ORNL/SPR-2018/1046

# FY18Q4 Quarterly Report: Radiation Enhanced Diffusion of Ag, Ag-Pd, Eu, and Sr in Neutron Irradiated PyC/SiC Diffusion Couples

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**October 2018**

OAK RIDGE NATIONAL LABORATORY
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Fusion and Materials for Nuclear Systems Division

# FY18Q4 QUARTERLY REPORT: RADIATION ENHANCED DIFFUSION OF AG, AG-PD, EU, AND SR IN NEUTRON IRRADIATED PYC/SIC DIFFUSION COUPLES

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Date Published: October 2018

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, TN 37831-6283
managed by
UT-BATTELLE, LLC
for the
US DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

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# ACRONYMS

| | |
|---|---|
| AGR | Advanced Gas Reactor (Fuel Development and Qualification Program) |
| dpa | Displacements per atom |
| GD-OES | Glow-discharge optical emission spectroscopy |
| HFIR | High Flux Isotope Reactor |
| IMET | Irradiated Materials Examination and Testing Facility |
| MS | Methylsilane |
| MTS | Methyltrichlorosilane |
| ORNL | Oak Ridge National Laboratory |
| PyC | Pyrolytic carbon or pyrocarbon |
| RBS | Rutherford backscattering spectroscopy |
| S-PyC | Support pyrocarbon |
| SiC | Silicon carbide (TRISO layer) |
| SIMS | Secondary ion mass spectroscopy |
| TRISO | Tristructural-isotropic (coated particles) |

# ACKNOWLEDGMENTS

This research is being performed using funding received from the DOE Office of Nuclear Energy's Nuclear Energy University Program through a joint NEET/NEUP R&D with NSUF access award (Project 16-10764). Significant support for this work was provided by Victoria Cox, J. Travis Dixon, and Tom Geer.

## ABSTRACT

An improved understanding of the diffusion of select fission product systems in materials representative of tristructural-isotopic (TRISO) coated particle fuel are being sought to improve the safety and efficiency of high temperature gas-cooled reactors. Novel diffusion couple samples have been produced to explore diffusion under neutron irradiation and at temperatures representative of safety testing conditions. An update on the status of the neutron irradiation study and high temperature thermal testing is discussed which build upon prior reporting [1–4].

## 1. INTRODUCTION

The exploration of diffusion of select fission product species in materials representative of TRISO coated particle fuel is necessary to develop an understanding of the factors impacting release during normal and off-normal operation. Prior efforts have focused on the development of a novel diffusion couple design to explore the diffusion of silver, silver and palladium, europium, and strontium (Ag, Ag+Pd, Eu, and Sr) in TRISO-SiC materials [1–4]. The design focuses on a planer sample geometry of sequentially layered pyrocarbon (PyC), silicon carbide (SiC), and support-PyC (S-PyC) structure. The diffusing species is introduced via ion implantation into the PyC layer and the system is sealed using methylsilane (MS) and methyltrichlorosilane (MTS) derived SiC. This design mimics the release pathway expected for fission product release from intact TRISO fuel as the diffusing species moves through the PyC layer before encountering the SiC layer. The development process is summarized in ORNL/TM-2018/1012 [5].

Diffusion couples with deviations from typical TRISO layer properties, as defined by the TRISO fuel produced for the Advanced Gas Reactor Fuel Qualification and Development Program (AGR), are also being explored to understand the impact of the specific layer attributes on diffusion in TRISO materials. The three variants being explored are Baseline, SiC Variant, and PyC Variant. The Baseline sample's microstructure represents typical AGR TRISO. The SiC Variant possess a relativity finer grainsize compared to Baseline to explore the role of SiC microstructure on diffusion. The PyC Variant possesses a higher density PyC layer relative to the Baseline to explore the impact of density and PyC/SiC interface on fission product interaction [1–4]. Additionally, commercial, direct ion implanted samples in polycrystalline SiC and single crystal 4H-SiC are also being explore to compare to prior experiments and determine the potential for lattice diffusion in SiC [1–4].

The planned test matrix is shown in Table 1. The experiment consists of two primary thrusts; neutron irradiation and high-temperature thermal diffusion. The neutron irradiation study consists of two irradiation capsules irradiated to SiC doses of 0.5 and 1.0 displacements per atom (dpa) at ~1100 ºC. A thermal equivalent sample will be produced which explores the same time at temperature as the neutron irradiated samples. This experiment will provide insight into the role of neutron irradiation on the diffusion of select fission product systems in the representative TRISO materials. The second thrust is the exploration of high-temperature thermal diffusion. This experiment explore diffusion at temperatures at accident and margin temperatures. These conditions have also been typically explored via safety-testing of irradiated TRISO [6].

Efforts this quarter focused yielded completion of the neutron irradiation efforts in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). The exploration of high-temperature thermal diffusion has also commenced along with supporting analysis and insights on the initial results.

