ORNL/TM-2023/3011

Be_2C synthesis, properties, and ion-beam irradiation damage characterization

Anne A. Campbell
Ercan Cakmak
Blake Henry
Katherine R. Johnson
Diego Muzquiz
Yuri Osetskiy
Erica Paxton
Stephen Raiman
Dino Sulejmanovic
Eva Zarkadoula
David Holcomb

August 2023

OAK RIDGE National Laboratory

ORNL IS MANAGED BY UT-BATTELLE LLC FOR THE US DEPARTMENT OF ENERGY

ORNL/TM-2023/3011

Molten Salt Reactor Program

BE_2C SYNTHESIS, PROPERTIES, AND ION-BEAM IRRADIATION DAMAGE CHARACTERIZATION

Anne A. Campbell
Ercan Cakmak
Blake Henry
Katie Johnson
Diego Muzquiz
Yuri Osetskiy
Erica Paxton
Stephen Raiman
Dino Sulejmanovic
Eva Zarkadoula
David Holcomb

August 2023

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, TN 37831
managed by
UT-BATTELLE LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS

AOI	area of interest
BEPP	beryllium exposure prevention plan
DFT	density functional theory
EHS	Environmental Health and Safety
ESH&Q	Environmental, Safety, Health, and Quality
fcc	face centered cubic
Materion	Materion Brush Inc. (Elmore, Ohio)
MIBL	Michigan Ion Beam Laboratory
MSR	molten salt reactor
ORNL	Oak Ridge National Laboratory
PBE	Perdew–Burke–Ernzerhof
PPE	personal protective equipment
SEM	scanning electron microscopy
SOP	standard operating procedures
TEM	transmission electron microscope
UofM	University of Michigan
XRD	X-ray diffraction

ACKNOWLEDGMENTS

Support for this work was provided by the US Department of Energy Office of Nuclear Energy Molten Salt Reactor Program. This research used resources of the National Energy Research Scientific Computing Center (NERSC), a U.S. Department of Energy Office of Science User Facility located at Lawrence Berkeley National Laboratory, operated under Contract No. DE-AC02-05CH11231 using NERSC award FES-ERCAP0025846. Oak Ridge National Laboratory is managed by UT-Battelle, LLC, under contract No. DE-AC05-00OR22725 for the US Department of Energy.

This work depended on the collaboration and input from many parties. The health and safety staff at Oak Ridge National Laboratory have been invaluable for ensuring the safe conduct of the beryllium work and for their assistance with developing plans for future work for this and other beryllium-related work. As such the authors wish to acknowledge April Case and Susan Fiscor.

ABSTRACT

This technical report is in submission of completion of the Level 2 milestone number M2AT-23OR1101022, titled "Be_2C synthesis, properties, and ion-beam irradiation damage characterization," for the AT-23OR110102 "Beryllium Carbide as moderator for MSRs—ORNL" WBS number 2.04.11.01 within the larger Advanced Reactor Technologies program at Oak Ridge National Laboratory. This effort is an initial study of the viability of Be_2C as a future neutron moderator for molten salt reactors and other high-temperature reactors. The research was awarded funding in September 2022, and this Level 2 milestone presents the work that completed by the end of July 2023. This work is a collaboration between Oak Ridge National Laboratory and the University of Michigan. This report discusses the status of this effort and planned work before the final reporting.

1. INTRODUCTION

This program was started under the Advanced Reactor Technology (ART) Molten Salt Reactor (MSR) program in September 2022. This effort resulted in a Level 2 milestone by the end of August 2023. The initial funding of $500k supports the preliminary studies of the possible use of Be_2C as a future neutron moderator for molten salt reactors. This current work is a collaborative effort between the US Department of Energy's Oak Ridge National Laboratory (ORNL) and the University of Michigan (UofM).

The effort at ORNL focuses on four tasks: Be_2C procurement, high-temperature stability testing, irradiation damage modeling studies, and degradation of Be_2C in the presence of hydrogen. ORNL purchased machined Be_2C solid specimens and high-purity powder from Materion Brush Inc. (Materion) in Elmore, Ohio. ORNL and Materion collaborated on the development of a sectioning plan for a previously manufactured solid body of Be_2C. Materion shipped the purchased materials to ORNL and UofM the first week of June 2023. As such, the experimental work presented in this report is a summary of work performed in the 2 month period of June and July 2023.

The effort at UofM focuses on two tasks: (1) development of the capability to perform ion irradiations of Be_2C and (2) installation of a hot isostatic press to facilitate future production of solid Be_2C bodies from high-purity Be_2C powder. The first task is broken down into multiple subtasks, including working with safety personnel at UofM to develop a personnel-monitoring plan, developing an encapsulation technique to reduce the risk of exposure to beryllium during specimen handling and irradiation, preliminary irradiation stage design and proof-of-concept irradiations on specimens that do not contain beryllium, and preliminary irradiation of Be_2C specimens.

2. BACKGROUND

Because of its moderating efficiency and low absorption cross section, Be_2C has long been recognized as having high potential to serve as a high-temperature tolerant neutron moderator [1]. It could serve as an alternative to graphite in many situations owing to its improved neutronic performance (greater slowing power) and potentially much better radiation damage characteristics. However, as a neutron moderator, Be_2C is much less mature than graphite. Graphite is the only proven, fuel-salt-compatible moderator; however, graphite has limited displacement damage tolerance and is not a volumetrically efficient moderator. The neutron moderation length in carbon is more than 60 cm vs. less than 6 cm in water [2]. Radiation damage to graphite has long been a known design issue for MSRs. Radiation damage to graphite was central to ORNL's decision in 1967 to shift from a dual- to single-fluid MSR and was a central rationale in halving the power density of the molten salt breeder reactor's conceptual design [3, 4]. Furthermore, graphite that has been exposed to fuel salt represents a significant contaminated waste stream.

To become a useful moderator material for MSRs, Be_2C would require technology development and maturation. Available Be_2C pieces are brittle and vulnerable to thermal stress cracking. Moreover, Be_2C is toxic, moisture sensitive, and chemically reacts with uranium. However, potential pathways exist to mitigate or even exploit Be_2C characteristics that have been perceived as disadvantageous, except for its toxicity. Much as with other carbides, fiber reinforcement may reduce brittleness [5]. A methanide, Be_2C decomposes into a hydrocarbon gas (methane) in the presence of hydrogen. One of the most difficult radionuclide containment challenges at MSRs is tritium. Conversion of tritium into methane would be useful at MSRs because tritium is the only radionuclide with significant potential to escape under normal operating conditions. Methane is readily trapped and does not diffuse through structural alloys. By contrast, Be_2C is chemically compatible with coolant salts but would require a protective layer (e.g., NbC) to be used directly with uranium-bearing salts. The most significant remaining uncertainty preventing the widespread use of Be_2C as a high-temperature neutron moderator is the lack of information on its radiation damage characteristics.

Like the exceptionally radiation damage resistant fluorite-type crystals (e.g., UO_2)—except with anions and cations reversed—Be_2C has an antifluorite crystal structure. A related antifluorite crystal (Li_2O) has also been shown to have high radiation damage tolerance [6, 7]. Although, the analogous material performance information provides a rationale for expending resources to assess of the radiation damage characteristics of Be_2C, the radiation damage characteristics of Be_2C remain speculative because only low-displacement irradiations have been performed [8-10]. With multiple, small cross-section, gas-generating threshold reactions, Be_2C does not activate substantially (Figure 1). Consequently, Be_2C pieces exposed to energetic neutrons may need to be occasionally baked out at temperatures above 1,000°C throughout the plant lifetime to remove gases that do not release at operating temperature but do migrate to form gas-stabilized voids.

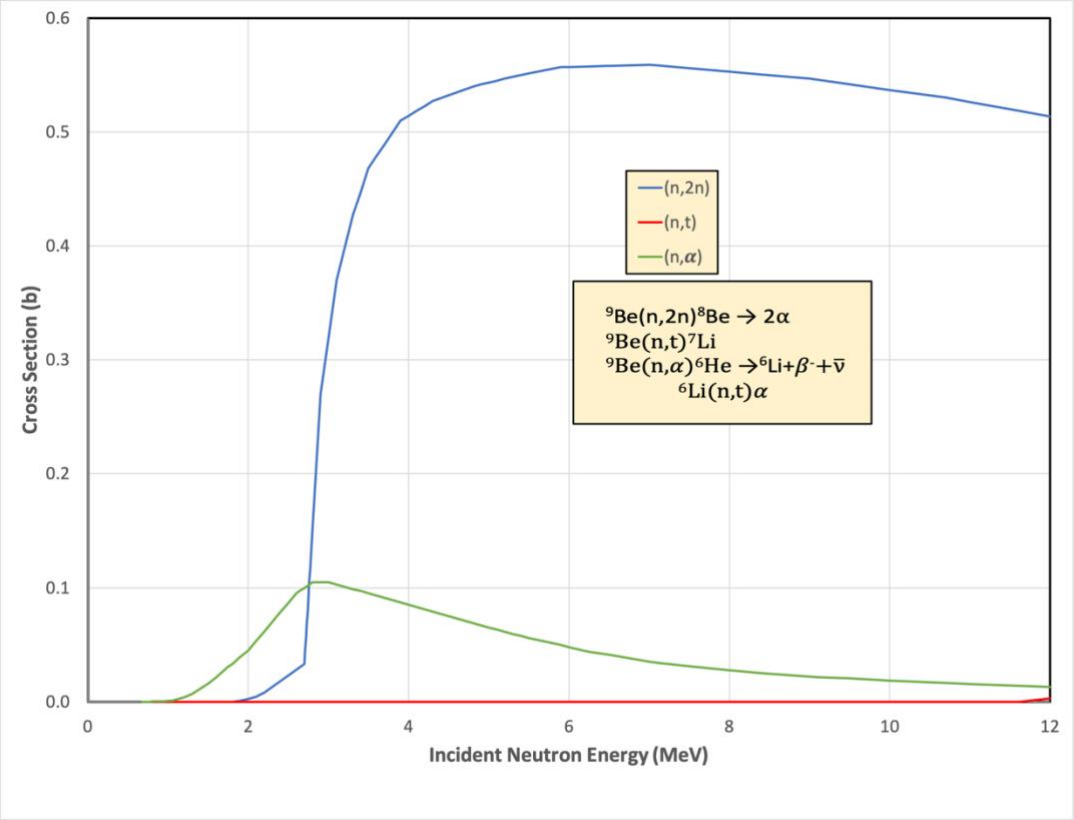


Figure 1. Beryllium gas-generating reactions (data from ENDF/B-VIII.0).

Part of the rationale for lack of prior development of Be_2C is the lack of need in gas-cooled reactors. Radiation damage sensitivity of graphite is of less concern to gas-cooled reactors because of their lower power density, which results from the decreased heat transfer provided by gas-phase coolants as opposed to halide salts. Additionally, Be_2C may be useful as a material for surrounding fusion plasmas. The major concern for use of Be_2C in fusion is that all fusion neutrons are born with high energy, and the Be_2C cross section for gas generation increases with increasing neutron energies. The major concern in fusion reactors is whether the generated gases are sufficiently mobile at operating temperature to result in gas-stabilized voids but not sufficiently high to release. Whereas gas generation is an issue in MSRs, fission neutrons have an energy distribution of 2–5 MeV and below, resulting in fewer neutrons with sufficiently high energy to generate gaseous atoms.

3. BERYLLIUM CARBIDE PROCUREMENT

Few places have the knowledge or physical capabilities to create solid pieces of Be_2C. Materion recently developed a method to produce a high-purity Be_2C powder. In anticipation of this work, Materion formed this high-purity Be_2C powder into a 1.5 in. diameter by 1 in. tall puck. Materion agreed to sell the compacted material to ORNL and UofM in support of this work and to cut this puck into shapes and sizes necessary for this effort. In addition to the purchase of this solid piece of Be_2C, 50 g of high-purity Be_2C powder is being purchased that UofM will use for hot isostatic press testing.

The cutting/sectioning plan for this puck was developed by ORNL with input from the engineers at Materion. The final sectioning and cutting plan is provided in APPENDIX A. Pieces to be cut from the puck include rods with lengths parallel (page 4 of cutting plan) to and perpendicular (pages 8, 10, and 11 of cutting plan) to the puck diameter. These lengths can be used for coefficient of thermal expansion measurements and flexural strength testing. The small capsule coupons (page 6 of the cutting plan) are used to study the high-temperature stability of this material in an inert atmosphere. Small specimens will be used to measure the interaction of Be_2C with oxygen, hydrogen, and moisture in the ORNL skimmer (page 7 of the cutting plan). Finally, small square coupons were cut that will be used by UofM for ion irradiation (page 10 of the cutting plan).

High-strength ceramics can pose challenges for cutting and machining without the added complexity of the reactivity of Be_2C. Therefore, because other traditional cutting methods do not work, all parts were cut by a slow-speed diamond saw while submerged in oil to prevent decomposition caused by exposure to humidity and oxygen. During cutting, specimens for thermal stability testing could not be drilled with a central hole as planned because Be_2C is extremely hard and brittle. Instead, the decision was made to use the diamond saw to cut slits into one side of the specimens to attach them with wire to the capsule lids. After cutting, Materion packaged the specimens into vacuum bags to reduce the likelihood of decomposition during shipping.

The finished specimens were received by ORNL and UofM in early June 2023. Once received, ORNL transferred the specimens into an inert-atmosphere glove box. The specimens were shipped in vacuum-sealed packets, and the packets were opened in the glove box only when needed for experiments. The as-packaged specimens are pictured in Figure 2 though Figure 6. All the specimens had a white coating—likely a thin layer of BeO—which was not uniform (Figure 5). Bag B2 is the only specimen bag that has been opened in the ORNL glove box because those specimens were selected for the thermal stability testing. Figure 7 shows a picture of these specimens after they were removed from the bag. Included is a higher magnification picture of one of these specimens showing the BeO surface layer. The glove box is not currently equipped removing the BeO layer, so the specimens were used without attempting to remove the BeO film.

Figure 2. Be_2C specimens as received from Materion.

Figure 3. Thermal stability specimens.

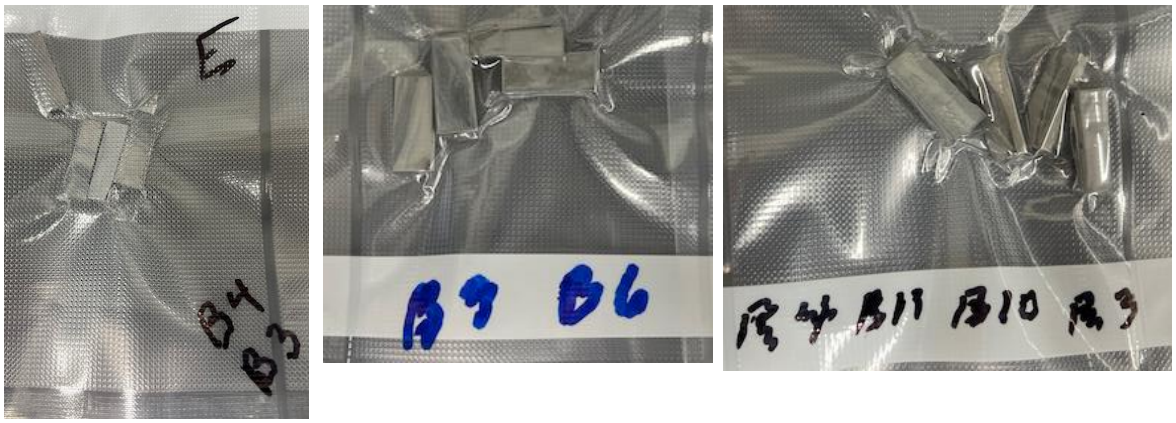


Figure 4. Bars with length parallel to puck thickness.

