93, 204] have observed that the transition from decreasing creep rate, in the secondary regime, to increasing creep rate (possible onset of tertiary creep) occurs around the turn-around fluence for dimensional change in the direction of applied load. The transition to tertiary creep occurs at a lower fluence when loading is in a tensile state because a tensile stress decreases the turn-around fluence in the direction of applied stress, but interestingly the minimum creep rate for both stress states is roughly the same immediately before changing from secondary to tertiary (see Figure 3 in Reference [204]). Even the high dose experiments have not gone past the cross-over fluence (return to original dimensions), so commentary on the tertiary creep is all speculative, but from trends seen in Figure 3 in Reference [204] it appears that the tertiary creep rate will continue to increase in a parabolic fashion with increasing dose.

8.3 Acute versus Chronic Oxidation Response

While uniform oxidation of test specimens is necessary to correlate degradation in properties to weight loss, acceptable methodology and criteria for achieving uniform oxidation have yet to be established [126]. Furthermore, different material property measurement techniques usually have distinct shape and size requirements likely to necessitate methods customized to each measurement technique.

Assuming tight control of contaminants in the helium coolant, chronic oxidation is not expected to penetrate beyond a few millimeters of the component surface, with local weight loss not expected to exceed 1% [126]. However, air or steam ingress could have much more serious consequences. Both the oxidation rate behavior and the correlation between density and the graphite material properties (for each graphite grade in question) need to be characterized to satisfy ASME code guidance. Graphite behavior models have been steadily improving, but proper model validation requires demonstration of performance at full scale. Uncertainty remains regarding the influence of radiation dose on graphite oxidation: conservatism could unnecessarily limit the design life of graphite core components. While monolithic graphite is inherently less vulnerable than small test specimens, data are not currently available to confirm expected performance with this vast difference in scale. Therefore, models cannot be relied upon without substantial corroborating evidence from inspection or monitoring in-reactor service performance.

8.4 In-Service Issues (Monitoring Problems/RIM Specific Issues)

Degradation modes that are relevant to a component performing its design function will need to be assessed in order to determine appropriate intervals for inspection and evaluation or replacement. Relevant degradation modes are expected to include some of the following:

- Thermomechanical Degradation
- Irradiation Induced Structural Issues
- Irradiation Related Non-Structural Issues (e.g., Fission Product Retention, Impurity Activation)
- Corrosion / Chemical Degradation
- Wear

These issues are discussed in detail in other sections of this report, as indicated above. In addition to projections of future degradation for each mechanism, RIM programs (see Section 8.1.1) will also require inspection capabilities. Non-destructive examination (NDE) techniques are discussed below in Section 8.4.1.

Depending on the level of understanding of the degradation mechanisms applicable to a specific component, the detection limits of available NDE, and the inspection intervals that are economically acceptable, some designers may develop a surveillance program to support continued operation of graphite components. Surveillance programs are discussed in Section 8.4.2.

Finally, it is possible that the presence of graphite in the reactor design may have effects on other components. These possibilities are discussed in Section 8.4.3.

8.4.1 NDE

Currently there is no guideline in the ASME BPV Code in Section III or Section XI for NDE of graphite core components. There is currently a supplement being written for Section XI addressing the RIM problems associated with graphite core components [205]. This supplement provides requirements for the RIM Program to identify and evaluate the degradation mechanisms of graphite core components, as well as other unique requirements that are particular to graphite that will supplement the RIM program. The proposed RIM program for graphite includes degradation mechanisms, fast neutron irradiation, and thermal oxidation. This supplement also includes specifications for component monitoring and nondestructive examination (MANDE). As there are currently no specifications in place for NDE of graphite core components, and there is very little information on the aging of graphite in high temperature irradiation environments being proposed for advanced reactor systems, there is a large gap in the understanding of how to monitor graphite while in service. Closing this gap will involve actions like the supplement for Section XI of the ASME BPV Code, as well as further understanding of graphite creep and fatigue in advanced reactor environments, and how those properties change between graphite grades.