**Table 1. Planned diffusion couple test matrix [3].**

| Condition | Sample Conditions |
|---|---|
| Neutron Irradiation<br>(0.5 dpa, 1100±50 °C) | Baseline: Ag, Ag+Pd, Eu, Sr<br>Commercial-SiC: Ag<br>SiC Variant: Ag, Eu, Sr |
| Neutron Irradiation<br>(1.0 dpa, 1100±50 °C) | |
| Thermal Diffusion<br>(Temperature & time equivalent of 0.5 dpa) | |
| Thermal Diffusion<br>(Temperature & time equivalent of 1.0 capsule) | |
| High-Temperature Thermal Diffusion<br>(1500 °C, 150 & 300 h)* | Baseline: Ag, Ag+Pd, Eu, Sr<br>PyC Variant: Ag, Ag+Pd<br>SiC Variant: Ag, Eu Sr |
| High-Temperature Thermal Diffusion<br>(1600 °C, 150 & 300 h)* | Baseline: Ag, Ag+Pd, Eu, Sr<br>PyC Variant: Ag, Ag+Pd<br>SiC Variant: Ag, Eu Sr |
| High-Temperature Thermal Diffusion<br>(1700 °C, 150 & 300 h)* | Baseline: Ag, Ag+Pd, Eu, Sr<br>PyC Variant: Ag, Ag+Pd<br>SiC Variant: Ag, Eu Sr |

*exposure times may be adjusted based on initial observations.

## 2. STATUS OF HFIR IRRADATION

In the last quarter two hydraulic rabbit capsules were fabricated containing 30 samples each (Table 2) [7]. The two capsules were designated DC01 and DC02 and targeted 0.5 and 1.0 dpa at 1100±50 °C, respectively. In the last quarter full irradiation approval was obtained and the capsules were irradiated in the HFIR at ORNL. The 1.0 dpa capsule, DC02, was the first to be inserted into the reactor. It was inserted during cycle 481 into location B3 in position 6 on July 24th at 16:34. The DC02 irradiation was completed on August 5th at 10:31. The DC02 capsule experienced 263.95 hours in reactor achieving the 1.0 dpa target, however, the irradiation soak was not constant as the capsule was removed to allow for extraction of other hydraulic capsules in the reactor. Figure 1 shows the total time to complete the two capsule irradiations. For DC02 a total of four interruptions occurred. The interruptions were 0.47 h, 1.07 h, 17 h, and 0.4 h respectively. This resulted in multiple temperature cycles during the course of the irradiation. The disruption between the 1.07 h and 17 h disruption is not expected to be significant as the capsule only experienced 0.05 h in reactor before being removed. These disruptions will be taken into account during diffusion analysis of the samples. The 0.5 dpa irradiation for DC01 was achieved in a single continuous irradiation (Figure 1). The capsule was inserted during cycle 481 into location B3 in position 3 on August 5th at 11:02:34 and removed August 10th at 10:56. The total duration was 119.9 h which reached the targeted 0.5 dpa. The actual irradiation temperature for both capsules will be determined through SiC thermometry after deconsolidation of the capsules [8].

**Table 2. Samples for HFIR irradiations [4].**

| *0.5 dpa* | Simulated Fission Product System | | | | |
|---|---|---|---|---|---|
| **Variant** | **Ag** | **Ag+Pd** | **Eu** | **Sr** | **Blank** |
| Baseline | 3 | 2 | 2^ | 2 | 4 |
| SiC | 3 | 0 | 2 | 2 | 0 |
| PyC | 0 | 0 | 0 | 0 | 0 |
| CVD-SiC | 5 | 0 | 0 | 0 | 0 |
| 4H-SiC | 5 | 0 | 0 | 0 | 0 |

| *1.0 dpa* | Simulated Fission Product System | | | | |
|---|---|---|---|---|---|
| **Variant** | **Ag** | **Ag+Pd** | **Eu** | **Sr** | **Blank** |
| Baseline | 3 | 2 | 2 | 2 | 4 |
| SiC | 3 | 0 | 2 | 2 | 0 |
| PyC | 0 | 0 | 0 | 0 | 0 |
| CVD-SiC | 5 | 0 | 0 | 0 | 0 |
| 4H-SiC | 5 | 0 | 0 | 0 | 0 |

^one Eu Baseline sample was compromised and therefore not included in the final build