Figure 5. Bars with length parallel to puck diameter.

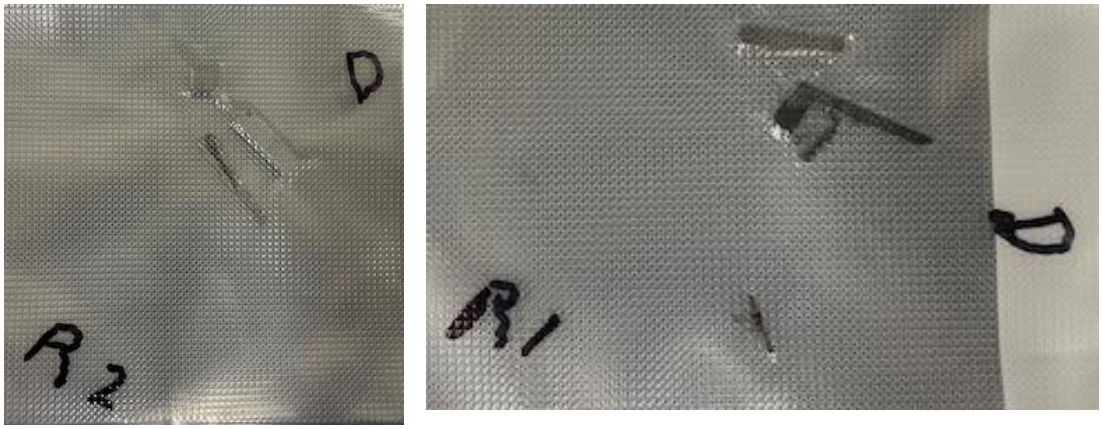


Figure 6. Small pieces for skimmer work.

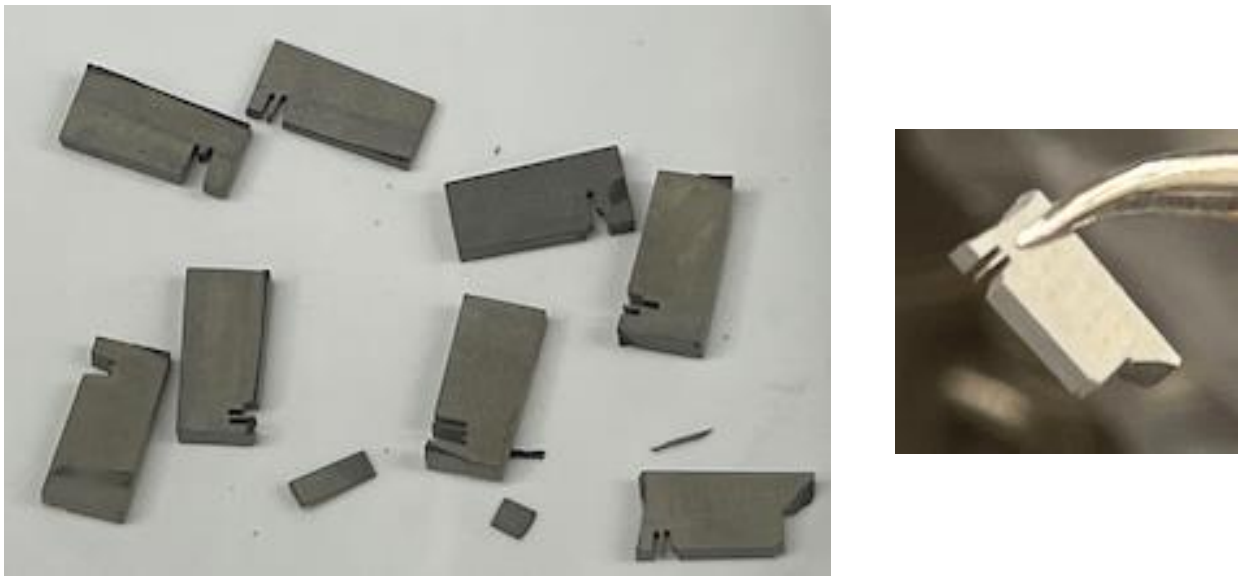


Figure 7. Bag B2 specimens selected for high-temperature thermal stability testing (left) and close-up picture of one specimen, highlighting the BeO layer (right).

4. FIRST-PRINCIPLES STUDY OF BULK AND RADIATION DEFECT PROPERTIES OF BE_2C

Density functional theory (DFT) was applied to study bulk properties, electronic structure, and point defects in Be_2C. The results regarding bulk properties and electronic structure are similar to those reported earlier, whereas properties of point defects in neutral states were obtained for the first time. Antisite defects, vacancies, and interstitial atoms were modeled, and their formation energy was estimated with high accuracy using large computational cells. Next, the interstitial configurations in beryllium and carbon subsystems were modeled, indicating that beryllium is stable in the octahedral configuration, whereas carbon interstitial is stable in the form of a C–C <100> dumbbell. A future modeling program is discussed for developing multiscale approaches for predicting Be_2C under fusion-irradiation conditions.

4.1 METHOD AND MODELING APPROACH

Electronic structures and energies have been calculated via DFT [11] using the generalized gradient approximation and the Perdew–Burke–Ernzerhof (PBE) [12] parametrization of the electronic exchange-correlation functional. Total energies and electronic structure were calculated using the plane-wave basis projector augmented-wave approach [13], as implemented in the Vienna Ab-initio Simulation Package (VASP) [14]. Supercells of three sizes were used to investigate different effects. The small supercell, 3 × 3 × 3 (324 sites), is used for bulk, electronic, and point-defect properties. Medium, 4 × 4 × 4 (768 sites), and large, 5 × 5 × 5 (1,500 sites), supercells were tested to ensure that point-defect structure and energy properties converge with respect to cell size. The large supercell will also be used to model Frenkel pairs and their stability vs. vacancy–interstitial separation and possible defect clusters. All calculations were made using the inner-energy cutoff E_{cut} = 450 eV. Small and medium supercells were modeled with 4 × 4 × 4 k-points, whereas the large supercell was modeled with 2 x 2 x 2 k-points. For the ionic relaxation, 10^{-6} eV energy convergence criterion was used. The ground-state phase of Be_2C is the face centered cubic (fcc) structure with the Fm–3m space group. In this lattice, two sublattices are distinguishable: the carbon fcc and internal simple-cubic beryllium sublattice. Together they form the antifluorite structure, as shown in Figure 8. This Be_2C structure was relaxed with fully optimized volume, shape, and ion positions. The equilibrium lattice parameter at 0 K was defined as a = 4.302 Å, which is well consistent with room temperature experimental results for which a = 4.32 Å [15], and recent Quantum Espresso calculations, for which a = 4.33 Å [16].

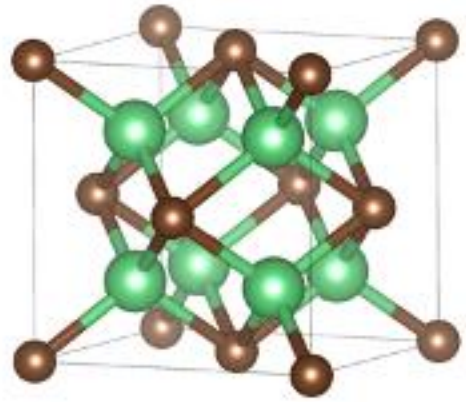


Figure 8. Atomic structure of antifluorite Be_2C structure (Be – green, C – brown).

The formation energy of Be_2C is estimated as $E_f = -0.214$ eV/atom, which is consistent with the literature [15]: $E_f = -0.231$ eV. The projected density of state calculated in the small supercell is presented in

Figure 9. The estimated Fermi energy of E_{Fermi} = 6.271 eV and the band gap E_g = 1.212 eV are also consistent with earlier calculations of 1.16 eV [11] and 1.22 eV [16], respectively.

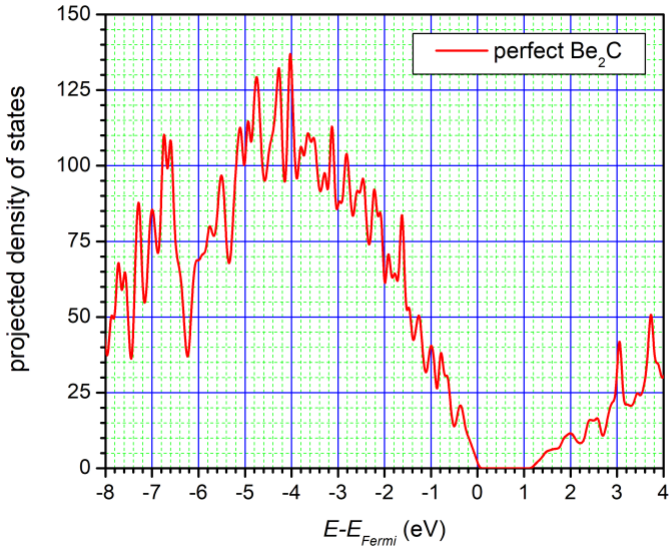


Figure 9. Projected density of states of Be_2C calculated for the small (3 × 3 × 3) supercell.

Defects in materials can be formed in different ways, such as temperature, deformation, or irradiation by energetic particles. Defects formed as a result of permanent ambient conditions such as temperature are native and coexist at thermodynamic equilibrium. These defects are important for understanding properties and evolution under equilibrium or relaxation toward equilibrium conditions such as diffusion, segregation, or phase transformations. Here, vacancies in beryllium and carbon sites (noted as V_{Be} and V_C respectively) as well as antistites of beryllium in carbon sites (noted as Be_C) and carbon in beryllium sites (noted as C_{Be}) were considered native defects. Native defects are expected to form during material fabrication, and their concentration depends on the equilibrium conditions considered. To estimate these concentrations correctly, defect energies must be calculated for the applicable charge states, and their minima should be used for thermodynamic equilibrium estimations. A literature search revealed no discussion about the possible charge states for the native and radiation defects in Be_2C, thus only neutral defects have been considered so far.

However, under irradiation conditions, defects are formed as a result of ballistic effects, and their evolution does not follow thermodynamic equilibrium conditions. Moreover, the charge states of primary damage defects, such as vacancy and interstitial atoms and their small clusters, are unidentified and probably depend on the irradiation conditions for ions, electrons, and neutrons combined with bulk and local composition. Interstitial atoms are typical examples of radiation-induced defects and are not included among the native defects considered for equilibrium conditions because of their high formation energy. No information on interstitial atoms in Be_2C has been reported so far; therefore, the current study reports the possible interstitial configurations for the first time.

Within supercell defect representation formalism, the formation energy $E_D^f(q)$ of a defect D in charge state q in Be_2C is expressed as follows:

$$E_D^f(q) = E_{defect}^{tot}(q) - E^{tot}(bulk) - n_{Be}\mu_{Be} - n_C\mu_C + q(E_v - E_F),$$

where $E^{tot}_{defect}(q)$ is the total energy of the supercell containing the defect in the charge state q, $E^{tot}(bulk)$ is the total energy of a similar supercell of the perfect bulk crystal, n_{Be} and n_C are the numbers of beryllium and carbon atoms added to or taken from the bulk crystal to create the considered defect, and μ_{Be} and μ_C are the corresponding chemical potentials. The value E_v represents the energy at the valence-band maximum of the defect-free system (here it is taken as energy $E_v = E_{Fermi} = 6.271$ eV; Figure 9). The value E_F is the current Fermi energy relative to E_v. In the case of vacancy, the corresponding n_{Be} and n_C are equal to −1, whereas for interstitial atoms, the corresponding n_{Be} and n_C are equal to +1. Here, chemical potentials were defined for beryllium—$\mu_{Be} = -3.765$ eV—and carbon—$\mu_C = 9.218$ eV—from modeling the ground states of beryllium in hexagonal close packed structure and carbon as graphite phase. Energies of pure beryllium and carbon, as well as that of the Be_2C unit cell used to estimate chemical potentials and the compound formation energy are listed in Table 1.

Table 1. Energy properties for pure components and Be_2C systems used to estimate chemical potentials μ_{Be} and μ_C: formation and cohesive energy of Be_2C and energy formation of antisites

Configuration	E_{rel} (eV)	$E_{formation}$ (eV)	Chemical Potential (eV)
Perfect Be_2	−7.5306	Be_2+C = −16.7482	μ_{Be} = -3.7653
Perfect C_4	−36.8703		μ_C = 9.2176
Perfect B_2C unit cell	−17.4021	−0.654 eV/molecule	
Perfect B_2C 3×3×3 supercell	−1,879.4310 eV		
Antisites			
Be in C	−1,868.5683	5.410	
C in Be	−1,876.7918	8.902	
C in Be + Be in C	−1,870.7276	8.703	$E_{binding}$=5.609 eV

4.2 RESULTS

The simplest defects are antisites, and their energies calculated in the small supercell are listed in Table 1. Both antisites have quite high formation energy and very high binding energy if the opposite antisites are created next to each other.

The next simplest defects are vacancies in different sublattices. Monovacancies were modeled in all three supercells, and their energies at neutral states are presented in Table 2.

Table 2. Neutral state formation energies of vacancies modeled in different subsystems and supercell sizes

Configuration / cell size	3x3x3 (324 sites)	4x4x4 (768 sites)	5x5x5 (1500 sites)
C-vacancy	5.321 eV	4.645 eV	4.679 eV
Be-vacancy	1.891 eV	1.819 eV	1.851 eV

Interstitials are the most complicated defects among point defects in the B_2C system. Interstitial atom structures are much more complicated than a monoatomic fcc lattice and have a great number of metastable configurations. About ten configurations were modeled for each type of interstitial atom, beryllium or carbon, based on expectations for fcc lattice such as dumbbells and crowdions along <100> and <110> directions as well as octahedral configurations. Some of them relaxed into the same configurations, and some relaxed into high-energy metastable states. The most stable configurations were found to be <100> dumbbell for carbon and octahedral configuration when the beryllium atom relaxed in

the carbon plane and positioned exactly along the <111> mixed direction. Formation energies of these configurations in different modeled supercells are presented in Table 3, and their configurations in the large cell are presented in Figure 10 through Figure 13.

Table 3. Neutral state formation energies of Be and C interstitial atoms modeled in different supercells.

