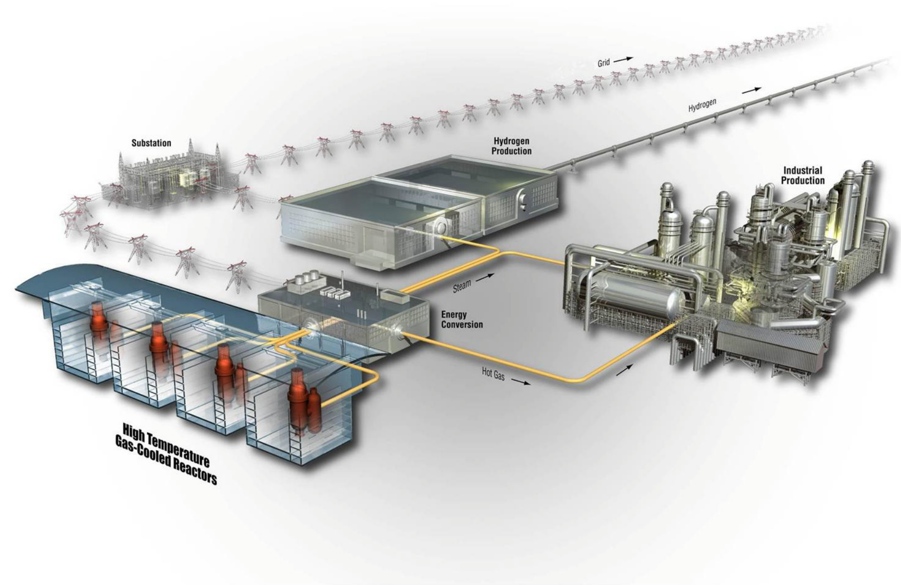
INL/EXT-11-22148

Carbon Characterization Laboratory Readiness to Receive Irradiated Graphite Samples

Karen A Moore

May 2011



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http://www.inl.gov

Prepared for the
U.S. Department of Energy
Office of Nuclear Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

Next Generation Nuclear Plant Project

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Approved by:

William Windes Graphite Lead	24-May-2011 Date
Travis Mitchell Graphite Project Manager	5/24/2011 Date

ABSTRACT

The Carbon Characterization Laboratory, located in Labs C-19 and C-20 of the Idaho National Laboratory Research Center, was established under the Next Generation Nuclear Plant Project to support graphite and ceramic composite research and development activities. The research conducted in this laboratory will support the Advanced Graphite Creep experiments—a major series of material irradiation experiments within the Next Generation Nuclear Plant Graphite program.

The Carbon Characterization Laboratory is designed to characterize and test low activated irradiated materials such as high purity graphite, carbon-carbon composites, silicon-carbide composite, and ceramic materials. It is fully capable of characterizing material properties for both irradiated and nonirradiated materials.

The major infrastructural modifications undertaken to support this new radiological facility are complete, equipment has been installed, radiological controls and operating procedures have been established, and work management documents have been created to place the laboratory in readiness to receive irradiated graphite samples.

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ACRONYMS

AGC	Advanced Graphite Creep
ALARA	As Low As Reasonably Allowable
INL	Idaho National Laboratory
IRC	Idaho Research Center
CCL	Carbon Characterization Laboratory
HEPA	high efficiency particulate air (filter)
NGNP	Next Generation Nuclear Plant
NESHAPS	National Emission Standards for Hazardous Air Pollutants

Carbon Characterization Laboratory Readiness to Receive Irradiated Graphite Samples

1. DESCRIPTION OF CARBON CHARACTERIZATION LABORATORY

The newly completed Carbon Characterization Laboratory (CCL) is located in Labs C-19 and C-20 of the Idaho National Laboratory (INL) Research Center (IRC). The CCL was established under the Next Generation Nuclear Plant (NGNP) Project to support graphite and ceramic composite research and development activities. The research supports the Advanced Graphite Creep (AGC) experiments—a series of major material irradiation experiments within the NGNP Graphite program.