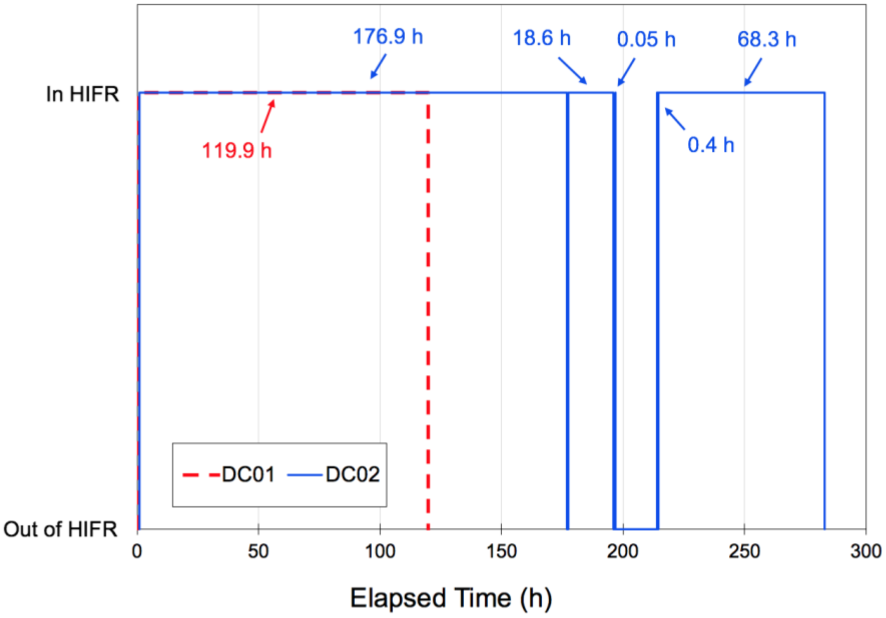

**Figure 1. Total time to complete irradiation of DC01 and DC02**

After irradiation the capsules were allowed to cool. They were shipped to the Irradiated Materials Examination and Testing (IMET) Facility on September 11th. The IMET facility is a hot cell facility which focuses on handling of irradiated structural materials. The capsules will be disassembled and individual samples will be partitioned and transferred to the Low Activation Materials Design and Analysis laboratory at ORNL for sample preparation and analysis. The disassembly is expected to be initiated by November 30th, 2018.

## 3. HIGH-TEMPERATURE THERMAL TESTING OF DIFFUSION COUPLES

Initial scoping tests were conducted to establish the feasibility of the furnace system for high temperature thermal exposures. The furnace system being used is a Thermal Technology Inc. graphite element ASTRO furnace located at ORNL. The furnace operates in vacuum and/or an argon gas atmosphere and has a 3” diameter by 5” working zone. A graphite crucible approximately 2.5” by 4” is used to contain the samples and help provide uniform thermal exposure and prevent against residual oxidation. Thermally conductive graphite foil is placed on the bottom of the graphite holder to assist with heating and further prevent oxidation. Samples were placed into a specially designed graphite diffusion couple holder used in HFIR irradiation tests, shown in Figure 2, to mimic the same containment system as that used for irradiated diffusion couples. These holders were then placed into the graphite crucible, which was placed directly in the furnace. Temperature is tracked on the system using an Yokogawa temperature recorder and by periodically checking the thermocouple during exposure. Successful demonstration of furnace operation was established prompting high-temperature thermal testing of diffusion couples samples.

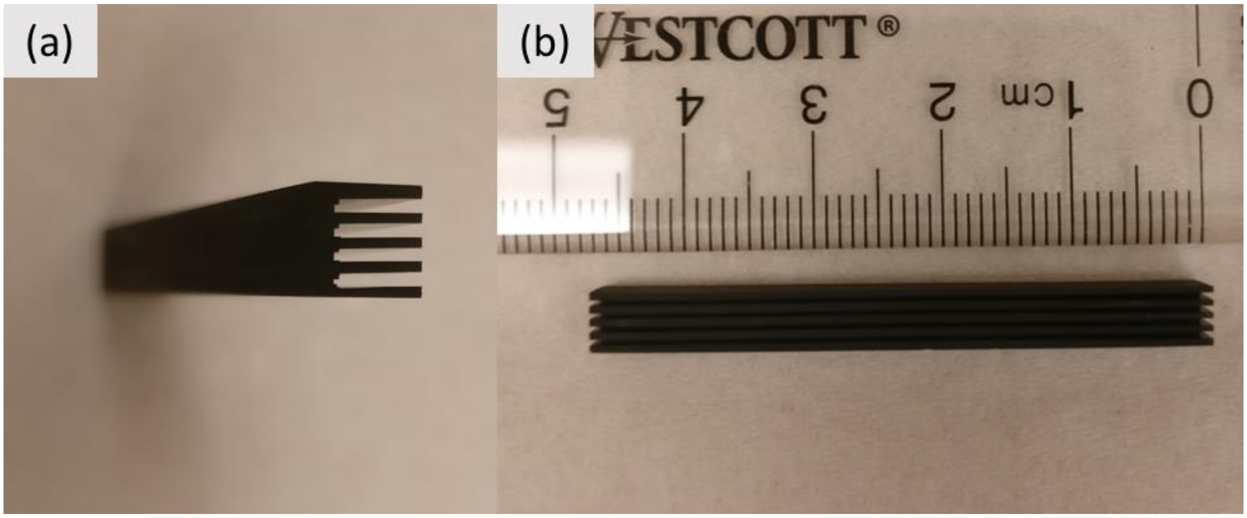

**Figure 2. (a) A side view of the graphite diffusion couple holder. Samples are placed inside one of the middle sections of the holder for even heating. (b) A top view of the holder.**

