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Fundamental Graphite Irradiation Behavior Research at ORNL for FY23

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ABSTRACT

This letter report is in submission of completion of the Level 3 milestone number within the larger Advanced Reactor Technologies Gas Cooled Reactor program at Oak Ridge National Laboratory (ORNL). The focus of this year's efforts is the aggregation of background information about the irradiation experiments for property change in H-451 graphite, getting the irradiation-induced property change of IG-110 and ETU-10 written up in journal articles for publication, and involvement with ASTM D02.F0 Manufactured Carbon and Graphite Products and the ASME Working Group on Nonmetallic Design and Materials (SG HTR)(BPV III). This report will document the status of these activities.

1. IRRADIATION STUDIES OF H-451 GRAPHITE

The primary focus of this fiscal year effort was to obtain records about the irradiation programs that were performed to determine the effects of neutron irradiation on the properties of H-451 graphite. This section presents information about the various irradiation capsules and programs that are currently available in open-access ORNL reports, only those that are available to the public at https://www.osti.gov/. Some ORNL reports that contain information about certain programs are still covered under various document controls and will require additional approvals before those can be discussed openly and as such are not included here, while other information is contained in reports that are not available in digital copies.

Over the years, graphite grade H-451 has been irradiated at ORNL both in the Oak Ridge Research Reactor (ORR) and High Flux Isotope Reactor (HFIR), but also in the High Flux Reactor (HFR) in Petten. The programs within ORNL included the OG, OC, HTK, HTF, HTU, and HTN series of capsules. The irradiation of H-451 in the HFR at Petten occurred in the GC capsule. From what was gathered from the reports all the capsules within the HT sets (HTK, HTF, HTU, HTN) all were passive temperature control and full-length HFIR target capsules (additional digging into non-public information is needed to confirm this). The OG and OC capsules were actively monitored with temperature control and were full-length ORR capsules (additional digging into non-public information is needed to confirm this).

1.1 OG CAPSULES

The first irradiations of H-451 graphite occurred in the OG (believe to stand for Oak Ridge Graphite) series of capsules. The OG-1 capsule is detailed in a report from General Atomics GA-A13089 [1]. OG-1 was irradiated in position C-3 in the ORR for cycles 113-115 (3200 full-power hours), at temperatures of 600°C to 1400°C and neutron fluences of 1.6-3.7x10^{21} n/cm^2 [E>0.18 MeV]. Some samples were previously irradiated in GEH-13-422 capsules at PNNL with General Atomics, while some OG-1 samples were included in OG-2 for additional fluence accumulation. The capsules was instrumented with Chromel-Alumel and W-Re thermocouples and a sweep gas that was used to control the temperature. The true temperature from the W-Re thermocouple true temperature calculated from indicated by:

$$T_t = \frac{T_i}{1 - 2.45 \times 10^{-23}\,F} \tag{1}$$

Where, T_t is true temperature, T_i is the temperature indicated by thermocouple, and F is the thermal neutron fluence (n/cm^2, E<0.17 eV). The fast fluence is reported in units of E>0.18 MeV and was determined from Fe and Ti dosimeter wires, while the thermal fluence was determined from Va-Co flux wires.

The details of the OG-2 capsule are also captured in a General Atomic report GA-A13556 [2]. OG-2 was irradiated in position C-3 in the ORR from July 17, 1974 – January 5, 1975, at temperatures of 600°C to 1400°C and neutron fluences up to ~9.5x10^{21} n/cm^2 [E>0.18 MeV]. In this report the authors changed the fluence units from fast fluence in the water moderated ORR [n/cm^2 E>0.18 MeV]$_{ORR}$ to fluence values that are expected for a gas-cooled reactor [n/cm^2 E>0.18 MeV]$_{HTGR}$. The conversion used is:

$$\text{Fluence [n/cm}^2\text{ E>0.18 MeV]}_{HTGR} = 0.894 \times \text{Fluence [n/cm}^2\text{ E>0.18 MeV]}_{ORR}. \tag{2}$$

The maximum fluence reported in HTGR units is ~8.5x10^{21} n/cm^2 [E>0.18 MeV], resulting in the maximum of ~9.5x10^{21} n/cm^2 [E>0.18 MeV] when reported for ORR flux wires. This maximum fluence was obtained for samples that were previously irradiated in the OG-1 capsule, and additionally some OG-

2 specimens were included in the OG-3 capsule. The capsules was instrumented with Chromel-Alumel and W-Re thermocouples and a sweep gas that was used to control the temperature. The true temperature from the W-Re thermocouple true temperature calculated from indicated by:

$$T_t = \frac{T_i}{1 - 4.9 \times 10^{-23}\,F} \tag{3}$$

Where, T_t is true temperature, T_i is the temperature indicated by thermocouple, and F is the thermal neutron fluence (n/cm^2, E<0.17 eV). The fast fluence in units of [E>0.18 MeV]$_{ORR}$ and was determined from Fe and Ti dosimeter wires, while the thermal fluence was determined from Va-Co flux wires.