The CCL is designed to characterize and test low activated irradiated materials such as high-purity graphite, carbon-carbon composites, and silicon-carbide composite materials. The laboratory is fully capable of characterizing material properties for both irradiated and nonirradiated materials. Lab C-20, which also serves as the entrance to Lab C-19, is used as a standard laboratory for the testing and characterization of nonirradiated samples. Currently, thermal, physical, and some nondestructive examination techniques are used to characterize the AGC samples before irradiation. Lab C-19 is the radiological facility. It has characterization capabilities similar those of Lab C-20, but has the added capability for mechanical testing. Figure 1 shows the layout of both labs. Two large windows were installed in the wall separating Lab C-19 and C-20 so that personnel can view work being performed in Lab C19. A portal monitor, located at the egress from Lab C-19, will be used to detect contamination on personnel before exiting the radiological facility. The work tables, equipment, storage cabinets, and desks in the radiological facility are arranged to ensure the maximum contamination control within the facility and protection of personnel.

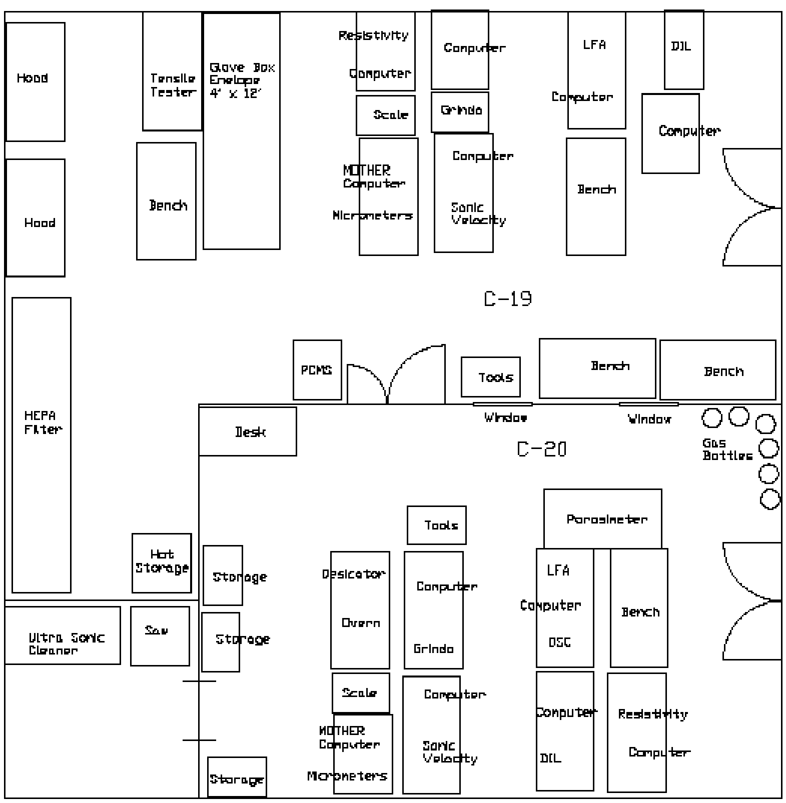

Figure 1. Layout of the Carbon Characterization Laboratory.

1.1 Lab C-19 Description

Lab C-19 was modified to receive and characterize irradiated material specimens. It is now prepared for a large graphite characterization campaign in support of the NGNP Project. Since graphite characterization involves the handling of nonsealed irradiated graphite, Lab C-19 was categorized as a Radiological Hazard facility and received an inventory evaluation by the National Emission Standards for Hazardous Air Pollutants (NESHAPS). The evaluation verified that the total radioactive inventory will remain under the criteria for a less than Hazard Category 3 (non-nuclear) facility, which is below 40 CFR 61, Appendix E[1] values for NESHAPS consideration. The engineering controls for radiological contamination in the workplace environment were evaluated in ECAR-469, "Radiological Control Recommendations for Post Irradiation Examination of Activated Graphite Performed at IRC."[2]

The irradiated materials to be tested and characterized in the CCL are assumed to have low activation levels. To further reduce personnel exposure, only a limited number of specimens will be out of controlled, shielded storage at any one time for testing. Handling of samples from current NGNP activities in the CCL are addressed in the as low as reasonably achievable (ALARA) review documents for each task.