The first set of diffusion couple samples that were exposed in the furnace were the 1700 ºC, 150 h runs to establish diffusion conditions at the highest temperature. The ramping and cooling rates from safety testing of irradiated TRISO fuel from the Advanced Gas Cooled Reactor Fuel Qualification and Development Program served as a reference when selecting the ramping and cooling rates for the diffusion couple study. Maximum ramp and cooling rates were taken from the safety testing of AGR-1 Compact 4-2-2 which demonstrated rates up to ~20 ºC/min that resulted in no failed particles during a temperature cycle [9], however standard safety-testing heating and cooling rates are considerably slower with typical ramp rates ranging 0.8-1.6 ºC/min [6]. While there are differences in construction and geometry between particles and disk samples, the rates from 4-2-2 were considered appropriate bounds. The selected ramp rate sequence includes an initial rapid ramp to 50 ºC, followed by a ramp to 1250 ºC, and final slower ramp to the final soak temperature (1700 ºC) to avoid thermal shock issues. Table 3 lists the ramp rates and dwell times for each step of the thermal heating test, and a sample heating profile is shown in Figure 3. The ramp rates of 5 ºC/min and 2.5 ºC/min are faster than those present in safety testing, but were selected to ensure the soak time dominated the kinetics of the experiment while staying below the identified 20 ºC/min maximum rate.

**Table 3. Thermal testing ramping and cooling rates.**

| Segment | Setpoint (°C) | Rate (°C/min) | Dwell Time (h) |
|---|---|---|---|
| 1 | 50 | 20 | 0 |
| 2 | 1250 | 5 | 0 |
| 3 | 1500–1700 | 2.5 | 150 |
| 4 | 0 | 20 | 0 |

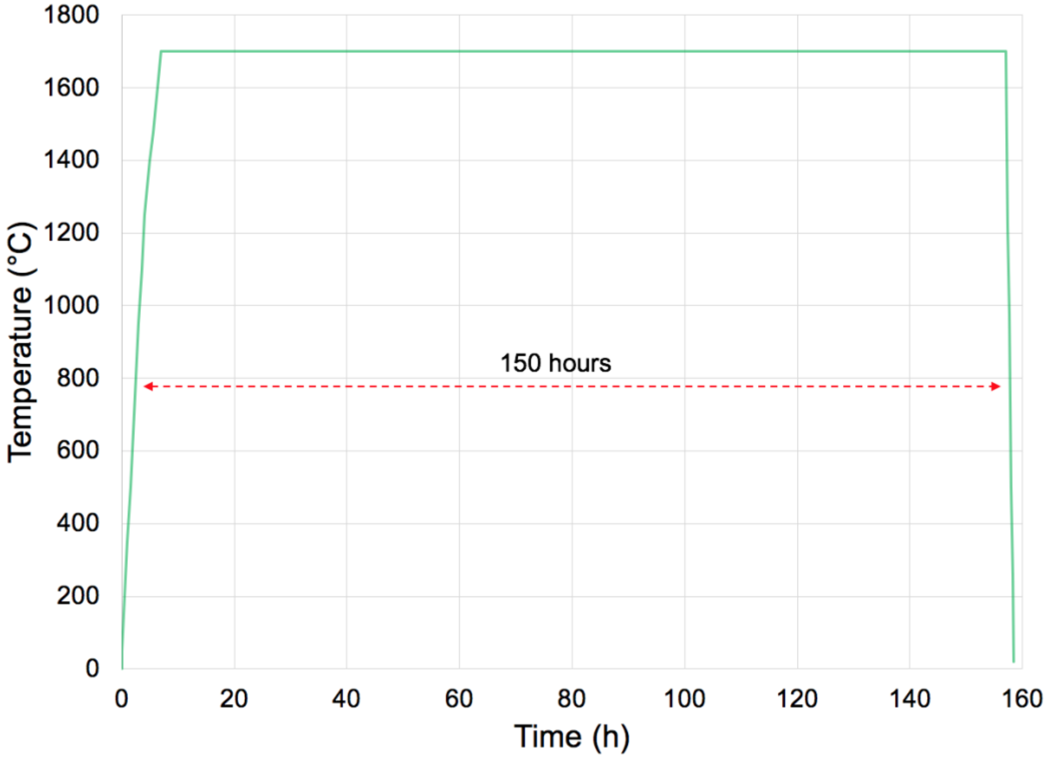

**Figure 3. Example of heating profile for 1700 ºC, 150 h thermal testing.**

Table 4 shows the thermal exposures completed to date. The first set of diffusion couple sample that were exposed in the furnace were the 1700 ºC, 150 h runs. This run was intended to establish diffusion conditions at the highest temperature. The subsequent samples, 1500 ºC, 150 h, were run next to establish a lower bounds for the high-temperature thermal testing. A decision to initially focus on Sr samples was made to help establish diffusion conditions for all samples. This was due to the excess number of samples for this system, to counter a perceived priority given to the Ag and Ag-Pd systems and the finite number of Ag and Ag-Pd samples. For the first run at 1500 ºC an issue with the cooling water system arose leading to premature shut down of the system leading to an exposure time of ~24 h. These samples will still be analyzed by glow discharge optical emission spectroscopy (GD-OES) as they represent a bounding case which may serve to better identify the appropriate exposure conditions.