Additionally, NDE methods for graphite are expected to eventually require a qualification program. However, the extent to which NDE would need to be qualified is likely to be a function of the requirements of the RIM program, since the need for NDE and its detection limits are expected to be just one aspect of the RIM program, with these needs balanced with the significance of the degradation mode to the performance of the component function as well as with the inspection interval. In some cases, performance demonstrations such as the generation of Examination Technique Specification Sheets [206] may be necessary. In other cases, a performance demonstration initiative such as that described in Reference [207] may be warranted. However, in other cases, no additional qualification beyond the development of an industry standard may be needed.

8.4.2 Surveillance Sampling

When commercial LWRs were initially licensed, constructed, and brought into operation, there were few data available on the long-term effects of radiation on reactor pressure vessel embrittlement. Testing in fast reactors, which matched long-term neutron fluence magnitudes but not spectra, indicated that embrittlement levels could affect vessel performance within the design lifetime of the plant. In order to provide assurance to regulators in the absence of other data, designers included surveillance specimens in the reactor under the following conditions:

- Samples were made of material representative of the actual reactor vessel.
- Samples were placed near the core such that they experienced more irradiation than the reactor vessel, ensuring that the samples were always experiencing more damage than the vessel.
- Samples were of sufficient quantity to allow for surveillance over the entire design lifetime of the plant.
- Samples were of a size and shape as to facilitate evaluation of the effect of irradiation.

These surveillance programs were highly successful in establishing confidence in the performance of the reactor pressure vessels and were accepted by regulators. The main deficiency in the program was that designers failed to anticipate that the operating lifetime of the reactors would be extended to double the length of the design lifetime. However, even this deficiency has been overcome by implementation of an industry-wide program leveraging all existing samples as well as efforts to recycle specimens already tested [208].

Given the state of knowledge of graphite degradation, it would be considered prudent for reactor designers to plan on a surveillance program. Such a program would be expected to be incorporated into a RIM program (see Section 8.1.1), and thus consider various other factors including the design life of the graphite component, the relevance of degradation modes to the functioning of the component, and the expected inspection intervals, as well as the confidence in models predicting future degradation.

8.4.3 Effects on Other Components

The focus of this report is the assessment of the deployability of graphite, and therefore much of the discussion is related to availability, quality, control, degradation, etc. of graphite. However, it must be recognized that graphite components in advanced reactors will contribute just one material of many to the reactor systems under consideration. Therefore, the effect of graphite on non-graphite components must also be considered.

One mechanism by which the presence of graphite could affect other non-graphite components is through carburization, the chemical reaction of carbon with metals to form metal carbides. This mechanism is not considered at all in the ASME BPVC Section III. It is briefly considered in ASME BPVC Section XI in Table VII-3.2-1 [11]. In that table, carburization is identified as a degradation mechanism that could be relevant as follows:

- For carbon or low alloy steel at >350°C, in the presence of helium with impurities
- For austenitic stainless steel at >500°C, in the presence of helium with impurities

Section XI does not address the level or type of impurities that could promote carburization.

Carburization has also been identified as a potential issue in molten salt systems [209, 210]. However, at this time, most work in this area appears to be mainly scientific in nature, i.e., the extent to which carburization in molten salt might affect the performance of metal components has not been assessed.

The possibility of galvanic corrosion in molten salts of metal components coupled to graphite has also been identified [211]. In this case, the graphite acts as a cathode, accelerating the corrosion of the coupled steel. As with carburization, this degradation phenomenon is still at the level of scientific investigations and the engineering significance of the degradation mode has not been assessed.