1.1.1 Exhaust System

The air in Lab C-19 will have a negative pressure to contain any contamination that may be released during testing. The additional air flow will be maintained by a make-up air supply from the IRC. A new independent exhaust system was installed in Lab C-19 to ensure that all gases and air born particles are exhausted separate from the rest of the IRC building. The new welded stainless steel high efficiency particulate air (HEPA) filtered ducting system inside Lab C-19 directs air flow from the radiological fume hood or glovebox through HEPA filters to a roof mounted continuously running exhaust fan. The new HEPA filter housing is a 2 × 1 bag-in/bag-out, side access design. Phoenix™ variable air volume valves and balancing dampers were installed for the air distribution system. Ducts and diffusers were removed from the ceiling to cap off the existing HVAC system, and new ductwork was installed to connect to the independent exhaust system. The new exhaust control system is integrated with the existing IRC Carrier Control System to assist with the additional air flow required for the make-up air.

1.1.2 Fume Hood

A radiological fume hood was installed for tasks that may generate contamination, such as cutting and mechanical testing.

1.1.3 Glovebox

To support receipt of 10 or more samples at a time, an atmosphere glovebox with lead shield glass was installed in Lab C-19 as shown in Figure 2. The CCL glovebox will be used for all activities where higher than normal levels of activity are anticipated. The CCL glovebox has a large diameter transfer port to accommodate receiving long sleeves of specimens.

1.1.4 Material Storage

When a sample is not being actively tested, it will be stored in shielded storage to reduce personnel exposure. The current shielded storage facility stationed inside Lab C-19 is designed to provide shielding for the total number of samples anticipated in an AGC capsule (approximately 500 samples). The cabinet is a multilevel carousel composed of carbon steel. The carousel is housed within a 1-inch-thick leaded cabinet. The anticipated radiation level at the outer surface of a fully loaded storage system is calculated to be 5 mR.

Figure 2. CCL glovebox.

1.2 Tasks in Lab C19

A Radiological Work Permit was created and a workability walkthrough performed to ensure that all radiological controls, training, and monitoring systems are in place. Work in Lab C-19 will require RadWorker II certification and work in the CCL glove box will require glove box training as stated in Lab Instructions LI-1577-09-IRC. The laboratory walkdown process and attendance list with comments is included in Appendix A.

1.2.1 Transfer of Irradiated Graphite Specimens to IRC

Receipt of irradiated graphite will occur using a 55 gallon drum for three sleeves of AGC-1 piggybacks, then by a 7A drum for the remaining inventory of AGC-1 samples. A process walkdown has occurred for removing the irradiated specimens from the drum and transferring them to the glovebox. The specimens are photographed, measured for dimensions and weight, placed into their bar-coded plastic container as shown in Figure 3, then placed into shielded storage.

1.2.2 Characterization of irradiated specimens

Future characterization of irradiated graphite will be constrained by the activation of the individual specimen. Modeling predictions were used to analyze which characterization tasks could be performed on a bench top within a contamination area. Only tasks such as cutting or breaking the samples will require a fume hood or glovebox for contamination control.