**Table 4. Complete thermal exposures**

| Sample | Exposure Condition |
|---|---|
| Baseline: Ag | 1700 ºC, 150 h |
| Baseline: Ag+Pd | 1700 ºC, 150 h |
| SiC: Ag | 1700 ºC, 150 h |
| 4H-SiC | 1700 ºC, 150 h |
| Baseline: Sr | 1500 ºC, 24 h* |
| SiC: Sr | 1500 ºC, 24 h* |
| 4H-SiC | 1500 ºC, 24 h* |
| Baseline: Sr | 1500 ºC, 150 h |
| SiC: Sr | 1500 ºC, 150 h |
| 4H-SiC | 1500 ºC, 150 h |

## 3.1 DEPTH PROFILING ANALYSIS

### 3.1.1 GD-OES Sample Preparation

In prior efforts an analysis approach was established to perform GD-OES analysis on the small diffusion couple disk samples [3]. The approach utilized an aluminum mount pre-form and a centering ring to establish vacuum and appropriate location over the anode. Efforts were undertaken to refine the sample preparation method this quarter. Identified criteria for an optimal sample include; overall conductivity, that the sample is flush with the aluminum mount, and the sample holds vacuum. Two approaches were attempted to produce mounts with the sample flush to the surface. The two approaches are defined as “polished” and “flush-mounted”.

Prior to mounting the sample in the aluminum mount, the correct interface must be determined as the PyC/SiC interface is the surface of interest. The Xradia MicroXCT-400, high-resolution x-ray tomography instrument with spatial resolution around 1 µm, is used to determine the correct side of the PyC/SiC/S-PyC structure to be analyzed. This system has been used for prior PIE efforts in the AGR campaign and is described elsewhere [10]. The system uses a microfocus X-ray source operated at 40 keV to produce a narrow-cone beam, which allows for the acquisition of high-resolution imaging of the x-ray scintillation. The PyC side is determined from the generated X-ray radiograph and identified based on differences in thickness of the two PyC layers.

The polished sample preparation approach utilizes the aluminum mount with a central recessed area to position the diffusion couple disk. A schematic for the “polished” sample preparation approach is shown in Figure 4. The sample is secured to the mount in the recessed area with the side of interest exposed using a small amount of super glue to adhere the sample to the mount. The gaps surrounding the sample are filled using a conductive colloidal graphite paint. The colloidal graphite is added sequentially to ensure no gaps occur during drying. The sample is then polished using an Allied Multiprep system to remove the excess colloidal graphite and eventually bring the sample flush with the top of the aluminum mount. The progress is tracked by periodically checking the difference in height between the aluminum mount and the exposed sample surface. Initial material removal is accomplished using 15–30 µm grit diamond film. Final polishing/thinning is accomplished using 1-µm diamond film and polishing is considered complete when the mount and sample are <20 µm apart.