Configuration/cell size	3 × 3 × 3 (324 sites)	4 × 4 × 4 (768 sites)	5 × 5 × 5 (1,500 sites)
C: <100> dumbbell	6.911 eV	7.158 eV	7.128 eV
Be: octahedral	5.931 eV	5.962 eV	5.829 eV

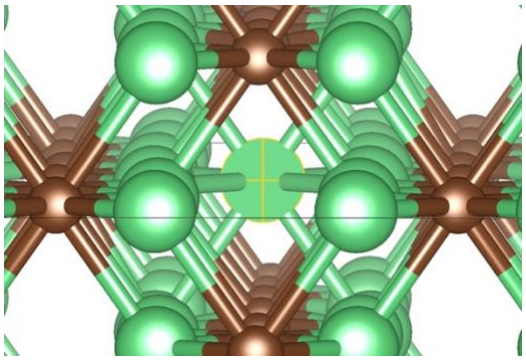


Figure 10. The most stable configuration for Be interstitial atom. A beryllium atom relaxes exactly in the center of octahedra (formed by the four nearest beryllium atoms in the same (001) plane and two carbon atoms above and below). Beryllium–interstitial creates eight new bonds with surrounding beryllium atoms; each is 1.998 Å length.

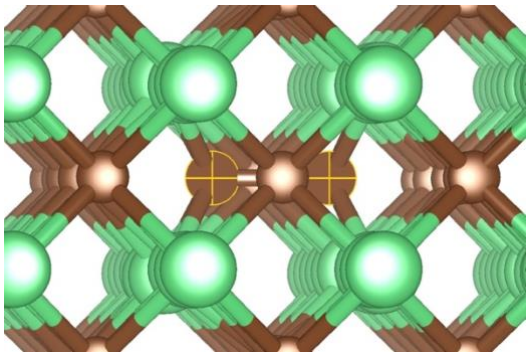


Figure 11. The most stable configuration for a carbon interstitial atom. Two carbon atoms create a symmetric <100> dumbbell with the distance between them 1.361 Å. Each carbon atom creates four bonds of length 1.696 Å with the nearest beryllium atoms.

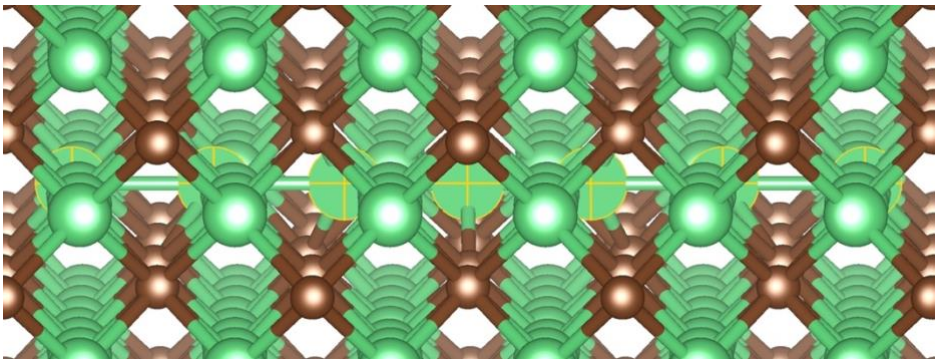


Figure 12. The metastable configuration for a beryllium interstitial atom. A beryllium–crowdion configuration along <100>. The seven beryllium atoms near the crowdion center shown by larger, yellow-crossed symbols are connected by newly created Be–Be bonds, the shortest of which is of length 1.705 Å.

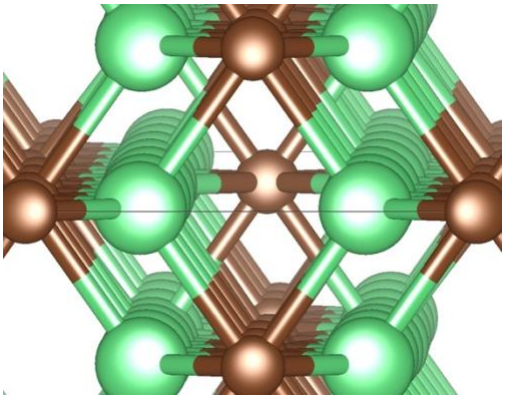


Figure 13. The metastable configuration for a carbon interstitial atom. A carbon atom relaxes exactly in the center of the octahedra (formed by the four nearest beryllium atoms in the same (001) plane and two carbon atoms above and below). The carbon interstitial creates eight new bonds with surrounding beryllium atoms, each is of length 1.895 Å.

4.3 CONCLUSIONS AND DIRECTIONS FOR FUTURE DFT-BASED MODELING WORK

The first DFT modeling of point defects in Be_2C has demonstrated that a supercell of a few hundred atoms can reproduce defect-formation energy with reasonable accuracy. Investigation of size dependence shows that even the smallest modeled cell, 324 atoms, reproduces qualitatively accurate and quantitatively reasonable properties of interstitial defects. For vacancies and antisites, this cell size provides accurate formation energy. In general, beryllium defects achieve better than carbon defect convergence, and, even in the smallest considered supercells (324 atoms), their energies are defined quite accurately. Carbon defects are more sensitive to the supercell size, and their energy converges in the medium-sized supercell (768 atoms).

In general, carbon defects need higher energy for their formation. Thus, formation energy of beryllium vacancies (E_{Be}^{f}=1.819 eV) assumes a thermodynamically equilibrium concentration compatible with that in refractory metals. By contrast, a carbon vacancy with E_{C}^{f}=4.465 eV is not likely to be formed thermally.

At this stage, the most interesting finding was that, among many different interstitial atoms configurations, two configurations were much more stable than others, that assumes a complex atomic transport mechanism under irradiation via interstitial migration. These are beryllium interstitial atom in

the octahedral configuration, as shown in Figure 10, and carbon interstitial atom as a C–C dumbbell along the <100> direction. Other carbon and beryllium interstitial configurations were found to be either unstable or have a high (>3–5 eV) formation energy.

According to the calculated electronic structure, Be_2C is a weak semiconductor (Figure 9) with a relatively narrow band gap of 1.2 eV, which assumes the existence of stable, charged defects in both beryllium and carbon subsystems. Investigations of the possible charge states and the Fermi energy dependence of stable configurations are planned for each defect.

The complex character of the Be_2C lattice structure assumes a great variety of possible metastable interstitial configurations. The static modeling reported herein does not guarantee finding the most stable global configurations. To resolve this issue, DFT-informed molecular dynamics models will be created to simulate beryllium and carbon interstitial atoms' behavior at different temperatures. These simulations will elucidate the dynamically stable configurations, the diffusion mechanisms, and diffusion coefficients if the modeling capabilities will allow sufficiently long interstitial atom trajectories.

Mesoscale models for predicting radiation-induced microstructure evolution in Be_2C will be developed based on a kinetic Monte Carlo (kMC) approach. This technique assumes knowledge of defect transition states and their energies, interactions between different defects, and energy properties of different defect clusters such as vacancies, interstitial atoms, and secondary phase precipitates. These data can be obtained from the extended DFT modeling based on the current findings. Mesoscale kMC modeling will allow estimation of defect diffusion and atomic transport coefficients as well as evolution mechanisms of the possible extended defects formed under fission reactor irradiation conditions. These results will be compared with future experimental observations of Be_2C.

Bulk, point defects, and cluster properties obtained by DFT modeling will be used to assess the only published set of empirical interatomic potentials set for the Be-C system [17]. This assessment will enable a conclusion on the applicability of interatomic potentials from the literature [17] for larger scale modeling of radiation effects in Be_2C. This issue is important for the possibilities of using classical atomistic modeling to significantly enlarge the spatial and time scales of the studied defects and phenomena.

5. THERMAL STABILITY TESTING

The thermal stability of Be_2C is a paramount property that must be understood before this material could be used as a neutron moderator in the extreme environments that it would be exposed to in an MSR. These extreme environments would include high temperatures and molten salts. The first step to understanding the thermal stability of Be_2C is investigating its behavior when exposed to high temperatures in an inert environment. This section discusses this work and preliminary analysis of the results.

5.1 THERMAL STABILITY EXPOSURES

Three Be_2C specimens were used for these thermal stability exposures. The capsule configuration and sizes are the same as those used at ORNL for molten salt compatibility studies [18], but instead of using molybdenum for the inner capsule, 316L was selected because of material availability, ease of machining, and the lack of a corrosive fill gas. The configuration uses two capsules: the inner capsule contains the specimen, which is attached to the capsule lid by a piece of inert metal wire (i.e., molybdenum or platinum), and the outer capsule acts as a secondary containment (Figure 14). The specimen is attached to the inner capsule lid (Figure 15), and then the inner capsule is filled with the desired environment (high-purity argon for this work). Next, the inner capsule is welded shut via electron beam welding. Once welded shut, the inner capsule is put inside the outer capsule, which is then welded shut using the same electron beam welder. This entire procedure was executed in an inert atmosphere glove box. During normal operations, the measured oxygen content within the glove box is less than 20 ppm; therefore, it is reasonable to assume that this oxygen content is the oxygen partial pressure within the capsule for the thermal stability tests.

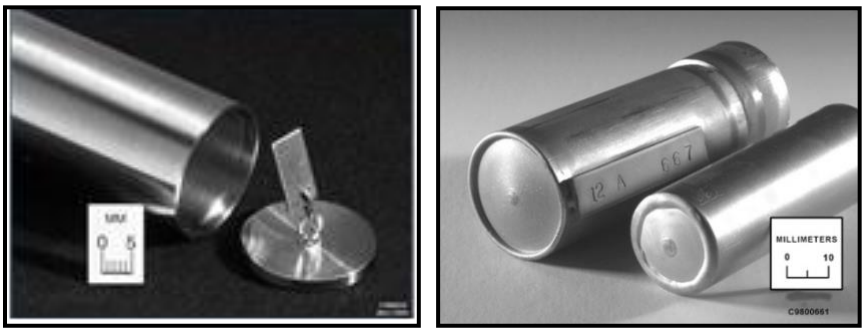


Figure 14. Capsules for thermal stability exposures. (left) Representative inner capsule with lid for specimen mounting and (right) inner and outer exposure capsules. Figures courtesy of J. Keiser.

The exposures were performed in a box furnace set to 650°C for exposure times of 1 day, 1 week, and 2 weeks. After the capsule completed the exposure time it was removed from the furnace and allowed to cool to room temperature. Once complete all the capsules were returned to the glovebox for opening and initial inspection. Upon capsule opening it was observed that all the specimen surfaces had changed from a dull gray with a light white powder coating to black (Figure 15). The masses of the specimens were measured before and after exposure to capture any changes. The results of the mass measurements are given in Table 4. Additional testing will be required to determine the source of the specimen discoloration, but it is suspected that Be_2C decomposed to BeO and C, and the discoloration of the surface is due to carbon that was left behind when BeO sloughed off the specimen surface.

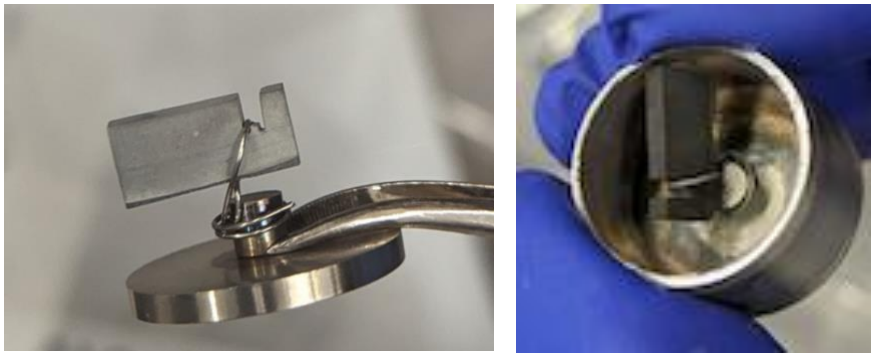


Figure 15. Specimen mounting. (left) A Be_2C specimen mounted on inner capsule lid before capsule welding and (right) a Be_2C specimen after thermal testing.

Table 4. Summary Be_2C specimen masses before and after high temperature thermal stability test, and calculated mass loss

Specimen	Exposure time	Pre-exposure mass (g)	Post-exposure mass (g)	Mass loss (%)
1	1 day	0.5596	0.5559	0.66
2	1 week	0.6283	0.6234	0.78
3	2 weeks	0.5960	0.5939	0.35

5.2 PHASE COMPOSITION

One concern related to the thermal stability of Be_2C is whether any of the material has changed phase or composition. The answer to this question was captured by performing X-ray diffraction (XRD) measurements on the Be_2C specimens that were slated for the inert atmosphere thermal-stability testing. To prevent unintended release of material and to prevent interaction of the Be_2C with ambient air, the specimens were enclosed in individual packets comprising two sheets of Kapton film that were taped shut with Kapton tape around the open edges (Figure 16) within the beryllium glove box. Kapton is an ideal encapsulation material for XRD because it is an amorphous material and therefore does not introduce extra peaks to the XRD spectra. This encapsulation technique was used for the XRD measurements both before and after the thermal exposures in the furnace.

Figure 16. Specimen encapsulation. (left) Kapton packet before loading, (center) with specimen loaded in glove box, and (right) fully sealed outside of glove box.

XRD measurements were performed using a Panalytical X'pert diffractometer equipped with a CuKα radiation (λ = 1.540598 Å) X-ray source. The scans were performed in a continuous θ–2θ setup from nominally 20° to 100° 2θ, with a scan rate of 0.0167°/s (~30 min scan time). All scans used 0.25° fixed

slits, 0.5° anti-scatter slit, and 0.04° Soller slits coupled with a 10 mm mask (beam length). A blank Kapton packet was run as a baseline measurement, and a zero-background plate was positioned below the specimens to remove any peaks from the metal specimen stage. For the phase identification procedure, a search match was conducted using the Jade software [19] and the ICDD database [20]. The XRD spectra for the three specimens are presented in Figure 17 through Figure 19, and the phase compositions are summarized in Table 5.