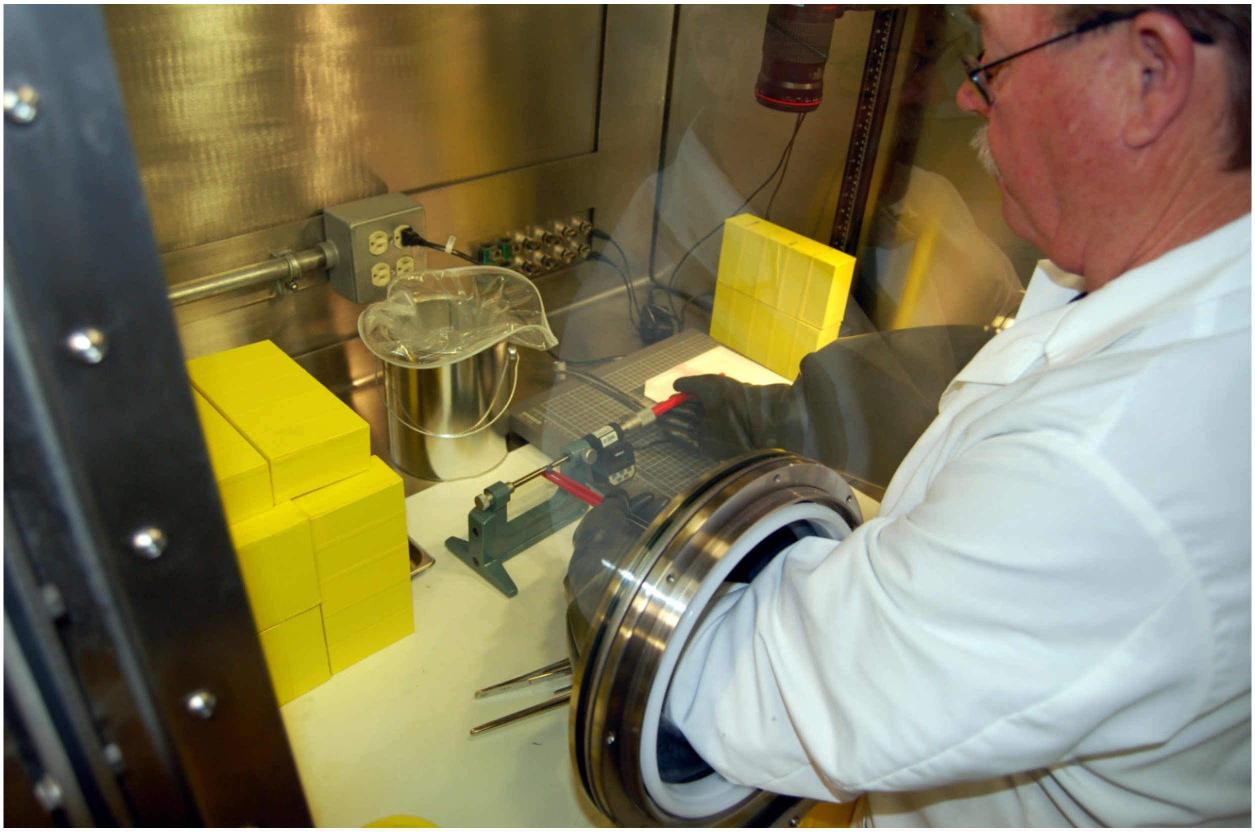

Figure 3. Measurement and transfer of samples to plastic containers.

2. ALARA

The ALARA review lists the work in Lab C-19 as a moderate radiological hazard because of total effective dose to the individual and contamination. The review is based on the initial transfer of AGC piggyback specimens to the CCL. These specimens are ½ inches diameter by ¼ inch long and weight 1.49 grams,. The estimated source term for these specimens is based on a calculated activation product summary table.[3] It is based on an activation period of 280 days with a 90-day cooling period prior to processing. The 10 most prominent radionuclides contributing to doses of one irradiated specimen are listed in Table 1. All are gamma-beta emitters.

Table 1 Radionuclides with estimated activity and dose.

Radionuclides in a ¼ inch long AGC-1 sample	Half-Life	Estimated gamma dose rate at 30 cm (mR/hr)
Ta-182	114 days	2.7
Sb-124	60 days	0.4
Co-60	5.1 years	0.3
Cs-134	2.1 years	0.3
Sc-46	83.8 days	0.3
Tb-160	72 days	0.1
Fe-55	2.73 years	0.04
Mn-54	312 days	0.03
Eu-154	8.6 years	0.03
Gd-153	240 days	0.02

The dose rates of the 12 AGC-1 piggyback specimens in their sleeve when they arrive in the CCL lab is 7.6 R/hr on contact (0.5 inches). The use of a 6-inch tool provides enough distance when handling to lower the dose rate to 190 mR/hr.