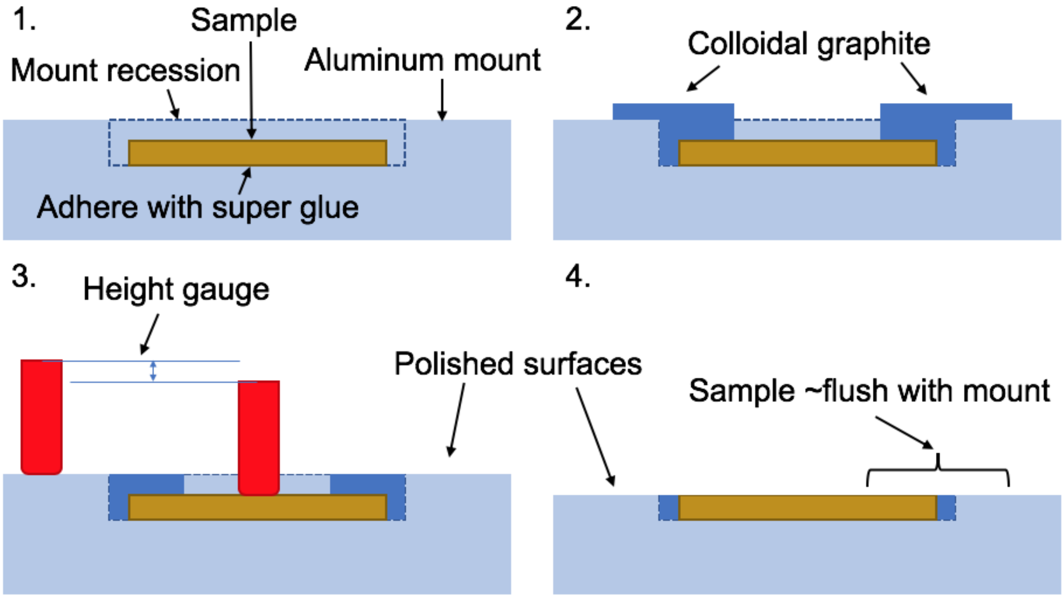

**Figure 4. Schematic of "polished" sample preparation approach.**

The "flush-mounted" approach attempts to minimize sample processing. A schematic of the mounting process is shown in Figure 5. The aluminum mounts vary from the "polished" approach in that a small 1/16th in through hole is drilled through the mount to allow backfilling creating a larger recessed volume. The diffusion couple sample is placed in the recessed area PyC/SiC interface out. The sample and mount are then adhered to double-sided tape mounted on a glass slide and the sample pressed into the tape via the through-hole. This ensures the sample is flush with the top of the aluminum mount. A steel pin is inserted into the through-hole with a small amount of super glue to secure the sample to the mount. After curing the super glue the sample is backfilled with colloidal graphite to provide a conductive sample. The sample and mount are then removed from the glass slide and tape.

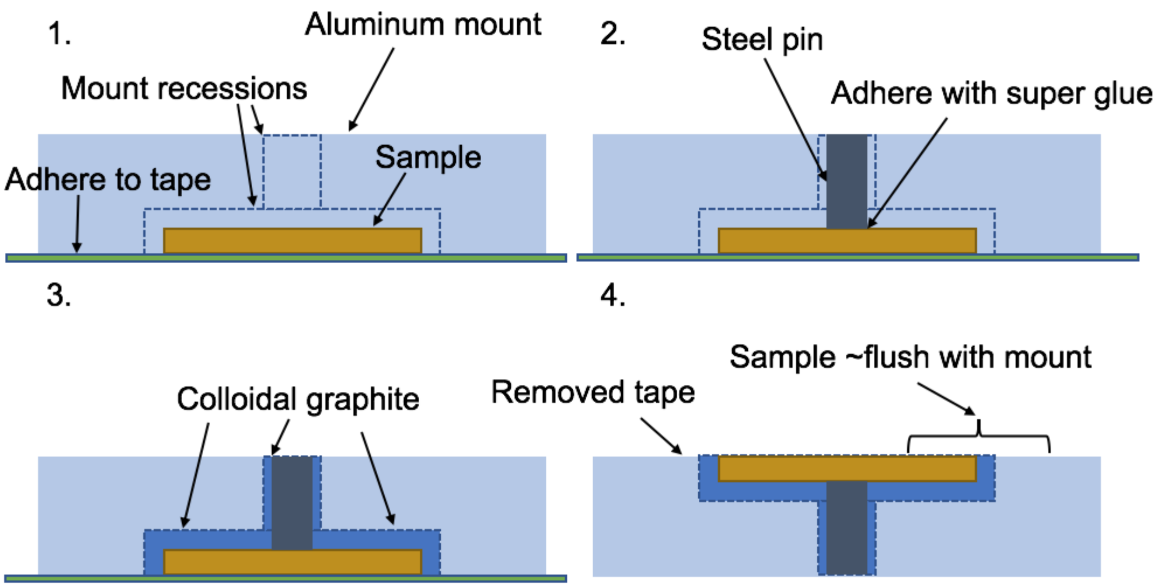

**Figure 5. Schematic of "flush-mounted" sample preparation approach.**

Both approaches led to successful GD-OES data acquisition. However, there were differences in the quality and repeatability of the analysis from the different approaches. The primary concerns were plasma instability and residual oxygen. Excess plasma instability was observed for the "flush-mounted" samples. The instability was most apparent in the beginning of the analysis. The source of the instability was likely residue transferred from the tape used to adhere the sample and/or excess super-glue which reached the sample surface. In select samples from the "flush-mounted" approach measurable oxygen contamination was observed. The excess oxygen was assumed to be due to an imperfect seal between the aluminum mount and O-ring used to seal the sample. Limited instability was observed in the "polished" sample preparation approach where the instability was assumed to be due to edge effects from the sample. Limited oxygen signal was observed as the polishing provides a relatively defect free surface which generates a good seal between the aluminum mount and O-ring. While the "polished" sample preparation approach is inefficient compared to the "flush-mounted" method the improved analysis makes it the preferred approach.