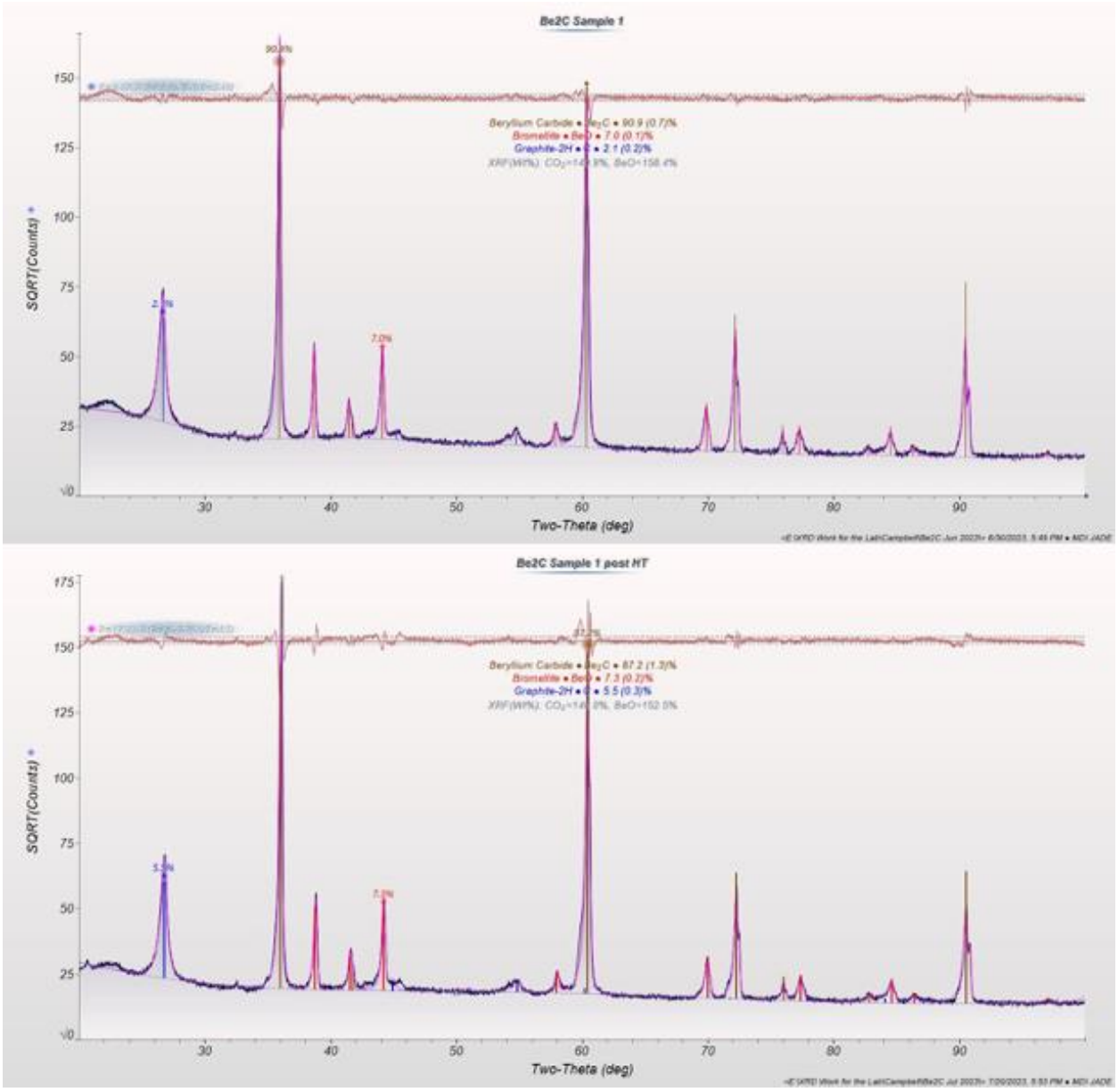


Figure 17. XRD scan of Be_2C sample #1. (top) Before and (bottom) after the high-temperature thermal-stability test.

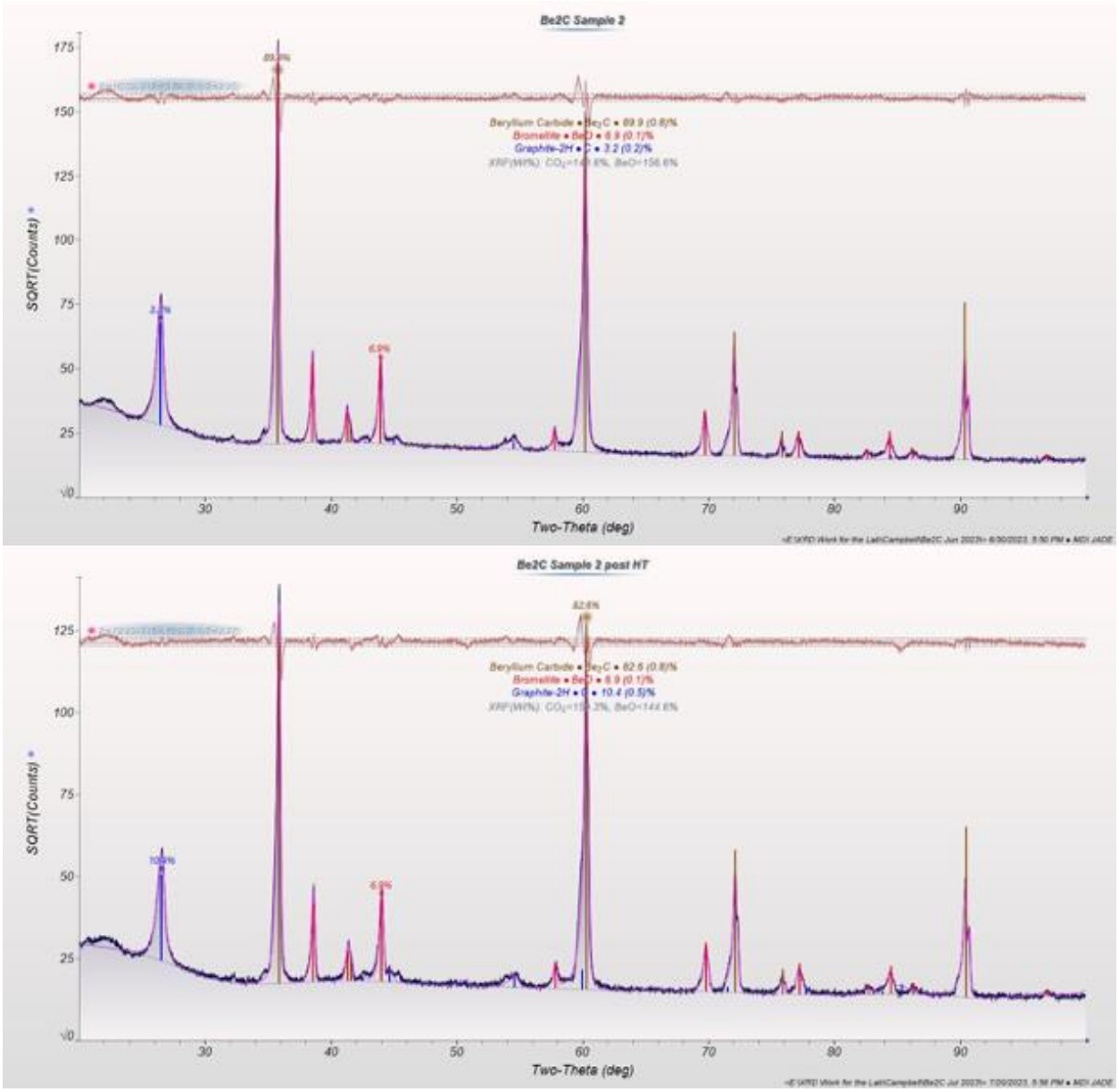


Figure 18. XRD scan of Be_2C specimen #2. (top) Before and (bottom) after the high-temperature thermal-stability test.

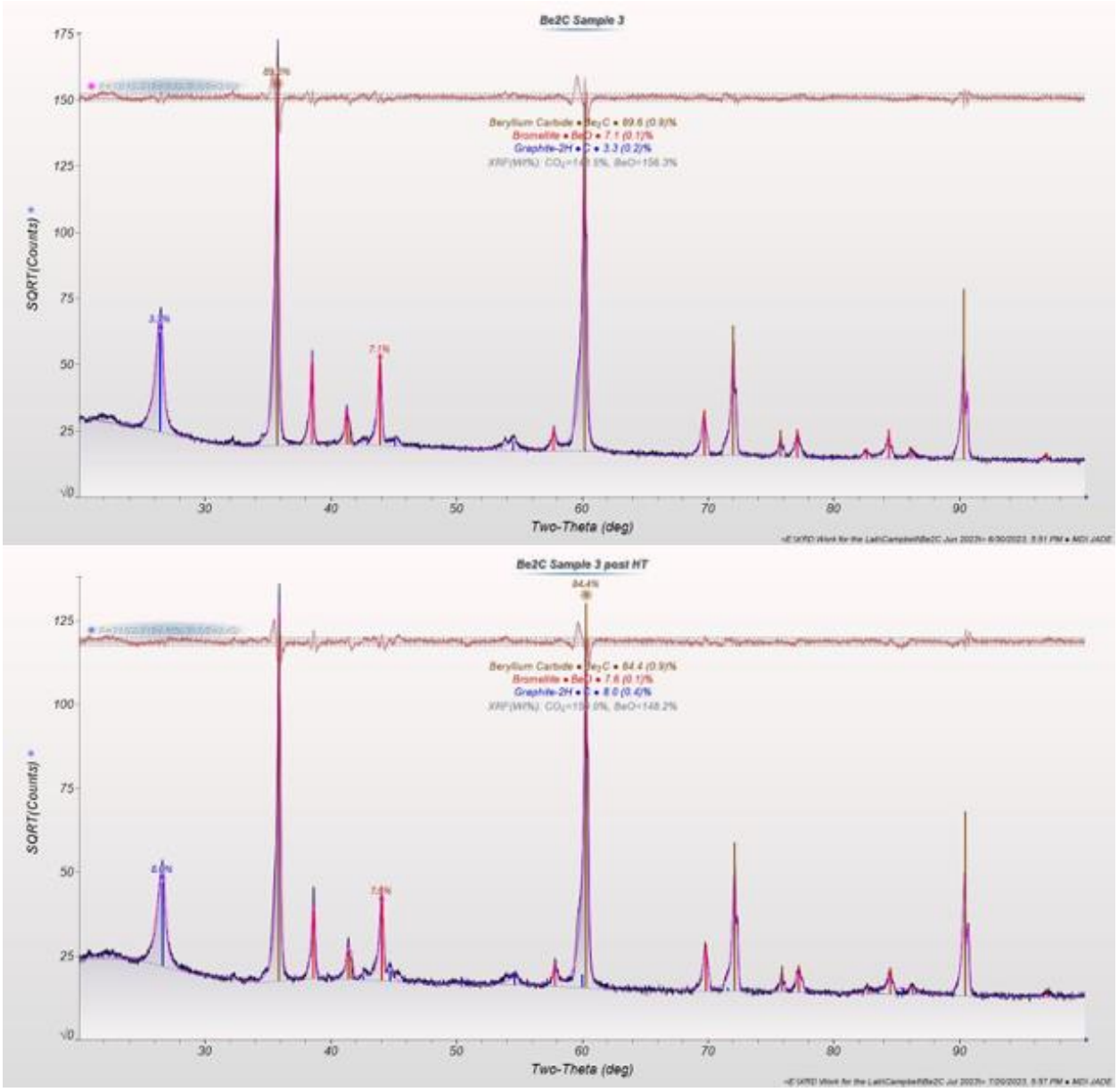


Figure 19. XRD scan of Be_2C specimen #3. (top) Before and (bottom) after the high-temperature thermal-stability test.

Table 5. Summary of phase composition of Be_2C specimens before and after the high-temperature thermal-stability test

Specimen	Exposure time	Be_2C phase before/after (%)	BeO phase before/after (%)	Graphite phase before/after (%)
1	1 day	90.2/87.2	7.0/7.3	2.1/5.5
2	1 week	89.9/82.6	6.9/6.9	3.2/10.4
3	2 weeks	89.6/84.4	7.1/7.6	3.3/8.0

Each specimen had a measurable increase in the percentage of graphite present after high-temperature stability testing. This result supports the previous speculation that some Be_2C decomposed to BeO and

carbon and could be the source of the specimen discoloration. The limited change to the BeO fraction from the XRD is suspected to be because the BeO layer that forms on the Be_2C specimens was easily removed, suggesting that the BeO formed but was then subsequently removed from the specimen surface before the post-exposure XRD measurement. This result was observed when the specimens were encapsulated within the Kapton packets before exposure: a white powder-like substance remained on the inside of the packet where the specimen had been (too difficult to capture in pictures).

6. ION IRRADIATION STUDIES

Neutron irradiations are multiyear efforts for any program, so any preliminary results require significant time and monetary investment. Ion irradiation can provide a preliminary investigation into the possible radiation stability of novel materials such as Be_2C. To better understand Be_2C response to radiation damage, experimental procedures, including heavy-ion irradiation, were designed. To safely irradiate Be_2C, infrastructure, including specimen-preparation capabilities, containments, medical plans, and preliminary irradiations, needed to be established. This section documents the progress made at UofM toward Be_2C handling and irradiation testing.

6.1 IRRADIATION EXPERIMENT DESIGNS

Beryllium is toxic when inhaled. In solid form, Be_2C is relatively safe; however, several processes in this experiment cause beryllium dust to be produced, thereby increasing the risk of inhalation. A two-layer containment system was designed and built to protect personnel from exposure. Standard operating procedures (SOPs) were also developed for safe handling of beryllium-containing materials.

6.2 GLOVE BOX SETUP

Materion shipped 14 slabs of solid Be_2C to UofM for irradiation. To fashion the specimens into the proper size with the proper surface finish, they will need to be cut and polished. These processes release particles of beryllium, which, if inhaled, can lead to chronic beryllium disease. To avoid this possibility, all necessary equipment was placed inside a glove box system designed specifically for safely handling beryllium-containing materials. The system consists of two glove boxes, as shown in Figure 20. The first glove box is filled with argon and is designed for handling materials susceptible to oxidation. This glove box contains a 1,100°C crucible furnace for conducting heat exposures. The second glove box, shown in Figure 21, is a containment-only box filled with air and kept at negative pressure. This box houses a slow-speed saw, mini-met polishing wheel with a full complement of metallography consumables, a gold sputter coater, and facilities for cleaning specimen surfaces with water and alcohol. All consumables such as cutting fluid and polishing suspension are oil because Be_2C is a methanide. The gloveboxes are both outfitted with several custom features designed for safe handling of beryllium, such as additional HEPA filters on the pipes used to backfill the antechambers. The boxes are joined by a common antechamber that can facilitate the transfer of items between them. When handling beryllium specimens, users wear personal protective equipment (PPE), including a HEPA-rated respirator as an added layer of safety. Details are included in the UofM SOP (APPENDIX B).

Figure 20. The dual glove box setup at the University of Michigan. The inert glove box is on the right, and the negative-pressure air glove box is on the left.

Figure 21. The specimen-preparation glove box at the University of Michigan.

6.3 SPECIMEN PREPERATION

The specimen-preparation glove box includes a precision saw, polisher, tripod transmission electron microscope (TEM) polishing kit, scale, furnace, and gold sputter coater so that all work can be done inside. For bulk irradiations, Be_2C specimens are first cut into 0.25 × 0.25 × 0.125 in. squares to fit within containment. Then, specimens are ground with a diamond pad for any finer adjustments and then polished

down to 1 μm. Before coating, specimens are cleaned using acetone. Specimens are then sputter-coated with up to 0.5 μm of gold on both sides to create a containment layer.

To minimize the amount of time needed to load the specimen onto the stage at the Michigan Ion Beam Laboratory (MIBL), the specimen is placed inside a secondary-containment molybdenum box with specific dimensions compatible with MIBL stages. The box is placed inside a plastic bag and a metal vessel for transportation. The vessel must be cleaned before being removed from the glove box to minimize any beryllium dust that may have adhered to it. At this point, the vessel is considered safe to remove from the glove box and hold without PPE.

6.4 CONTAINMENT

During irradiation, sputtering may introduce beryllium into the chamber, possibly contaminating the surfaces. The primary containment for the Be_2C is a gold coating that covers the entire ceramic and is applied in the glove box. Stopping and Range of Ions in Matter simulations and experimental safety tests have shown that a 250 nm layer of gold on all sides is sufficient to protect the Be_2C during testing. The safety tests were executed in extreme conditions with increased fluence and heating duration to provide a larger safety margin.