Although these specimens are solid there is apotential for dispersible contamination. Smear surveys in the CCL glovebox will assist in determining how much contamination can be expected. Lead bricks are available inside the CCL glovebox for shielding when a sleeve of specimens are in the glovebox.

It is anticipated that two personnel will perform the work. Removing the specimens from the drum, placing them in glovebox, and then moving them to a storage cabinet will result in a dose of 90 mrem for each worker. The daily characterization work is estimated to result in an average 4 mR/hr. The total dose estimate at the fingertips is 400 mrem.

3. REFERENCES

1. 40 CFR 61, "National Emissions Standards for Hazardous Air Pollutants," Appendix E, "Compliance Procedures Methods for Determining Compliance With Subpart I," *Electronic Code of Federal Regulations*, U.S. Government Printing Office.

2. ECAR-469, "Radiological Control Recommendations for Post Irradiation Examination of Activated Graphite Performed at IRC," D. Mecham, March 2011

3. INL/EXT-10-18039, "Laboratory for Characterization of Irradiated Graphite," W. Windes, March 2010

4. Memo to Karen Moore on Graphite Activation Analysis from Jim Parry, 03/29/2011.

Appendix A

CCL Walkdown Checklist and Attendance List with Comments

Appendix A—CCL Walkdown Checklist and Attendance List with Comments

Check List for the Small Quantity Transfer of AGC-1 Piggybacks Using the 8500 Container

Prerequisites:

______ Personnel prepared for extended work period inside RBA

______ Training complete for performing personnel

______ Radiological Work Permit (RWP) is approved

______ LI-1577-09-IRC is approved

______ Performing personnel signed into RWP and proper dosimeters issued.

______ Shipping drum received to INL Research Center (IRC) and located in C-19

______ Daily Briefing complete:
Scope of work
Identify hazards and mitigation
Discuss possible unexpected events and our reaction to them
Remind everyone that they have stop work authority.

Checklist

1. ______ Equipment staged in glovebox:
    a. snap cap storage containers with specimen ID barcodes
    b. Labeled Cryotubes for temperature monitors
    c. Plastic bags for specimen transfer
    d. Plastic bags for filter paper transfer
    e. Wet terry towels
    f. Wrenches for transfer tube
    g. Two pair 12 inch tweezers on each side of glovebox
    h. One pair 12 inch hemostats on each side of glovebox
    i. Watman filter paper for surveys
    j. scissors
    k. paint can for waste disposal and zip lock bags
    l. shallow transfer pan with v-block
    m. blotter paper
    n. camera and stand
    o. dial micrometer
    p. calibrated length standard
    q. lead bricks.

2. _____ Equipment staged in hood:
   a. pair of scissors
   b. three pair 12 inch tweezers
   c. water squirt bottle
   d. blotter paper
   e. terry towels
   f. lead bricks
   g. paint can for waste.
3. _____ Equipment staged for drum lid removal and sample transfer:
   a. leather gloves
   b. bolt cutters
   c. 15/16" end wrench
   d. blotter paper doughnuts
   e. ratchet or impact driver
   f. 8500 lid lifting tool
   g. 12 or 18 inch hemostats
   h. blotter paper for placement of drum, drum lid, 8500 lid and packing material
   i. two long handle transfer pans
   j. sample storage tweezers.
4. _____ Dimensional measurement software operational
5. _____ Camera is operational in CCL glovebox and talking to the computer
6. _____ Differential pressure is indicated on the exhaust Magnehilic gage
   a. Note pressure ____________in. of H_2O
7. _____ Inner door of transfer port closed.
8. _____ Outer transfer port door open
9. _____ Remove shipping drum lid.
10. _____ Survey lid and drum as required by RCT
11. _____ Remove packaging material
12. _____ Install blotter paper doughnut into barrel
13. _____ Remove 8500 lid
14. _____ Remove plastic wrapped specimen sleeve using long hemostat or lifting hook and place it in transfer pan.
15. _____ Place pan with specimen sleeve into glovebox transfer port and close exterior door.
16. _____ Replace 8500 lid.
17. _____ Open interior door and bring specimen sleeve into glovebox.
18. _____ Remove specimens and temperature monitor from sleeve.
19. _____ Perform general contamination survey of graphite specimen stack.