9 DISPOSAL

Many countries, including the US, have operated reactors that contained graphite core components but have been shutdown and dismantled. In each country, and in some cases for each reactor, the removal, processing, handling, and disposal of graphite is handled differently. This is due to the fact that no two reactor designs will produce irradiated graphite with similar properties when considering disposal. Operational histories have a large bearing on the content and location of potential radioisotopes found within graphite core components. Even within reactors of similar type, variations in the graphite core components due to manufacturing processes and raw materials can change the material properties and the material property response to irradiation and thermal degradation. This leads to differing, and in some cases seemingly competing, methods of removal. For example, France plans to dismantle the UNGG reactors under water to provide shielding in response to thermal neutron effects. The UK does not see any purpose for underwater dismantling for the Magnox reactors or AGRs, as underwater dismantling can result in isotope leaching from the graphite core components into the water, resulting in additional complications. Disposal of graphite is more complicated than simply removing the pieces and storing them away. Understanding the level of activity in individual graphite components as well as the available methods of removal, processing, handling, and storing are all parts of the disposal process. Much of the information considered in this section is derived from the IAEA TECDOC series on processing of irradiated graphite for disposal [28].

9.1 Characterization of Irradiated Graphite

Graphite waste needs to be characterized for proper disposal. The features of irradiated graphite as they are removed from the reactor determine the disposal path. Four principal categories of irradiated graphite have been identified by the IAEA through a coordinated research project centered on the disposal of graphite core components. The categories are based on the identification of the radiological status of the graphite components and the physical and chemical parameters and properties based on structural data and impurities. The four principal categories based on this information are defined by the disposal path that is recommended for that category, as follows [28]:

- *Fuel-contaminated graphite* consists of irradiated graphite in need of immobilization or treatment before being safely stored or disposed of. This is considered high level waste (HLW).
- *General irradiated graphite* has available disposal pathways but will require appropriate conditioning before safe storage and disposal.
- *Treatment-expedient irradiated graphite* can be removed in a shorter timeframe due to available treatments.
- *Decontamination-expedient irradiated graphite* is suitable for decontamination within its waste stream.

Different reactor designs will produce different effects on graphite over the course of its lifetime within the core. Variations in irradiated graphite properties occur from the position within the core, as well as the characteristics of the core irradiation field. Irradiated graphite has been characterized by IAEA CRP studies as an inhomogeneous material with non-homogenously distributed radionuclides [212]. Because of this, and the effects discussed in Section 5.1, graphite core components become more disordered and complicated during their lifetime in reactor cores.

Graphite structure changes have been discussed in previous sections but also affect the dismantling and disposal of graphite core components. Irradiation and thermally induced expansion of core components can result in difficulties in removal of the components, as their expansion can hold them together with more force than originally intended. This can bring up issues of safety in the dismantling method. Additionally, graphite can act as a cathode in the right environment to produce galvanic corrosion of other materials [213]. Submerged dismantling is of concern for this, as excessive delays or introduction of strong electrolytes could affect the surrounding components in contact with graphite core components.

Radionuclide production in graphite is also of concern for removal, processing, and disposal. The level of activity as well as the types of radionuclides contained within graphite is determined by the impurity concentration, as well as the irradiation characteristics of the reactor. C-14 is of larger concern, as the activation of C-13 is possible given that graphite contains about 1.1% of C-13 atoms naturally [28]. Other impurities within graphite components could be activated, and these need to be considered in order to safely remove the components. Optimal removal, handling, treatment, and disposal requires an understanding of the location of radioisotopes within the structure of the graphite core components. There is a wealth of research on the production, propagation, and characteristics of radionuclides within graphite core components. In some cases, the location of radionuclides can be determined through the processing of graphite core components as well as the coolant employed in the reactor core. Inventories of short-lived isotopes allow for confidence in the detection and processing of those chemicals. However, longer lived beta emitters are of concern due to lack of inventories and possible concern of increasing concentration in repository storage [28].