A secondary containment was made as a failsafe in case the primary containment were to fail, as shown in Figure 22. It consists of a three-layer molybdenum box that surrounds the coated specimen during irradiation. The first layer serves as a thermal plate in contact with the heater on the stage. This layer is the thinnest layer to ensure proper heat transfer and is large enough to have four thermocouples welded to it for monitoring during irradiation. The second layer is a holding plate designed to fit a 0.25 × 0.25 × 0.125 in. slab. The cutout is custom to the dimensions received by Materion and will apply even pressure to the Be_2C, reducing the chances of fracture. The final layer is a sputter plate that will reduce the direction of sputtered beryllium. A 4 mm diameter aperture at the center of the sputter plate allows the beam to pass through. This area is the only location where the gold-coated Be_2C is exposed to the environment. Several stages at MIBL have different designs for specimen loading, leading to several variations of the secondary containment. All variations have been tested and proven functional.

The scope of the project requires the use of several labs at different locations. Therefore, a transportation vessel is required. The vessel consists of five layers of containment, making it safe to store outside a glove box and move without PPE. Containment layers one and two are the gold coating and molybdenum boxes, respectively. These two layers are always present when specimens are removed from the glove box. Layer three is an airtight bag holding the prepped specimen, reducing exposure to air and moisture. The fourth layer is a plastic box equipped with four latches. Two latches are required to be open before the lid can be removed, reducing the chances of an accidental spill if dropped. The last layer of containment is a stainless-steel cylindrical vessel with three latches. The vessel is padded inside and requires two latches to be opened before the lid may be removed. With all layers properly packaged, the chances of an accidental spill are minimal. If a spill does occur, then lab members are equipped with respirators and a HEPA-rated vacuum to clean or quarantine the area. UofM Environmental Health and Safety (EHS) must be contacted if a spill occurs.

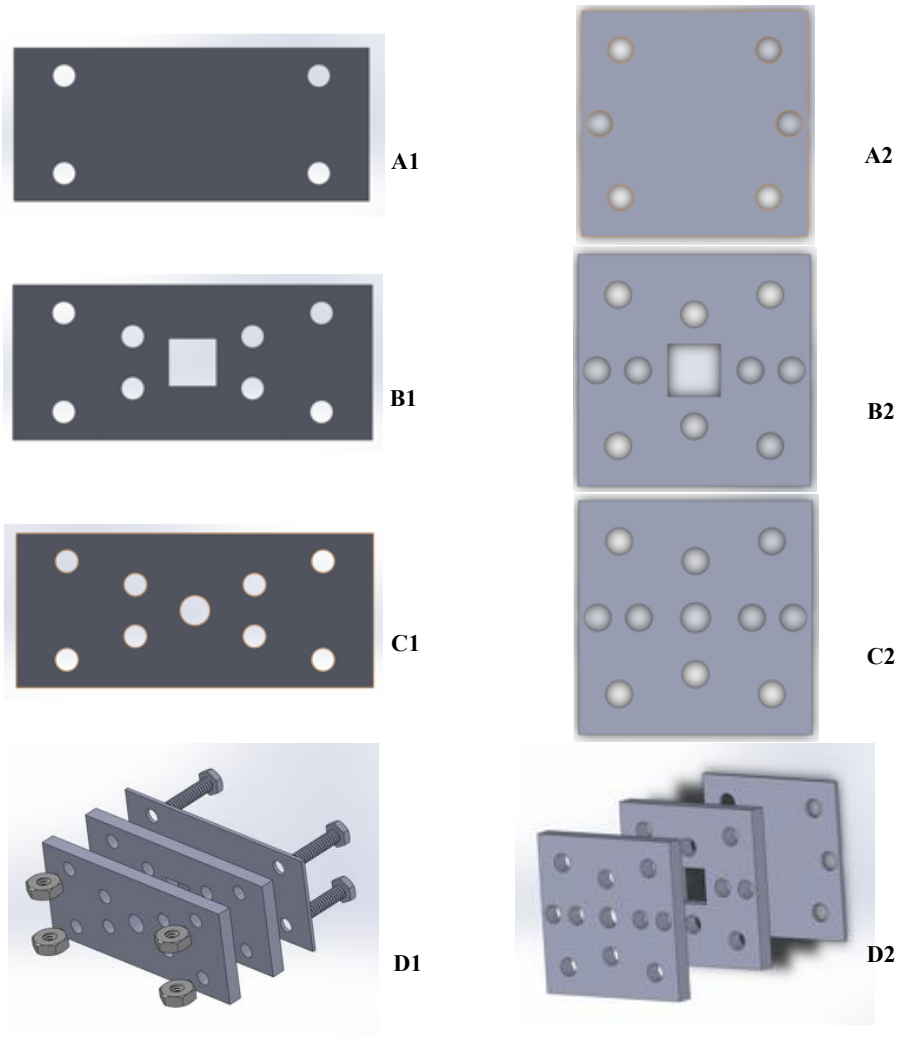


Figure 22. Secondary containment. (A) Thermal plate, (B) holding plate, (C) sputter plate, and (D) assembly; 1 fits a high-temperature stage and 2 fits a nickel stage.

6.5 MEDICAL PLAN

A detailed medical plan and SOP has been written and approved by UofM EHS for all steps of beryllium handling. The plan has been separated into four major sections: specimen preparation, irradiation, microcopy, and transportation. Two members of the group have been fitted for respirators so that they may offer help with any specimen work. The respirator includes two sets of HEPA filters rated to last a minimum of 1 year. The respirator fitting has been approved to last 5 years before requiring a new fitting. A voluntary blood test program is underway to monitor beryllium exposure every month for beryllium workers. The test begins with a baseline blood sample to see how sensitive the lab members are to beryllium. Following this baseline, a monthly blood test will be done, regardless of whether any beryllium was handled, to monitor exposure levels. Finally, frequent smear tests will be conducted on surfaces such as glove box antechambers, beamline chambers, and transportation vessels to confirm no contamination is present. The SOP is included in APPENDIX B.

6.6 LITHIUM OXIDE IRRADIATIONS

To prepare for the Be_2C irradiations, several preliminary tests were executed to better understand the necessary engineering safety precautions. These tests included two Li_2O and two SiC irradiations.

Two irradiations were performed with Li_2O specimens because of their common antifluorite crystal structure with Be_2C. The Li_2O specimens were sintered at Texas A&M University by spark plasma sintering in advance. The Li_2O specimens were irradiated with oxygen ions in the Multi-Beam Chamber at MIBL with a high-temperature stage. The achieved parameters are listed in Table 6.

Table 6. Achieved parameters from preliminary Li_2O tests

Parameter	Test 1	Test 2
Dose (dpa)	48.69	5.24
Damage rate (dpa/s)	2.00×10^{-4}	3.31×10^{-4}
Temperature (°C)	698.6 ± 3.3	698.0 ± 27.1
Current (μA)	0.244	0.251
Energy (MeV)	1.5	1.5
Time (h)	69.50	4.58
Beam area (cm^2)	0.25	0.25
Ion beam	O^+	O^+

Both tests revealed concerns with specimen loading that needed to be addressed before conducting Be_2C irradiations. Fracturing the specimen was problematic when loading to the stage because of the brittle nature of ceramics. Custom stage attachments were manufactured to prevent fracturing with further tests. The stage attachments were tested within the beamline and proved to be a viable solution to prevent fracturing. Figure 23 shows an image of the high-temperature secondary containment while in the beamline. The image was taken with the forward-looking infrared camera to ensure that the angle of the specimen was still visible. The boxes represent the areas of interest (AOIs) where temperature will be measured. Two AOIs were placed within the aperture that will give a temperature reading average for the top and bottom of the irradiation area.

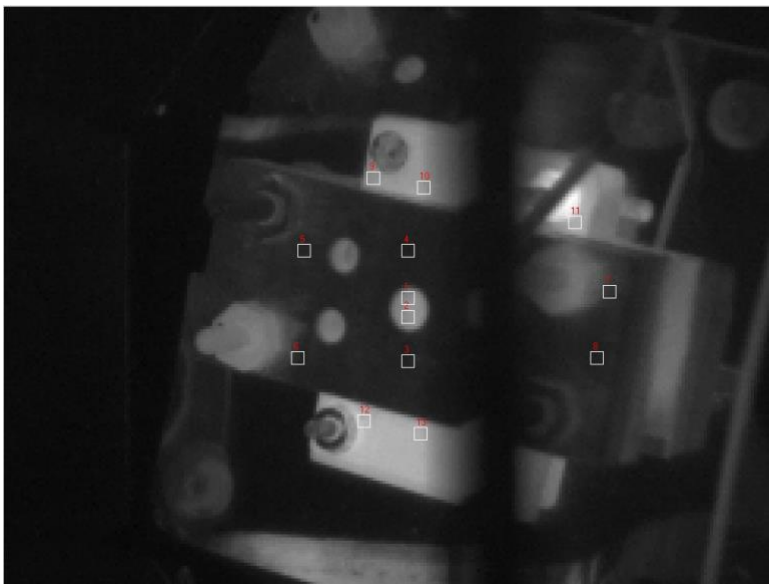


Figure 23. Forward-looking infrared image of high-temperature secondary containment within beamline.

6.7 SILICON CARBIDE IRRADIATIONS

Two SiC irradiations were executed to test the effectiveness of the gold-coating containment. The first specimen was cut and polished to a 1 μm finish followed by a three-layer gold coating. The coatings were 55, 250, and 350 nm thick and were irradiated at the same time. Specimen 2 was polished to 1 μm but coated with 150 nm of gold. Figure 24 illustrates both specimens with their different gold thicknesses listed on the faces.

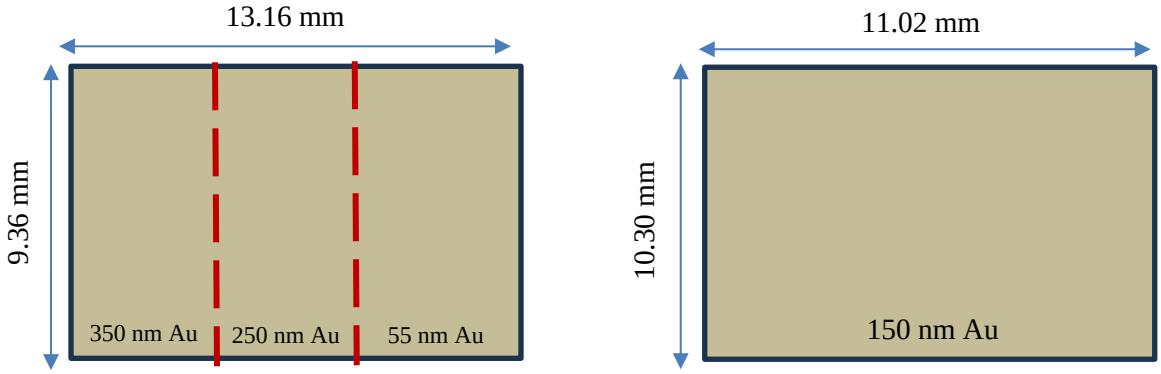


Figure 24. Silicon carbide irradiation specimens. (left) Test 1 and (right) Test 2.

The specimens were irradiated at 550°C with the highest fluence attainable to test the integrity of the coating. For safety reasons, the irradiations were conducted on beamline 2, which is more isolated than other beamlines in MIBL. Beamline 2 is limited to a nickel stage that is limited to a maximum temperature of 550°C. All other achieved parameters for both SiC tests are listed in Table 7.

Table 7. Achieved parameters from preliminary SiC test one and two.

Parameter	Test 1	Test 2
Dose (dpa)	35.06	10.04
Damage rate (dpa/s)	2.02×10^{-4}	2.34×10^{-4}
Temperature (°C)	547.8 ± 38	531.0 ± 42
Current (μA)	1.122	0.938
Energy (MeV)	9.00	9.00
Time (h)	48.17	12.23
Beam area (cm^2)	0.18	0.16
Ion beam	C^{3+}	C^{3+}

Scanning electron microscopy (SEM) was conducted on both specimens after irradiation to investigate the performance of the varying layers of gold coatings. The 55 nm thickness gold coating clumped, revealing large portions of the SiC. The 150 nm coating exhibited the same effect to a lesser extent. The 250 nm and 350 nm coatings exhibited no gold coating clumping for any portion of the irradiated area. The 150 nm coating was heated for a shorter duration than the rest of the layers because of time limitations. Figure 25 shows SEM images of all the layers at varying magnifications. The lighter regions represent the gold coating, whereas darker areas show the SiC surface. This analysis suggests that a coating of 250 nm or more would be sufficient for the primary containment of the Be_2C specimens.

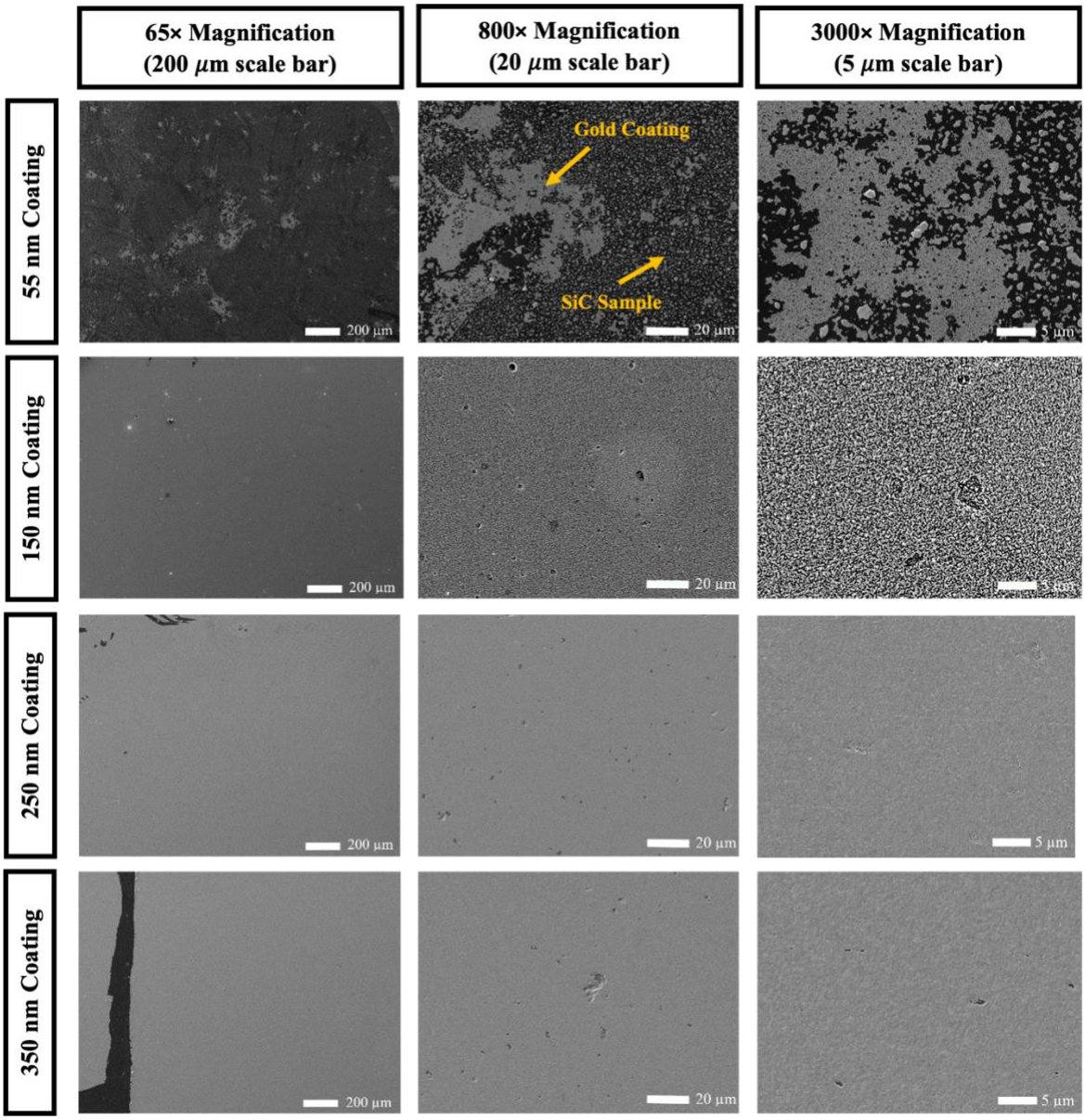


Figure 25. SEM imaging of 55 nm, 150 nm, 250 nm, and 350 nm gold coatings on SiC specimens after irradiation.