### 3.1.2 Initial GD-OES Analysis

Of the initial exposed samples the Baseline: Ag, SiC: Ag, and 4H-SiC 1700 ºC, 150 h exposures were analyzed by GD-OES. Examples of the acquired data are shown in Figure 6–Figure 8. The initial plasma instability is seen during the first 100 s of data acquisition. For the Baseline: Ag sample, silver is distributed across the PyC layer with no clear observation of direct diffusion into the SiC layer. The absence of clearly observed diffusion into the SiC layer is due to the limited sensitivity of GD-OES below the presumed solubility limit of silver in the PyC. However, the measured distribution shows the design is successful as the implanted silver concentration diffuses across the PyC layer to the SiC layer of interest. No silver was measured in the SiC: Ag sample. Two hypotheses for the lack of measured silver are that a failed seal-coating led to loss of the implanted silver or the diffusivity in the SiC Variant sample was enough to facilitate complete diffusion of silver out of the system. The fine-grained SiC Variant is expected to have a higher effective diffusivity than the Baseline sample based on the difference in grain size [11]. This behavior will be further understood with the examination of additional diffusion couple samples and conditions.

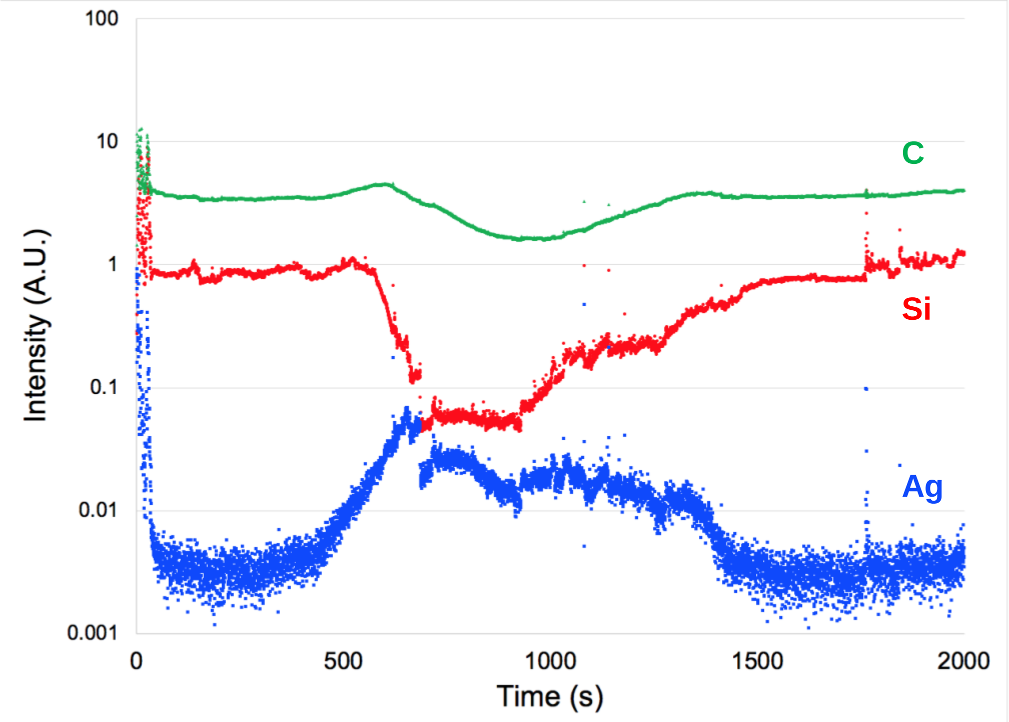

**Figure 6. GD-OES depth profile of 1700 °C, 150 h Baseline: Ag diffusion couple. Time correlates to depth.**

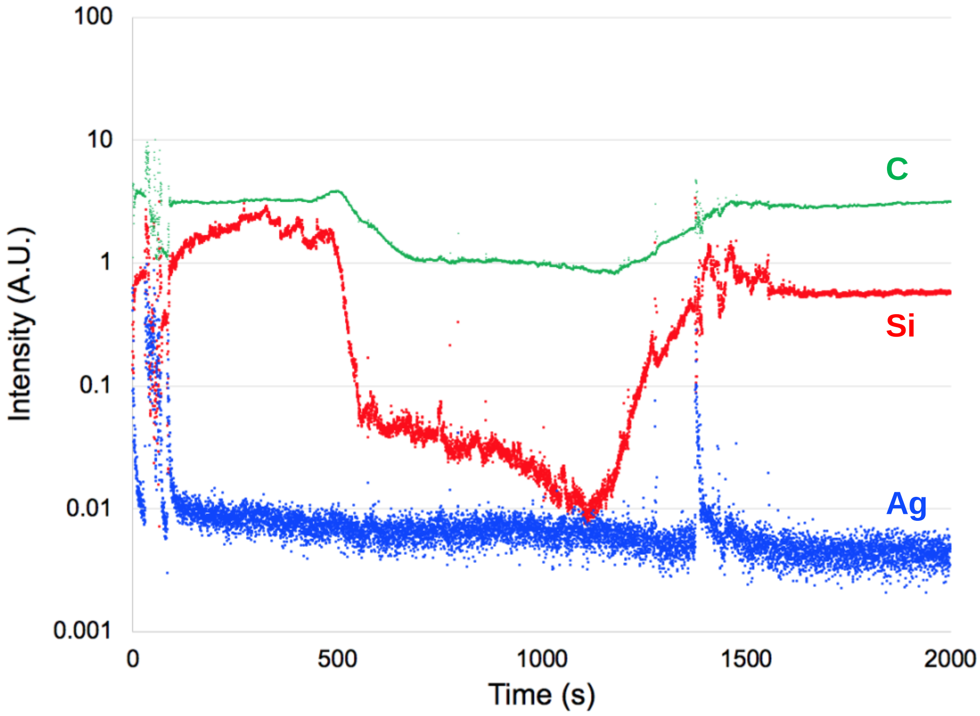

**Figure 7. GD-OES depth profile of 1700 °C, 150 h SiC: Ag diffusion couple. Time correlates to depth.**

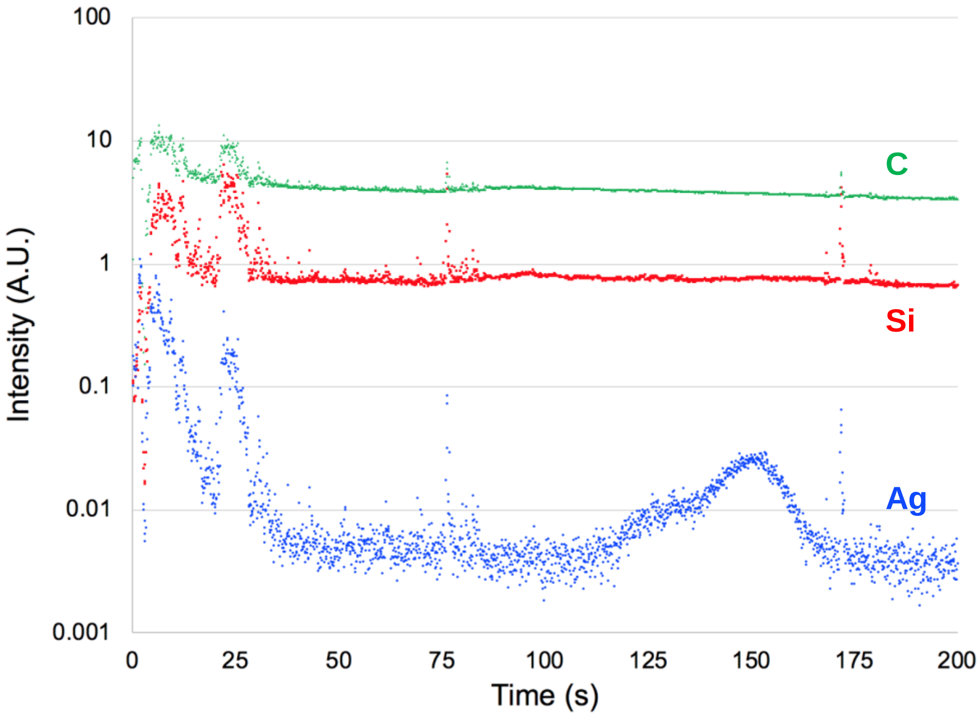