    Spec#_________ Swipe #_________contamination level ________ rad level _________

20. _____ Remove temperature monitor from the specimen pan and place in cryo-tube that has been marked with an identification number that corresponds to the temperature monitors location in the experiment. Below is the list of temperature monitors placed in cryo-tube containers and the below signed verify that the correct temperature monitor was placed in the correct container.

Temperature monitor: _______________ _______________ _______________

Verified By: ______________________________ Date:_________

Verified By:______________________________ Date:_________

21. _____ Bag cryo-tube and place in lead cave

22. _____ Identify the HLM specimen. Specimen number will start with the letter "J".

23. _____ Take contamination and rad level surveys of specimens as needed. Record readings. HLM specimen should be one of those measured.

Spec#_________ Swipe #_________contamination level ________ rad level _________

Spec#_________ Swipe #_________contamination level ________ rad level _________

Spec#_________ Swipe #_________contamination level ________ rad level _________

Spec#_________ Swipe #_________contamination level ________ rad level _________

24. _____ Bag specimen smears and transfer out of the glovebox as directed by RCT

25. _____ Identify specimen and scan the corresponding bar-coded snap cap into photo file name and dimensional software file name.

26. _____ Photograph and measure specimen.

27. _____ Place specimen into the corresponding plastic container and verify the correct specimen ID and bar-code on the container match. Below is the list of specimens placed in bar-coded snap-cap containers and the below signed verify that the correct specimen was placed in the correct container.

Specimen Number: _______________ _______________ _______________

Verified By: ______________________________ Date:_________

Verified By:______________________________ Date:_________

28. _____ Turn off micrometer power.

29. _____ Place container in a zip lock plastic bag.

30. _____ Repeat steps 21 thru 24 until as many specimens that can be photographed and measured that day are complete.

31. _____ Place bagged specimens into pan inside transfer port and close transfer port interior door.

32. _____ Both glovebox personnel survey out of the glovebox contamination area.

33. _____ Remove pan of bagged specimens from transfer port and place in hood.

34. _____ Remove snap cap specimen container from plastic bag and survey for contamination. If survey is clean pass container out of the hood to be placed in lead storage cabinet. If specimen snap cap does not survey clean, wipe down snap cap container with moist towel and suvey container again. When clean transfer to lead storage cabinet.

35. _____ Log specimen location in lead storage cabinet.

36. _____ At the end of each day secure hood for unattended safety.
   a. Secure all contaminated trash and any other sources of loose contamination.
   b. Remove all specimens. Specimen containers which have not yet been cleaned and placed into specimen storage should be placed into a clean zip lock bag and placed back into the glove box.
   c. Survey hood and post as necessary.

37. _____ At the end of each day secure Glove Box for unattended safety.
   a. Place any remaining samples behind lead bricks
   b. Survey glovebox and post as necessary.

CCL Walkthrough to Demonstrate Preparedness for Receipt of Irradiate AGC-1 Piggybacks

Date: April 19, 2011

Attendees:

Char Bain	F&SS IRC Facility Operations
Doug Hilde	F&SS IRC Facility Management
Steve Lindberg	F&SS Facility Management
Tom Luther	IRC Lab Manager
Earl Johnson	E&E Operations
Michael Connolly	ALD EE S&T
Debbie Puccinelli	Industrial Hygiene
James Durrant	R&D ES&H
John Yankeelov	DOE-ID
Keith Lockie	DOE-ID
Cliff Fineman	DOE-ID
Tim Miller	ES&H Manager
Declan Detrick	Radiological Control
Dan Mecham	Radiological Control
Robert Free	Radiological Protection Manager
Lawrence Burke	Radiological Protection
Will Windes	NGNP Graphite Program
Karen Moore	Materials Engineering

Comments and Resolution:

Leather Gloves are needed when removing the drum ring. The LI for Lab C19 will be modified to include the hazard and the required protection.

Ergonomic assessment was needed. An ergonomic assessment will be performed by Charlene Johnson.