6.8 BERYLLIUM CARBIDE WORK

Materion supplied 14 Be_2C slab specimens, which have been safely placed inside a glove box. As of July 13, 2023, four of the specimens have been cut and polished to properly fit within the designed containment. The specimens will be coated with a 350 nm gold layer. One of the specimens will be designated for a nonirradiated safety test, and the other three will be used for irradiation experiments.

To observe the behavior of the Be_2C during heating, one specimen will be heated in the glove box furnace to 600°C for 24 h. The furnace will be in an argon environment so that specimen oxidation is minimal. The thermal test will ensure that the gold coating on the specimen will stick for the duration of the experiment. To determine whether any beryllium gas is emitted, volatilization will be checked by

measuring the weight of the specimen before and after the test. A smear test will be performed by EHS on the surroundings of the specimen to gather a more detailed measurement.

The first irradiation experiment was performed during the week of August 14, 2023. The actual conditions for the first experiment are listed in Table 8. The target parameters for the second and third ion irradiations (Table 8) will be kept constant with the first experiment except for the target total dose. A report on the results of the ion irradiation will be issued once the work is complete and the results have been analyzed.

Table 8. Target parameters of Be_2C ion irradiation.

Parameter	Experiment 1 Actual Conditions	Experiment 2 Planned Conditions	Experiment 3 Planned Conditions
Dose (dpa)	5.02	10	15
Damage rate (dpa/s)	1.21×10^{-4}	1.21×10^{-4}	1.21×10^{-4}
Temperature (°C)	500 ± 10	500 ± 10	500 ± 10
Current (μA)	0.211	0.211	0.211
Energy (MeV)	9.00	9.00	9.00
Time (h)	11.45	22.8	34.2
Beam area (cm^2)	0.0784	0.0784	0.0784
Ion beam	C^{3+}	C^{3+}	C^{3+}

Specimens will be characterized by SEM, focused ion beam, and TEM after irradiation. Initial foci will be on viewing amorphization, voids, and loops. All specimens will be saved and stored inside a glove box in case further viewing is required.

6.9 FUTURE WORK

Planning is underway to conduct in situ irradiation of TEM foils to enable observation of degradation phenomena in real time. Furthermore, in situ TEM makes characterization easier because the irradiated specimen is small, rendering the risk of beryllium contamination negligible.

7. CHEMICAL COMPATABILITY TESTING

The kinetics of Be_2C decomposition in the presence of oxygen, hydrogen, or moisture is not well understood. The final effort of this year was to perform a set of measurements to begin to understand these kinetics. This effort will utilize the Netzsch STA 409, or skimmer, that is located at ORNL and has been operational since 2019 [21]. The skimmer can be operated up to 2,000°C and provides a unique capability to measure the off-gassing of specimens in a reducing environment using an appended mass spectrometer (single quadrupole) while collecting thermogravimetric data.

The skimmer has not yet performed measurement with beryllium-containing materials. As such, the focus of the effort this year has been the coordination among researchers, equipment operator, and ORNL Environmental, Safety, Health, and Quality (ESH&Q) Directorate staff to develop a hazard analysis and procedure for future experiments involving beryllium-containing materials.

The hazard analysis in development by B. Henry and E. Paxton is not yet available for distribution but is included in the current draft state because key questions about specimen transport, handling, and waste have been addressed and resolved.

7.1 BERYLLIUM HAZARD ANALYSIS

The following analysis describes a collaborative project between the Materials Science and Technology Division and Nuclear Energy and Fuel Cycle Division using Be_2C and its associated beryllium controls. Work control will be split by group, with a beryllium exposure prevention plan (BEPP) in place for the associated tasks. The project involves taking three Be_2C coupons (less than 5 mg per coupon) and using a Netzsch skimmer (Figure 26) to perform in situ weight measurements at 650°C while monitoring for the presence of methane gas. This research is exploratory and is expected be a stand-alone project. If methane is detected, then a reaction within the beryllium carbide occurred, potentially releasing beryllium or BeO.

Figure 26. Netzsch skimmer setup in the laboratory.

7.1.1 Specimen-Handling Procedure

1. A surrogate sample not containing beryllium will be analyzed to help identify ramp up times and temperatures.
2. Coupons will be stored in the following locations during the experiment:
    a. Glove box in 4500S-C147 during sample prep stages
    b. Beryllium storage location in 4500S-S135 while waiting for sampling results for clearance to move.
        i. Coupons will be double bagged and labeled per SBMS requirements.
    c. Beryllium storage location in 4505-26 in a designated beryllium storage area
        i. Coupons will be double bagged and labeled per SBMS requirements.
3. Coupons will be prepared in 4500S-C147 by placing a coupon in a crucible and sealing the top with plastic wrap.
    a. The crucibles will be double bagged and sampled for release.
        i. When waiting for sample results, the crucibles will be stored in 4500S-S135 in a designated beryllium storage area.
4. Crucibles will be transported to 4505-26 and stored in a designated beryllium storage area.
5. Each crucible will be loaded into the skimmer for analysis.
    a. Each test will last approximately 4 h.
    b. A crucible will be placed on the sample holder, which is then raised into the coupling system, creating a closed system.
    c. Each test will be conducted at elevated temperature and pressure, burning off the plastic wrap, exposing the Be_2C coupon inside the system under a cover gas.
    d. After testing, the sample holder is lowered, and the crucible and Be_2C coupon are collected stored in a designated beryllium area.
6. After all samples are tested, the sample holder, crucibles, Be_2C coupons, and associated PPE (e.g., gloves, wipes) will be double bagged, labeled, and placed in a beryllium waste stream as described by waste services in Research Safety Summary 17606
    a. The crucible and beryllium carbide coupon will be discarded after each experiment.

Crucible preparation and sampling will be conducted in accordance with the Corrosion Science and Technology group BEPP. The following sampling records provide a history of sample results: SID16481, SID16502, SID16504, SID16564.

The skimmer analysis is new and will have a detailed assessment below to include controls with sampling requirements. This information will be carried into the BEPP.

Katie Johnson will be the skimmer operator. Other work may occur during analysis, but the skimmer is isolated in a radiation-control area, where no one else will enter.

7.1.2 Hazard Considerations:

1. Dermal exposure potential:
    a. Inside a glovebox, the coupons will be seated in a crucible and the top sealed with plastic wrap, then they will be sampled for release. Each crucible will be double bagged and labeled and will be removed from the bag just before skimmer analysis. Minimal potential exists for dermal exposure during this process. When the crucible is lowered out of the skimmer, the Be_2C coupon will be exposed to atmosphere, creating a potential for dermal exposure.
2. Airborne exposure potential:

    a. No airborne exposure is possible during the crucible-loading or skimmer-loading processes. The glove box and transport procedures prevent the coupon from being exposed to air, preventing any reactions that may create free beryllium or BeO. When the crucible is lowered out of the skimmer, the experiment parameters and the Be_2C coupon's exposure to air create the potential for free beryllium or BeO to be present on the coupon surface.
3. Co-located worker exposure potential:
    a. Other work may be occurring in the lab space during analysis. The lab has badge reader restricted access, so only authorized individuals may be in the area. The skimmer is the only piece of equipment located in a radiation-control area, creating a natural barrier that prevents anyone from accidentally entering the workspace. No expectation exists for beryllium exposure potential to others working in the lab space.

7.1.3 Control measures:

1. Engineering controls:
    a. A glove box is used during crucible loading and sealing, mitigating exposure potential. The skimmer is designed to be a closed system with HEPA filtration.
2. Administrative controls:
    a. Cover gas will flow in the skimmer after each analysis for an additional 15 min. A typical flow rate during normal use is estimated to create approximately 2 air exchanges per minute, allowing any free beryllium or BeO to be removed from the skimmer atmosphere. Crucibles loaded with Be_2C coupons will be immediately double bagged and stored after lowering out of the skimmer coupling system. This process is estimated to take less than 1 min per occasion. All project participants are trained on their respective pieces of equipment and are authorized beryllium workers, complete with medical surveillance requirements. All work tasks are identified and covered under a BEPP.
3. Personal protective equipment:
    a. Nitrile gloves are worn during all crucible-handling activities. Standard lab PPE—including safety glasses, close-toed shoes, lab coats, and long pants—is required, ensuring that no skin is exposed.

7.1.4 Skimmer sampling expectations:

1. Wipe sampling will be conducted on the skimmer before introducing any beryllium carbide to establish a baseline. Sampling locations will include the following:
    a. skimmer platform
    b. immediate surrounding platform area
    c. bottom of the furnace casing
2. Personal air monitoring will be conducted during sample analysis, specifically during crucible lowering and double bagging processes.
3. Post-project wipe sampling will be conducted to mirror baseline samples and capture any additional points of concern identified during analysis.
    a. Determinations regarding internal contamination and future beryllium considerations will be made after these results are obtained.

8. OTHER PLANNED WORK

Additional work was originally planned for this year's effort, but multiple challenges arose. As mentioned in Section 3, bar samples were cut from the solid body and were slated to be used for coefficient of thermal expansion measurements. The plan was to measure the thermal expansion parallel to and perpendicular to the puck thickness to capture whether any preferential orientation may have resulted from the forming process. The thermal expansion measurements were originally slated to be performed at Argonne National Laboratory in a dilatometer located inside a glove box that could measure beryllium materials. Owing to a lack of use and future interest, the dilatometer was removed from their glove box, and there are currently no plans to reinstall the system back into the glove box. Idaho National Laboratory was contacted; unfortunately they did not have any dilatometers set up within glove boxes that could perform this work. No other laboratories (national laboratory complex or at universities) are currently configured for thermal property measurements of solids containing beryllium.

9. SUMMARY AND FUTURE WORK

The environments neutron moderators will encounter in advanced nuclear reactor concepts are only becoming more extreme. These extreme conditions mean that many of the historically used materials may need to be replaced with materials not previously used or considered. In the case of MSRs, graphite may not be the ideal choice of neutron moderator because of its porosity (allowing salt to penetrate into the pores) and the inherent large volume of material necessary to moderate neutrons. One material that could be used to replace graphite is Be_2C. Unfortunately, very little effort has been undertaken in the past to study the use of Be_2C as a potential core structural and moderating material. Multiple reasons for this lack include the production of material that has a high chemical purity and limited inclusion of non-Be_2C phases (e.g., BeO or graphite), difficulty of machining a high-strength and brittle fully dense ceramic, material toxicity, and the high sensitivity to moisture that results in breakdown to the biological hazard BeO.

The experimental work performed at ORNL and UofM is preliminary and has not produced any results that would suggest a reason to not continue studying the potential of Be_2C as a new neutron moderator. The list of future work to continue this effort is long but not unobtainable. The first task will be discussions with Materion to develop powder and solid-body synthesis methods that will produce high-purity Be_2C. Some of these discussions will focus on development of advanced cutting and machining that will be required for more complex shapes, and others will look at the option to tailor the microstructure and materials properties. The preliminary results from ion irradiations will need to be evaluated to determine the feasibility of continuing ion irradiation studies and the development of a neutron irradiation program. A neutron irradiation program will require significant investment in facilities and equipment within the national laboratory complex that will be able to handle and measure the pre- and postirradiation materials properties of Be_2C samples in an inert and controlled environment. Experiments will also need to be designed to investigate the nature of the interaction and behavior of gases produced from neutron capture and transmutation (Figure 1) within the material.

Continuation of the modeling effort to expand from DTF calculations to molecular dynamic models will be necessary to begin modeling and understanding the effects of collision cascades in Be_2C. Modeling will then progress to kinetic Monte Carlo, which is suited for modeling defect behavior between collision cascades and on longer time scales. Later efforts will utilize cluster dynamics, discrete dislocation dynamics, and crystal plasticity to understand how larger features in Be_2C affects the response to irradiation damage and larger defect behaviors. One of the key long-term modeling efforts will have to focus on the behavior of the gasses produced from neutron capture and transmutation (Figure 1) within the material.

10. REFERENCES

1. Quirk, J.F., "Beryllium Carbide", in *The Reactor Handbook, Vol. 3 – Section I, General Properties of Materials*, 1955, United States Atomic Energy Commission, AECD-3647.
2. Kloosterman, J.L., "HTR Design Aspects", Available fom: http://www.janleenkloosterman.nl/gen4fin_201009.php.
3. Rosenthal, M.W., R.B. Briggs, and P.R. Kasten, "Molten-Salt Reactor Program Semiannual Progress Report for Period Ending August 31, 1967 (E.S. Bettis, Chapter 5 *Design*)", ORNL-4191, (1967).
4. Rosenthal, M.W., R.B. Briggs, and P.R. Kasten, "Molten-Salt Reactor Program Semiannual Progress Report for Period Ending February 29, 1968 (E.S. Bettis, Chapter 5 *Design*)", ORNL-4254, (1968).
5. Peacock, A.T., W.J. Haws, and M.A. Pick, "Development of beryllium-carbide fibre reinforced beryllium for fusion applications", Proceedings of the 15th IEEE/NPSS Symposium on Fusion Engineering-Supplement, 1993, 50-53.
6. Moriyama, H., S. Tanaka, and K. Noda, "Irradiation effects in ceramic breeder materials", *Journal of Nuclear Materials*, 258-263, (1998) 587-594.
7. Noda, K., T. Tanifuji, Y. Ishii, H. Matsui, N. Masaki, S. Nasu, and H. Watanabe, "Irradiation effects on lithium oxide", *Journal of Nuclear Materials*, 123, (1984) 908-912.
8. Maya, I., K.R. Schultz, R.F. Bourque, E.T. Cheng, R.L. Creedon, J.H. Norman, R.J. Price, J. Porter, H.L. Schuster, and M.J. Simnad, "Inertial confinement fusion reaction chamber and power conversion system study. Final report", UCRL-15750; GA-A-17842; ON: DE86006725, United States (1985).
9. Marion, R.H. and W.A. Muenzer, "Development of Be 2 C--graphite--UC 2 fuel for pulsed reactors", *ANS Transactions*, SAND--78-0227C, CONF-780622, (1978).
10. Feldman, M.H. and L. Silverman, "Chemical Stability of Be_2C Under Cyclotron Irradiation", NAA-SR-114, (1951).
11. Kohn, W. and L.J. Sham, "Self-Consistent Equations Including Exchange and Correlation Effects", *Physical Review*, 140, (1965) A1133-A1138.
12. Perdew, J.P., K. Burke, and M. Ernzerhof, "Generalized Gradient Approximation Made Simple", *Physical Review Letters*, 77, (1996) 3865-3868.
13. Blöchl, P.E., "Projector augmented-wave method", *Physical Review B*, 50, (1994) 17953-17979.
14. Vienna Ab initio Simulation Package, http://cms.mpi.univie.ac.at/vasp/.
15. "Materials Project", Available fom: https://next-gen.materialsproject.org/materials/mp-1569.
16. Gruszka, K., "First Principle Study of Structural and Electronic Properties of CBe2 Compound", *Acta Physica Polonica, A*, 139, (2021) 617-620.
17. Björkas, C., N. Juslin, H. Timko, K. Vörtler, K. Nordlund, K. Henriksson, and P. Erhart, "Interatomic potentials for the Be–C–H system", *Journal of Physics: Condensed Matter*, 21, (2009) 445002.
18. Keiser, J.R., P.M. Singh, M.J. Lance, H.M. Meyer, K.G. Myhre, T.M. Lowe, D. Sulejmanovic, E. Cakmak, V.A. Cox, C.S. Hawkins, and A.W. Willoughby, "Interaction of beryllium with 316H stainless steel in molten Li2BeF4 (FLiBe)", *Journal of Nuclear Materials*, 565, (2022) 153698.
19. Jade 2010, Materials Data Inc., Livermore, CA 94550, https://www.materialsdata.com/prodjd.html.
20. PDF-4+, 2021, International Centre for Diffraction Data (ICDD), https://www.icdd.com/pdf-4/.
21. McFarlane, J., J.W. McMurray, and A. McAlister, "Installation and Commissioning of the Netzsch Skimmer", ORNL/TM-2019/1263, (2019).