The details of the OG-3 capsule are also captured in a General Atomic report GA-A14211 [3]. OG-3 was irradiated in position C-3 in the ORR from June 21, 1975 – December 5, 1975, at temperatures of 600°C to 1400°C. The total accumulated fluence for this capsule was 3x10^{25} n/m^2 [E>29 fJ]$_{HTGR}$ (29 fJ = 0.18 MeV), where the same correlation between ORR and HTGR fluence in Equation 2 still holds. The maximum accumulated fluence on any one specimen was up to 9x10^{21} n/cm^2 [E>0.18 MeV]$_{HTGR}$ or ~10x10^{21} n/cm^2 [E>0.18 MeV] when reported for ORR flux wires. The capsules was instrumented with Chromel-Alumel and W-Re thermocouples and a sweep gas that was used to control the temperature. The true temperature from the W-Re thermocouple true temperature calculated from indicated by:

$$T_t = \frac{T_i}{1 - 7 \times 10^{-27}\,F} \tag{4}$$

Where, T_t is true temperature, T_i is the temperature indicated by thermocouple, and F is the thermal neutron fluence (n/cm^2, E<0.17 eV). The fast fluence in units of [E>0.18 MeV]$_{ORR}$ and was determined from Fe and Ti dosimeter wires, while the thermal fluence was determined from Va-Co flux wires.

1.2 OC CAPSULES COMPRESSIVE CREEP

The primary source of irradiation creep data for H-451 comes from the OC (believe to stand for Oak Ridge Creep) series of capsules. The OC series had five capsules, that had irradiation temperatures of 600°C (OC-2, OC-4) and 900°C (OC-1, OC-3, OC-5). Data and discussion of the performance of these capsules are presented in a wide range of reports. The most comprehensive is a report from 2009 from M. Davies and T.D. Burchell [4], while earlier discussion of some of the results were presented in the Ph.D. thesis of A.S. Mobasheran at The University of Tennessee, Knoxville [5]. Both the Davies report and the Mobasheran thesis discuss the results well but have only some discussion about the experiment design and control.

Experiment OC-1 was designed to be irradiated at 900°C in the E-5 position of the ORR from April 1, 1976 until May 22, 1976, with a maximum fluence of 1.3x10^{25} n/m^2 [E>0.18 MeV] [6]. Temperature was monitored with two moveable and 28 fixed thermocouples and controlled with MgO-insulated Nichrome Vanadium heaters located within the specimen holder and changes to the sweep gas [6]. 20 specimens were compressively loaded at 13.8 MPa, while 8 had reduced diameters to get to 20.7 MPa stress, and all specimens had shallow holes in each end for graphite pins and spacers for centering [6]. Half of the specimens were overloaded, and while a 0.5% overstrain 13.8 MPa specimens had the same creep coefficient as normal, the 1-1.5% overstrain of the 20.7 MPa specimens did show a reduced creep coefficient [7].

OC-2 was a 600°C creep experiment, irradiated in the E-5 position of the ORR for 2 cycles from June 28, 1978 – October 16, 1978, with a maximum fluence of 2x10^{21} n/cm^2 [E>0.18 MeV] [8]. The same design and operation of OC-1 was used for OC-2 for the applied stresses and temperature monitoring and control. The two differences between OC-1 and OC-2 was the alignment of the compression system was improved and the change from a linear differential transformer to a pneumatic load measuring device [8]. Flux analysis was performed for OC-2, which included 20 dosimeters at 4 radial and 5 axial positions [9]. The dosimetry wires were Fe, Ti, V-Co, and V-Fe where the ^{58}Fe and ^{59}Co were used to quantify the total flux, while ^{46}Ti and ^{54}Fe were used for quantifying the fast flux [9].

OC-3 was another 900°C creep experiment with the same overall design of OC-1 and OC-2 (it has yet to be confirmed that irradiation was done in E-5 position of the ORR or the dates of irradiation). Some samples from OC-1 were included in OC-3 to increase the peak fluence to ~3.2×10^{25} n/m^2 [9], a few of which were in stressed positions, while 5 were put into unstressed positions to study creep recovery. Analysis of the online temperature results of OC-3 showed the time-averaged temperature to be 899.9°C±3°C [5].