9.2 Processing of Irradiated Graphite

The IAEA has expanded their discussion and research of processing irradiated graphite to include alternative dismantling methods; disposal routes other than interim, near-surface, or repository storage; the potential to recover particular radioisotopes; the potential to reduce the waste class of the irradiated graphite; and the potential to recycle the graphite for further use in the nuclear industry. The processing options included by the IAEA in their current CRP include the nibble-and-vacuum method, decontamination, gasification, and molten salt oxidation. Each method seeks to achieve a different goal in graphite disposal for different situations of reactor core conditions as described below:

- The nibble-and-vacuum process includes the breaking down of graphite core components into small pieces, and their removal through suction. The UK is employing this strategy due to their lengthy reactor operating periods. The length of time spent in the reactor core has

induced irradiation and thermal degradation in the graphite, making removal of intact blocks difficult due to distortion. One concern addressed by this method is whether whole graphite blocks are suitable for final storage, since, in some cases, powder or small pieces can be stored more efficiently than blocks retaining their original geometry. The UK is also considering the use of thermal treatment in order to separate radionuclides from the irradiated graphite to avoid geological containment [28, 214].

- Decontamination processes under consideration include the use of intercalation to separate irradiated contamination from graphite, or at least separate high or intermediate level waste graphite from low level waste graphite. Intercalation strategies involving sulfuric acid and hydrogen peroxide to exfoliate the graphite. This in turn is intended to facilitate an extraction process to remove a large portion of radioactive contamination from the graphite core components [28].
- The gasification method involves the incineration of graphite core components in order to separate the radionuclides from safe graphite. This has been under investigation due to the potential release of C-14 containing CO_2 and the production CO. There has been consideration that this method could be used to process graphite that is highly contaminated with fuel debris [1, 28].
- Molten salt oxidation aims to produce graphite that is glass like at normal temperatures. The molten salts are held at high temperature in order to oxidize graphite components. The glass like nature of the resultant graphite is more stable than the original graphite core components, allowing for easier handling and storage [28].

9.3 Disposal of Irradiated Graphite

Disposal pathways for graphite include various forms of underground storage. Different methods are practiced depending on the amount of irradiated graphite needing to be disposed of in different countries. For example, most countries with irradiated graphite in need of storage are using underground storage techniques, whether near surface or geological. However, Spain has only 3500 tons of graphite needing disposal. Spain is pursuing the option of selective C-14 removal and immobilization of graphite using glass to prevent further release of radionuclides. This process is normally quite expensive, but with the low amount of irradiated graphite in Spain's reactors, an underground storage facility would be much more expensive [28].

Most other countries are considering the placement of graphite in underground storage with special containers. Underground storage, even near surface storage, reduces the amount of decay fission products released into the immediate environment and imposes multiple barriers between contaminated matter and the environment. In the case of more mobile radionuclides, such as Cl-36, it is necessary to contain them in specialized units. Graphite containing higher amounts of Cl-36 will be surrounded by inorganic chlorides, such as a salt dome or lined container in order to limit the amount of diffusion and dispersion available to Cl-36 radionuclides [28].

9.4 Challenges in Graphite Disposal

While many countries have plans in place for the processing, handling, and disposal of irradiated graphite waste, not every solution is ideal. Underground storage of irradiated graphite waste meets the same criticism given to underground storage of other nuclear waste, with space and cost being reasonable issues. Some countries are approaching the problems with varying solutions, including off gassing from pyrolysis into CO_2 emissions from fossil-fired power stations. However, these solutions will need further research and modeling in order to understand the potential dispersion implications. The main issue facing irradiated graphite is the lack of consistency available across reactors. As mentioned before, no two reactors, no matter how similar the design, will produce the same irradiated graphite due to the pre-service inconsistencies and the variability in operation for graphite core components. This means that graphite disposal programs may need to be very high level and general in order to allow for individual reactors to develop their own more specified program when decommissioning is needed.

9.5 Summary of Disposal Gaps

As with other nuclear wastes, graphite component waste has numerous challenges. However, from the perspective of advanced reactors, these gaps are not a near-term issue. Multiple enhancements to the disposal process are being developed. Several reactors containing graphite components will be decommissioned in the relatively near future. Therefore, there are no gaps related to advanced reactors that need to be addressed at this time.

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