APPENDIX A. BERYLLIUM CARBIDE CUTTING PLAN

APPENDIX A. BERYLLIUM CARBIDE CUTTING PLAN

OAK RIDGE National Laboratory

Be_2C Cutting Plan for Materion

Anne Campbell

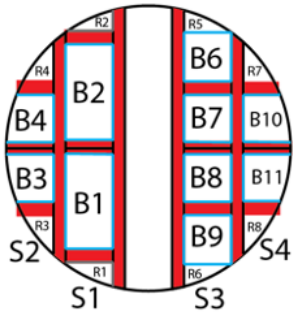
January 13, 2023

ORNL is managed by UT-Battelle, LLC for the US Department of Energy

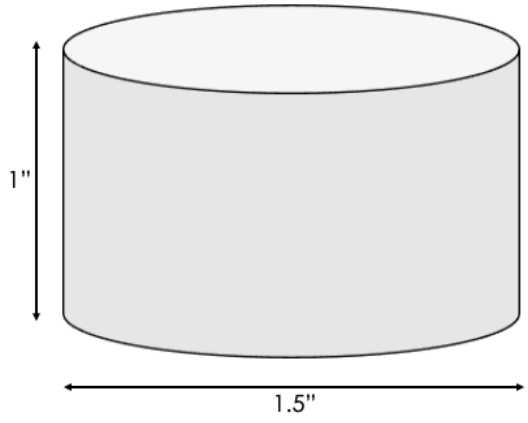
U.S. DEPARTMENT OF ENERGY

As-Produced Be_2C (1.5" diameter, 1" tall)

Cutting allowance of 0.0625" accounted for throughout



Top View sectioning plan
To scale
Red area is cutting allowance

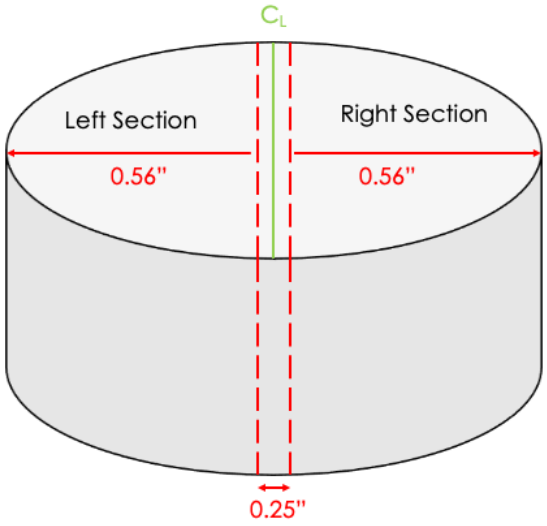


2

OAK RIDGE National Laboratory

First Cuts

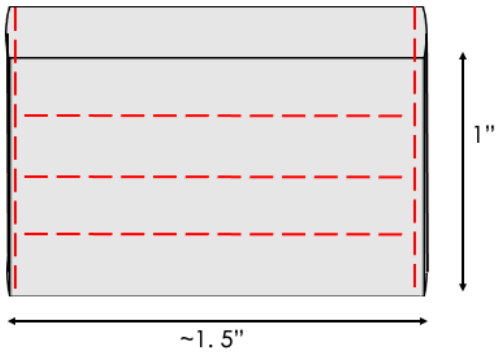
1. Find centerline of the puck
2. Make 2 cuts 0.125" on each side of centerline to have central slab that is 0.25" wide



Drawings NOT to scale

Center Section Cutting (rods)

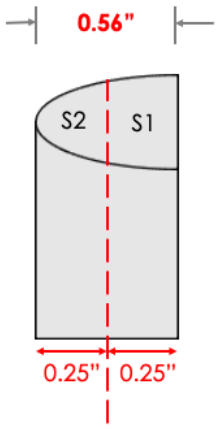
1. Cut into 4 bars, each roughly 0.2" tall
2. Cut ends of each bar to be flat and parallel with each other



Drawings NOT to scale

Left Section Cutting

Section left side into 2 ~0.25" slabs (labeled S1 and S2). Leave remaining piece whole

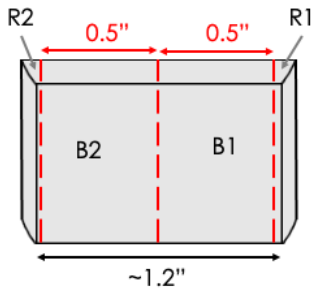


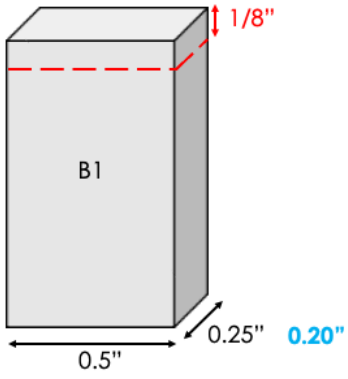
5 OAK RIDGE National Laboratory

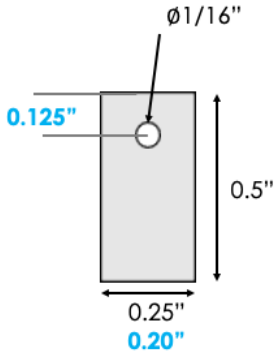
Drawings NOT to scale

Sectioning S1 (capsule coupons)

1. Cut in half
2. Slice off round ends to make ~0.5" wide bars as shown
3. Slice B1 and B2 into coupons 0.5"x0.25" by 1/8" thick
4. In each coupon, drill a 1/16" diameter hole in location shown





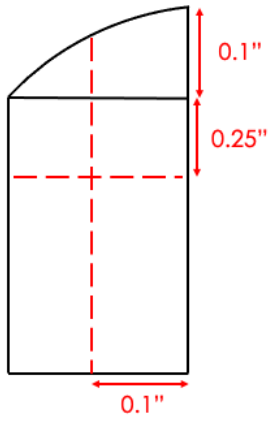


6 OAK RIDGE National Laboratory

Drawings NOT to scale

Sectioning R1 and R2

1. Slice into 0.25" sections (probably 3 pieces each)
2. On each section slice off pointed end to have pieces semi-square pieces that are 0.1x0.1x0.25"

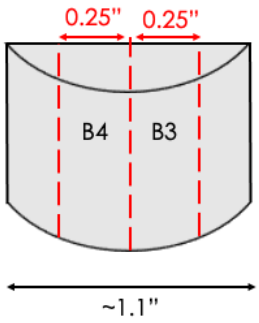


OAK RIDGE National Laboratory

Drawings NOT to scale

Sectioning S2 (rods)

1. Cut piece down the middle
2. Cut two ~0.25" wide bars as shown

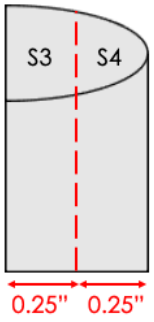


OAK RIDGE National Laboratory

Drawings NOT to scale

Right Section Cutting

Section right side into 2 ~0.25" slabs (labeled S3 and S4). Leave remaining piece whole

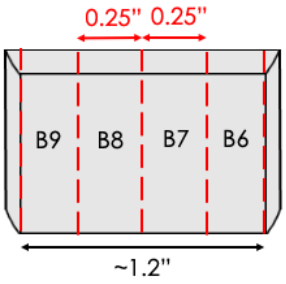


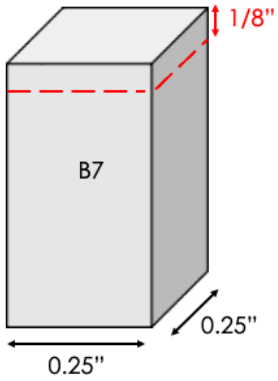
OAK RIDGE National Laboratory

Drawings NOT to scale

Sectioning S3 (Irradiation Pieces)

1. Slice one end to make flat
2. Cut 0.25" wide bars as shown (B7 and B8)
3. Cut curved ends flat to make B6 and B9 (will be smaller than 0.25" wide)
4. Slice B7 – B8 into coupons 0.25"x0.25" by 1/8" thick



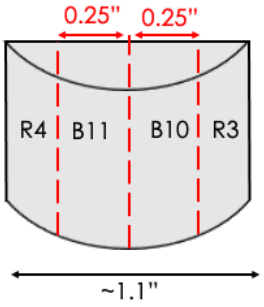


OAK RIDGE National Laboratory

Drawings NOT to scale

Sectioning S4 (rods)

1. Cut piece down the middle
2. Cut two ~0.25" wide bars as shown



11

OAK RIDGE National Laboratory

Drawings NOT to scale

APPENDIX B. UofM SOP for BERYLLIUM CARBIDE WORK

APPENDIX B. UOFM SOP FOR BERYLLIUM CARBIDE WORK

ENVIRONMENT, HEALTH & SAFETY
UNIVERSITY OF MICHIGAN

Beryllium Carbide Irradiation

Standard Operating Procedure

Revision Date: 03/17/2023

Laboratory Director (LD) Approval is Required Prior to Performing this Procedure

Description [Provide additional information as it pertains to your research protocol]

Beryllium Carbide (Be_2C) slabs will be irradiated one at a time and put under microscopy to view their tolerance to radiation damage. Testing in an ion accelerator, Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM) will be necessary to carry out the experiment and properly gather data.

One Be_2C sample consists of dimensions 0.25 x 0.25 x 0.125 in^3 with a mass of 0.25 grams. Slabs samples of Be_2C that have been cut, grinded, and polished are considered "finished articles." Be_2C samples broken as a result of unexpected breakage during testing or other laboratory operations are NOT considered "finished articles" and precautionary measures **must** be followed in order to clean-up and dispose of broken Be_2C sample fragments. This SOP will cover pre-cut, unpolished solid samples and **storage** or powdered Be_2C. **No testing of Be_2C powder will be covered within this SOP and will only include how to properly store the Be_2C powder.**

A broken Be_2C sample may cause potential personal exposure to beryllium in the form of dust or chips, damage to equipment and considerable downtime for the lab. Therefore, it is important to prevent fracture or release of the Be_2C samples by following the steps in this document.

Process

1. To prepare the Be_2C sample for testing, it must be cut, sanded, polished and coated inside a glovebox located in NEL 1066. A wet oil saw will be used for cutting samples into the desired dimensions. Because oil is used, Be_2C particles will not be thrown up in the air. A grind wheel with grit ranging from 200-1200 will be used for sanding the samples. This will produce Be_2C contaminated sandpaper that will be disposed of in a dedicated Be_2C waste container located inside the glovebox. A polishing wheel with a 3-6 µm paper will be used for polishing the sample. This will produce Be_2C contaminated polishing paper that will be disposed of in a dedicated Be_2C waste container located inside the glovebox. Finally a gold coating will be used to cover the Be_2C sample with a thickness of 50 nm. This gold coating will be applied on both sides of the slab encapsulating all the material. Following this, the sample will be placed inside a molybdenum box designed for testing within the ion beam accelerator. This box will have a 4mm diameter hole exposing the gold coated sample and will have a piece of tape covering the

hole. This will be considered the "prepped sample". Proper PPE including gloves, respirator, goggles, and lab coat must be worn for this entire process.

2. The prepped sample will be placed inside a plastic bag followed by a padded box to transport it by foot to the Michigan Ion Beam Lab (MIBL) located at the Naval Architecture And Marine Engineering building Rm 120. The packing material in the box should prevent the sample from being jostled during transport.
3. Once at MIBL, the prepped sample will be removed from the padded box and bag. Proper PPE must be worn **before** any loading can be done at MIBL. PPE includes gloves, goggles, lab coat, and respirator. Once properly protected, the prepped sample will be loaded onto the high temperature stage and placed in the Multi Beam Chamber (MBC) or Beam Line 2 (BL2). The piece of tape will be removed right before the stage has been loaded to the chamber. PPE may not be removed until the stage has been loaded into the chamber.
4. The sample will be heated and irradiated within the beamline. Respirator, gloves, and lab coat are not required while monitoring the irradiation from the control room. However, if for any reason the sample must be removed from the beamline then proper PPE is necessary including respirator, gloves, lab coat, and goggles.
5. Following testing, the stage will be unloaded from the MBC and a new piece of tape will be applied to the exposed hole immediately after removing the stage. The prepped sample will be placed within a plastic bag and padded box to be transported by foot to NEL 1066 glovebox.
6. The prepped sample will remain inside a plastic bag followed by a padded box to transport it by foot to the Michigan Center for Materials Characterization Lab (MC^2) in NCRC Building 22 Rm G010.
7. The prepped sample will be removed from the padded box and plastic bag along with being removed from the molybdenum box. The Be_2C coated slab will be placed inside a SEM and TEM for microscopy.
8. Once finished with microscopy, the sample will be placed within the molybdenum box, plastic bag, and padded box to be transported by foot back to the NEL 1066 glove box. A smear test for Be_2C will be done on the padded transportation box while unloading the sample.
9. The samples will remain stored inside the glovebox for the remainder of time.

Potential Hazards [Provide additional information as it pertains to your research protocol]

Inhalation of beryllium dust and/or particles can cause chronic beryllium disease (CBD) or beryllium sensitization in exposed individuals. CBD is a chronic and sometimes fatal lung condition. Beryllium sensitization is a condition in which a person's immune system may become highly responsive or allergic to the presence of any beryllium within the body. In the case of beryllium sensitization, the concentration of beryllium may be different for each individual person.

Other routes of entry that may contribute to these conditions also include ingestion and skin contact or absorption. Furthermore, both beryllium and beryllium compounds are considered to be class 1 carcinogens.