OC-4 was a 600°C creep experiment, irradiated in the E-5 position of the ORR from November 25, 1980 – January 15, 1981, and February 4, 1981 – March 16, 1981, July 20, 1981 – August 20, 1981, with a maximum fluence of 2.5×10^{21} n/cm^2 [E>0.18 MeV] [10]. Some specimens from OC-2 were included to achieve a peak fluence of 4×10^{21} n/cm^2 [E>0.18 MeV] [10]. Analysis of the online temperature results of OC-4 showed the time-averaged temperature to be 601.8°C±10°C [5].

OC-5 was the final 900°C creep experiment, irradiated in the E-5 position of the ORR from December 17, 1981, through March 8, 1982. with a maximum fluence of 1.12×10^{25} n/m^2 [E>29.3 fJ] [7] (i.e., 1.12×10^{21} n/cm^2 [E>0.18 MeV]). Previous specimens that were irradiated in OC-1 and OC-3 were included as well, giving a maximum fluence of 50×10^{24} n/m^2 [E>29.3 fJ] [7] (i.e., 5×10^{21} n/cm^2 [E>0.18 MeV]). This capsule irradiation ended early due to the temperature difference between the central movable thermocouples being 30°C-50°C [7]. During capsule disassembly soot was observed, which suggests there was oxygen that got into the capsule, which was most likely due to leaks from the bellows system.

1.3 GC CAPSULE TENSILE CREEP

Tensile creep of H-451 graphite was performed at the HFR reactor in Petten, under the umbrella agreement between the USA and the Federal Republic of Germany (FRG). Most of the information about this experiment is not currently available for the public to access. The only note within ORNL reports comes from ORNL-6502 [11] that states "all data given to ORNL and under review".

1.4 HTK CAPSULES

The HTK series (believe to stand for HFIR Target Kernforschungsanlage) consisted of 7 capsules. HTK-1 capsule was irradiated in the HFIR G-5 position for 5 cycles from March 28, 1978 – July 24, 1978, with a peak fluence of 10.7×10^{21} n/cm^2 [E>0.18 MeV] [8]. The samples from HTK-1 were reloaded into HTK-2. HTK-2 was irradiated in the B-1 position of HFIR for 6 cycles lasting September 14, 1978 [8] until June 28, 1979 [9]. HTK-3 was inserted into HFIR from May 30 1979 – August 30, 1979 [9], with a design temperature of 620°C [12] (total fluence is not reported). HTK-4 had a design temperature of 715°C, it was irradiated in the HFIR target position B-1 from August 17, 1980 - April 9, 1981 (10 cycles), with a peak fluence of 2.2×10^{22} n/cm^2 [E>0.18 MeV], assuming flux of 1.18×10^{15} n/(cm^2*s) [10]. HTK-5 had a design temperature of 600°C [10, 13] (620°C actual [7]), it was irradiated in the HFIR target position B-1 from December 24, 1981 – December 12, 1982 for 15 cycles and a peak fluence of 3.8×10^{26} n/m^2 (no neutron energy given assume E>0.18 MeV} [7] or 4.2×10^{26} n/m^2 [E>50 keV] [14]. HTK-6 had a had a design temperature of 900°C [7, 13], it was irradiated in the HFIR F-7 position from July 18, 1982 – January 5, 1983 (listed as January 5, 1982 in [7]) for 7 cycles to a peak fluence of 1.8×10^{26} n/m^2 (assume E>0.18 MeV) [7], or 2×10^{26} n/m^2 [E>50 keV] [14]. HTK-7 had a design temperature of 600°C, it finished irradiation after 16 HFIR cycles in January 18, 1992 with a peak fluence of 3.8×10^{26} n/m^2 [E>50 keV] or 2.89×10^{26} n/m^2 [E>18 keV] [15]. From what can be observed from the reports these capsules had no active temperature monitoring or control.