Occupational Exposure Limits (OELs):

The OSHA beryllium final rule established an action level of 0.1 $\mu g/m^3$ and reduced the permissible exposure limit (PEL) for beryllium to 0.2 $\mu g/m^3$. Both values are 8-hour time weighted average concentrations. The standard also established a short term exposure limit (STEL) of 2.0 $\mu g/m^3$ as determined over a sampling period of 15 minutes. Should any of these limits be exceeded, all beryllium handling will be stopped while re-evaluations and changes are made to reduce exposures.

OSHA did not set a numerical surface contamination limit, but requires beryllium users to establish procedures to maintain all surfaces in beryllium work areas as free as practicable of beryllium. The Department of Energy (DOE) housekeeping level of 3$\mu g/100\ cm^2$ and release criteria of 0.2 $\mu g/100\ cm^2$ as numerical criteria to ensure that surface levels are being maintained as free as practicable of beryllium.

Engineering Controls

This document covers only the handling for Be_2C (Beryllium Carbide), which will only be handled in the solid form. Each experiment will only handle one sample at a time with each weighing 0.25 grams. The dimensions of the sample before coating are 0.25 x 0.25 x 0.125 in^3.

1. **Sample prep.**
   When preparing the sample, all cutting, sanding, and polishing will be done within a glove box at negative pressure. The samples will be coated with a layer of gold within the glovebox to encapsulate the Be_2C.
2. **Irradiation.**
   When loading the sample to the beamline, it will be coated with a layer of gold to encapsulate the Be_2C reducing any sputtering from occurring. A molybdenum box will be used to enclose the coated sample only leaving a 12.5 mm^2 area exposed. Proper PPE must be worn during loading and unloading. A smear test for Be_2C will be performed on the padded transportation box when unloading and loading the sample.
3. **Microscopy.**
   The sample will be coated with a layer of gold and placed inside a SEM that is under vacuum. Proper PPE must be worn during loading and unloading.
4. **Transportation.**
   The Be_2C will be coated with a layer of gold and placed inside a molybdenum box. The prepped sample will then be placed inside a plastic bag. All of this will be put in a padded box that is properly labeled for transportation.

Work Practice Controls

1. **Sample preparation:**
   Sample preparation must always be conducted within the glovebox. If a sample is not in use then it must be properly stored in a desiccator that is labeled. Be_2C samples should never be left out in the glovebox unattended. While work is being done in the glovebox, all other persons in the lab must be wearing proper PPE including gloves, lab coat, goggles, and respirator. No one is allowed to enter or leave the lab while cutting, sanding, or polishing is underway. The interior of the glovebox must be cleaned after every preparation session. This includes whipping down all machines, packaging all samples (in desiccator storage or transportation containment) and throwing away any Be_2C contaminated waste in designated Be_2C waste containers.
2. **Irradiation:**
   MIBL staff must be notified when a Be_2C sample will be in or is in the lab. The sample must not be removed from its transportation containment until all preparations have been done first. Preparations include gathering the high temperature stage, wrenches, bolts, and pliers needed to assemble the stage. The beamline chamber must also be adjusted to the proper position before loading of the stage is done. Proper PPE must be worn when loading and unloading the sample to the beamline. When loading /unloading, staff must be aware and stand a proper distance away if not wearing PPE.
3. **Microscopy:**
   MC^2 Staff must be notified when a Be_2C sample will be in or is in the lab. The sample must not be removed from its transportation containment until all preparations have been done first. Proper PPE must be worn when loading and unloading the sample to the beamline. When loading /unloading, staff must be aware and stand a proper distance away if not wearing PPE.
4. **Transportation:**
   The padded transportation box must be inside a plastic bag to cover it in the glovebox when sample cutting, sanding, or polishing is underway. A large Kim wipe is to be placed under the padded box so that it is never in contact with the surface of the glovebox. When loading the prepared sample to its transportation containment, verify that the plastic bag and padded box has been closed properly. Visually inspect that no contaminants can be seen on the exterior of the box after packaging. When removing the transportation containment from the glovebox, inform all other persons currently in the lab. Public or personal transportation is **not** permitted when transferring the sample from locations and must be walked by foot.

Personal Protective Equipment **[Provide additional information as it pertains to your research protocol]**

All sample prep will be conducted inside the glovebox. Even when conducting sample prep using the glovebox, proper PPE is required in the event that a tear in the glovebox occurs. When loading and unloading the sample to the beamline or SEM, PPE must be worn as well. Proper PPE include.

- Goggles
- Disposable Lab Coat
- Gloves
- Respirator

Respirator use requires employee participation in the Respiratory Protection Program, which involves medical clearance and annual fit testing and training

Once the sample has been loaded to the beamline, safety glasses are only necessary for monitoring the experiment **from the control room**. So long as you are working in the prep station, full PPE will still be required. PPE is not required when the sample has already been inserted into the SEM. Those who have handled sample transfer are to wash their hands after taking off their PPE to minimize risk of particles that could have been unknowingly transferred.

Contact EHS for assistance in performing an exposure assessment.

Transportation and Storage

All Be_2C samples will be inside the glovebox and stored inside a desiccator to be kept dry. **Be_2C in the form of powder will be placed inside 2 layers of plastic bags and stored inside the desiccator.** The container will be labeled with the material inside and its hazard warnings.

Whenever transportation is necessary the Be_2C slab will be packaged with the following contaminants in this specific order.

- Place sample inside molybdenum containment box (with tape covering exposed area). The containment must be cleaned with Kim wipes before the sample is placed inside.
- Package inside a plastic bag that can be properly sealed. The bag must be clean before loading. Disposable bags are recommended and must be properly disposed of in a beryllium dry waste container.
- Insert into a padded box that can be properly sealed. The padded box must be cleaned before the sample is placed inside.

All packaging will be done within the glovebox and inspection of the final transportation box must be conducted before it is removed. A large kim wipe will be placed under the padded box so that contact with the surface of the glovebox never occurs.

Waste Disposal

Solvent contaminated Be_2C wipes, and debris are often generated during sample prep cleaning and maintenance activities conducted in research laboratories. Wipes/towels that have been in contact with Be_2C are considered hazardous waste and must be managed appropriately. Waste will be sealed in two layers of containment such as an impermeable bag or container. The outer surface of the barriers must be free of beryllium contamination. This will be done by having a plastic bag over it and removing it when it is removed from the glovebox. If the Large antechamber is used, a smear test will be performed to view levels of contamination.

All samples that have been tested will not be disposed of. Properly labeled containers will be placed inside the glove box for long term Be_2C sample storage.

The hazardous waste label must be on every container of Be_2C waste. It helps researchers properly manage their waste and stay in compliance with federal, state, and local rules and regulations. All waste generators must complete a manifest that accurately describes the waste Hazardous Materials Management (HMM) will be collecting.

The following process describes what laboratory personnel must do to prepare Be_2C waste for collection:

- Complete the WASTE MANIFEST
- Store chemical waste in a safe, accessible, and easily identifiable closed jar located inside a glovebox. The waste jar must be always visible for EHS personnel to inspect for completeness and transport.
- Contact HMM at (734) 763-4568 or complete the online Waste and Supply Request form for collection.

Because most spent, unused, and expired chemicals/materials are considered hazardous wastes, they must be properly disposed of. **Do not dispose of chemical wastes by dumping them down a sink, flushing in a toilet or discarding in regular trash containers, unless authorized by Environment, Health & Safety Hazardous Materials Management (EHS-HMM).** Contact EHS-HMM at (734) 763-4568 for waste containers, labels, manifests, waste collection and for any questions regarding proper waste disposal. Also, refer to the EHS Hazardous Waste Web page for more information.

Exposures/Unintended Contact

In the event that unintended contact occurs with Be_2C, the following steps should be taken immediately depending on the form of contact.

Inhalation:
Remove victim to fresh air and keep at rest in a position comfortable for breathing. Oxygen or artificial respiration if needed. Do not use mouth-to-mouth method if victim inhaled the substance. Induce artificial respiration with the aid of a pocket mask equipped with a one-way valve or other proper respiratory medical device. Call a physician or poison control center immediately.

Skin Contact:
Remove contaminated clothing immediately and wash skin with soap and water. In case of eczema or other skin disorders: Seek medical attention. Wash contaminated clothing before reuse.
Eye Contact:
Do not rub eyes. Immediately flush eyes with plenty of water for at least 15 minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Get medical attention if irritation develops and persists.

Ingestion:
Call a physician or poison control center immediately. Rinse mouth. Do not induce vomiting without advice from poison control center. If vomiting occurs, keep head low so that stomach content doesn't get into the lungs. Do not use mouth-to-mouth method if victim ingested the substance. Induce artificial respiration with the aid of a pocket mask equipped with a one-way valve or other proper respiratory medical device.

⚠ ***If the employee is in need of emergency medical attention, call 911 immediately.*** ⚠

injury type	action	notes
Exposure-Eyes	1. Flush with water for at least 15 minutes 2. Seek medical attention.	Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.
Exposure-Skin	1. Remove contaminated clothing and footwear. 2. Flush with water for at least 15 minutes. 3. Remove contaminated clothing. 4. Seek medical attention.	Indicate if contaminated clothing can be laundered for reuse or must be discarded as hazardous waste.
Inhalation (including from spills outside the fume hood)	1. Remove patient from the contaminated area. 2. Encourage patient to blow nose to ensure clear breathing passages. 3. Ask patient to rinse mouth with water but to not drink the water. 4. Seek medical attention.	
Ingestion	1. If swallowed, refer for medical attention, where possible, immediately.	Urgent hospital treatment is likely to be needed.
NOTE: If an ambulance is needed, call the University of Michigan Division of Public Safety and Security (DPSS) at 911 to request assistance.		

Report all work related accidents, injuries, illnesses or exposures to Work Connections within 24 hours by completing and submitting the Illness and Injury Report Form.

Follow the directions on the Work Connections website Where to go for treatment to obtain proper medical treatment and follow-up.

Complete the Incident and Near-Miss Report form.
https://ehsa.oseh.umich.edu/EHSA/public/injuryillnesssubmit/injuryillnessinitialedit

Treatment Facilities

U-M Occupational Health Services -- Campus Employees
Mon-Fri 7:00 am - 4:30 pm

C380 Med Inn building
1500 East Medical Center Drive, Ann Arbor (734) 764-8021

University Health Services -- University students (non-life threatening conditions)
Mon-Fri 8 am - 4:30 pm, Sat 9 am - 12 pm
Contact for current hours, as they may vary
207 Fletcher Street, Ann Arbor (734) 764 - 8320

UMHS Emergency Department -- after clinic hours or on weekends
1500 East Medical Center Drive, Ann Arbor (734) 936-6666

Spill Procedure

If a Be_2C samples breaks while under irradiation, transportation or microscopy, follow these steps:

- Immediately shut down the vacuum pump in the beam line or SEM and keep samples inside the equipment.
- Leave the lab and call U-M DPSS at 911 with a contact name and phone number for someone with knowledge about the lab to report potential beryllium contamination in the lab.
- Post a sign at the entrance of the room "Danger: Suspect Beryllium Contamination Do Not Enter."

U-M DPSS will contact EHS to advise them of the problem. **Do not do anything else until EHS has evaluated the situation and advised that you may proceed.**

EHS may collect wipe samples of the lab and surrounding areas and send them for analysis to determine the potential for contamination outside the chamber. *It will take a day or two to receive analytical results of the wipe samples. The lab will remain closed during this time until EHS grants approval to re-enter the lab to clean up the contamination.*

While waiting for the analytical results of the wipe sample, contact EHS-HMM at 763-4568 to obtain the following supplies for cleanup:

- Hazardous waste containers and waste labels
- Tyvek suits and shoe covers
- A dedicated HEPA filter vacuum

A **chemical spill of any size** requires active assistance from emergency personnel. Report spills in corridors or common areas, e.g., hallways, elevators, eating areas, rest rooms, offices, etc., to University of Michigan Division of Public Safety and Security (DPSS) at 911.

Do not attempt to clean up a major chemical spill or one that occurs outside a fume hood. In the event of a major chemical spill, use the following information for a safe spill response.

- Attend to injured or contaminated persons and remove them from exposure.
- Alert people in the laboratory to evacuate.

- If spilled material is flammable, turn off ignition and heat sources. Don't light Bunsen burners or turn on other switches.
- **Call University of Michigan Division of Public Safety and Security (DPSS) at 911 immediately for assistance.**
- Close doors to affected area.
- Post warnings to keep people from entering the area.
- Have person available that has knowledge of incident and laboratory to assist emergency personnel.

For additional information regarding spill response procedures, refer to the EHS Hazardous Waste Spill Response Web page.

Donning PPE for Clean Up

Place a tacky mat just inside the door of the lab. Position a waste container near the tacky mat to be used for contaminated PPE when finished with the cleanup, prior to exiting the lab. A clean area to place goggles when doffing PPE should be designated within reach of the tacky mat.

Don the following required PPE prior to entering the lab for cleanup:

- Goggles
- Disposable lab coat
- Disposable gloves
- Respirator

Respirator use requires employee participation in the Respiratory Protection Program, which involves medical clearance and annual fit testing and training

Cleaning Up the Spill

- A dedicated HEPA filter vacuum may be used to suck out small pieces and to vacuum out loose dust, but microscopic dust contamination may still pose a hazard.
  NOTE: The vacuum will be considered contaminated and will need to be cleaned by EHS afterward. **Do not remove** the vacuum from the immediate area of the cleanup.
- Use water or alcohol-wet wipes to wipe down all accessible surfaces. Place the used wipes in plastic bags and dispose of them in the hazardous waste container to be removed by EHS-HMM.
- **Do not blow air onto any contaminated surfaces**.

Removing PPE after Clean Up

Remove PPE **prior** to leaving the lab. It **must** be taken off in the following order:

1. Step on Taki-mat to remove any suspected dust particles
2. Remove the **outer pair** of gloves and place them in the waste container.
3. Remove the lab coat by rolling it down, so the inner surface is on the outside as you roll. Place the in the waste container.
4. Remove goggles and place in pre-designated "clean" area.
5. Remove respirator and place in container.

6. Remove the **inner pair** of gloves and place in the waste container.

A donning and doffing station will be at the inside entrance of NEL 1066 including tacky mats on the inside and outside of the door. A beryllium waste container will be placed near the donning/doffing station in order to throw away any suspected dry Be_2C contaminated waste.

Follow Up

After the surfaces of the chamber have been cleaned, contact EHS to complete a series of wipe tests to determine the highest accessible level of dust that remains. The test will focus on the transportation box as it is the highest risk of contamination.

Emergency Reporting

Report all emergencies, suspicious activity, injuries, spills, and fires to the University of Michigan Police (DPSS) by calling 911 or texting 377911. Register with the University of Michigan Emergency Alert System via Wolverine Access.

Training of Personnel

All personnel shall read and fully adhere to this SOP when handling this chemical.

Certification

I have read and understand the above SOP. I agree to contact my Lab Director if I plan to modify this procedure.

Name	Signature	UMID #	Date
Diego Muzquiz		80282431	03/17/2023

Lab Director Revision Date

Major Revisions (Tracking purposes only -- Do not print as part of SOP)

Date	Revision