1.5 HTF CAPSULES

The HTF series (believed to stand for HFIR Target Fracture) included 3 capsules without active temperature monitoring or control. HTF-1 had a design temperature of 600°C, was irradiated for 6 cycles (May 4, 1983 – September 20, 1983), but upon disassembly a large amount of soot was observed in the gas gap and capsule and upon further inspection it was determined that the samples had corroded so no results were possible [14]. HTF-2 1 had a design temperature of 900°C, was irradiated for 6 cycles (May 4, 1983 – September 20, 1983) to a maximum dose of 1.5×10^{26} n/m^2 [E>50 keV] [14]. HTF-3 was originally supposed to be a duplicate of HTF-1 but was to be irradiated for 15 cycles. Instead, the loss of the HTF-1 specimens reduced the number of cycles to 5 [14]. HTF-3 had a design temperature of 600°C, was irradiated for 5 cycles (May 28, 1983 – September 20, 1983) to a maximum dose of ~1.25×10^{26} n/m^2 [E>50 keV] [14].

1.6 HTU CAPSULES

There was another set of "HFIR Target" capsules entitled the HTU series (believed to stand for the HFIR Target United) as the capsules were sponsored by United Nuclear to investigate TSX graphite (the grade used in the N-reactor) and three possible replacement grades. From what can be found H-451 was irradiated in the HTU-1 and HTU-2 capsules. HTU-1 had a target temperature of 575°C, and was irradiated from August 9, 1979 – March 5, 1980, for a total of 9 HFIR cycles to a maximum fluence of 2.3×10^{22} n/cm^2 [E>50 keV] [16]. From this capsule it was observed that even with HFIR having a flux 50 times higher than the N-reactor there was not difference in the dimensional change for TSX graphite irradiated in the two different reactors [16]. HTU-2 was designed with a target temperature of 450°C to a maximum fluence of 3×10^{22} n/cm^2 [E>50 keV] [11].

1.7 HTN CAPSULES

The final series of H-451 irradiation capsules were the HTN series (may have stood for HFIR Target New) as these capsules were to irradiate new graphite grades made with isotropic petroleum cokes as replacements for H-451. HTN-1 had a design temperature of 900°C, was irradiated for 4 cycles in HFIR (cycles 294-297) with a peak dose of 9.75×10^{25} n/m^2 [E>50 keV] or 7.41×10^{25} n/m^2 [E>0.18 MeV] [15]. Passive temperature monitors suggested the actual irradiation temperature in HTN-1 was ~800°C [15]. HTN-2 had a design temperature of 600°C, was irradiated for 16 cycles in HFIR (cycles 294-309) with a peak dose of 3.83×10^{26} n/m^2 [E>50 keV] or 2.91×10^{26} n/m^2 [E>0.18 MeV] [15]. HTN-3 had a design temperature of 900°C, was irradiated for 9 cycles in HFIR (cycles 304-312) with a peak dose of 2.18×10^{26} n/m^2 [E>50 keV] or 2.91×10^{26} n/m^2 [E>0.18 MeV] [15]. HTN-4 and HTN-5 were never assembled or irradiated because of the closure of the program funding this effort.

2. JOURNAL ARTICLE STATUS

The second focus of this fiscal year is to publish the results from two graphite irradiation campaigns that were performed at ORNL. These two campaigns captured a wide range of irradiation-induced property changes in IG-110, IG-430, and ETU-10, all grades that are being put forward as core structural materials for advanced gas-cooled reactors. The IG-110 and IG-430 data is being curated into a final report, with changes that were requested by the graphite manufacturer. After this report revision is complete the two papers describing the results will be ready for submission to the ORNL internal document review system, where they will undergo an internal review before being sent out to journals for publication. The first paper will describe the effects of neutron irradiation, over a range of temperatures and doses, on the materials properties (mass/density, elastic, mechanical, and thermal properties). The second paper will present the results of the irradiation creep experiments performed on IG-110 and IG-430, which will also include a comprehensive discussion about the newer creep mechanism proposed by Dr. Campbell. The ETU-10 data aggregation is complete, and the paper is in the preliminary draft stage and will be ready for submission to the ORNL internal document review system before the end of this fiscal year, where they will undergo an internal review before being sent out to journals for publication.

3. CODES AND STANDARDS DEVELOPMENT

The third focus of this fiscal year is the involvement in the development of materials codes and testing standards communities. This involvement includes contributions to be the development of reactor safety codes in the American Society of Mechanical Engineers (ASME) and development, testing, and implementation of materials testing standards within the American Society for Testing Materials (ASTM).

Dr. Campbell was selected as the ORNL voting member in the ASTM Subcommittee D02.F0 Manufactured Carbon and Graphite Products. Additionally, she is a non-voting member of the Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants, Committee C28 on Advanced Ceramics, Subcommittee C28.01 Mechanical Properties and Performance, and Subcommittee C28.03 Physical Properties and Non-Destructive Evaluation. Involvement in the D02.F0 subcommittee started with participation in the virtual subcommittee meeting on November 21 and 22, 2023. Most of the effort related to this meeting was listening to the current committee discussing needs related to development of new standards and updating of ones that are due for review. Dr. Campbell offered to collaborate with Dr. Wilkinson (National Nuclear Laboratory) on changing C559-16 (reapproved 2020) from a “Standard Test Method” to a “Standard Guide” for the June 2023 subcommittee meeting. This change is due to nuances between a “Test Method” that requires interlaboratory testing to determine the repeatability and reproducibility, while a “Standard Guide” does not include information about the repeatability and reproducibility. Dr. Campbell has been nominated by the D02.F0 subcommittee chair, Dr. A. Tzeleip, for the ASTM 2023 Emerging Professionals Program. If awarded Dr. Campbell will receive dedicated training on the consensus process within ASTM, she will have dedicated networking opportunities with technical experts, and the award includes travel assistance with flights and two nights at the code week hotel.

Within the ASME Dr. Campbell was appointed as a contributing member to the “Working Group on Nonmetallic Design and Materials (SG HTR)(BPV III)”. Within this working group Dr. Campbell is actively involved with the “Irradiation Effects in Graphite Task Group (Nonmetallic working group)”, the “Composites Task Group (nonmetallic working group)” and the “Design Task Group (nonmetallic working group)”. The “Irradiation Effects in Graphite Task Group (Nonmetallic working group)” is focused on understanding trends in the changes to the irradiation properties of materials and whether a generalized code case can be developed that can be used to allow for the use of new graphite grades to low operating doses (before turn-around), without the need for a comprehensive code qualification irradiation program. The "Composites Task Group (nonmetallic working group)" is focused on updating and revisions to Subpart B within the Section III Division 5 that details needs related to the use of composites within high temperature nuclear reactors.

The "Design Task Group (nonmetallic working group)" is focused on the revision of article HHA-3217 that describes how to determine the probability of failure (POF) of graphite core components. The primary concern is how the process zone volume (V_m) is defined in the code. In the 2019 and earlier versions of the III-5 code, V_m was defined as a cube with sides that are equal to a length that is 10 times larger than the grain size. With this definition, the finite element stress analysis for *superfine* grain graphite grades results in a POF that is overly conservative if not untenable. This means that any POF calculation for *superfine* grain graphite grades will not allow for use of these grades for any core components. In the 2021 version of the III-5 code the definition of V_m was changed to be based on the process zone size (r_c), which is calculated from the critical stress intensity factor (K_{Ic}) and the mean tensile stress. This change was implemented to results from and ORNL report [17] that suggested that V_m for *superfine* grain graphite grades could be used that were a factor of 10,000 larger than the V_m defined by the grain size. The problem with this change is there was an error in the calculation of r_c in the original ORNL report, which has since been corrected in Revision 1 released in 2022 [18]. Once this correction is applied, the V_m for *superfine* grain graphite grades is within a few percent of the V_m calculated from the grain size, meaning that a new process for defining V_m for *superfine* grain graphite grades is necessary.

4. ADDITIONAL FY23 ACTIVITIES

Besides the tasks listed in sections 1-3, there were two activities undertaken in FY23 that were not initially part of the work scope. The first of which was the co-hosting of the two Vendor Irradiation Capsule (VIC) workshops. The aim of the VIC workshops was to get reactor designers, graphite vendors, and irradiation testing experts together to discuss the future data needs to support ASME code cases for new graphite grades. The primary aim was to develop a plan that would allow for single design irradiation capsules for either the ORNL HFIR, or the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL) that could share design costs, space, transport, and disassembly costs between multiple entities rather than each reactor vendor or graphite manufacturer funding their own irradiation programs. The first workshop was hosted at ORNL October 3-4, 2022, and has a summary report that will be issued before the end of FY23. The second workshop was hosted at INL April 4-6, 2023, and will have a separate report issued by the end of FY23.

The other unplanned effort was the involvement on the "Graphite for Advanced Nuclear Reactors

Deployment Readiness Review" report that is being led by EPRI with input on sections from researchers at ORNL and INL. The contribution to the report from the fundamental irradiation effects area will be in the Degradation chapter, with a specific focus on the "Irradiation Response" section and the subsections on "Bulk Property Change" and "Irradiation Creep". At the time of this report, this EPRI report is in the preliminary draft stage, but most of the effort at ORNL will be completed before the end of FY23.

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