**Figure 8. GD-OES depth profile of 1700 ºC, 150 h 4H-SiC Ag sample. Time correlates to depth.**

The 4H-SiC samples consist of a 3–4 µm MTS-SiC seal coating layer over the direct ion implanted SiC layer. The MTS-SiC layer serves to protect the 4H-SiC layer from decomposition and act as a sink for the diffusing impurity species. In the analysis of the 1700 ºC, 150 h sample, a small shoulder extending into the MTS-SiC seal coating layer this is possibly indicative of diffusion into the seal-coating layer. In the 4H-SiC sample, the non-direct implanted side with also be profiled to observe potential segregated silver at the 4H-SiC/MTS-SiC interface. The presence of segregated silver on the opposite side would be indicative of lattice diffusion across the single crystal SiC sample. Insight on the diffusivity can be inferred through the difference in measured concentration as a function of exposure time.

Depth profiling analysis via GD-OES will continue on diffusion couple samples to measure the change in concentration profiles. However, the challenge with detection limits of the GD-OES technique appears to limit the direct observation of diffusion into the SiC layer. Past studies have successfully utilized secondary ion mass spectroscopy (SIMS) [12–14]. The SIMS analysis is cost prohibited and would greatly limit the total scope of the analysis. Rutherford backscattering spectroscopy (RBS) is planned through collaboration with the Michigan Ion Beam Laboratory. The RBS technique has potential to demonstrate greater sensitivity than GD-OES and is being pursued.

## 4. CONSTANT SOURCE APPROXIMATION: PD-AG FOIL DESIGN

An alternative design to the seal-coated diffusion couple approach was considered and has been developed to provide a constant source of reactant species in a PyC/SiC system. The effort is expected to demonstrate the impact of the PyC layer and variation of PyC layer density on Pd-Ag interactions. The concept was influenced by the high temperature interfacial reaction study by Demkowicz et al. [15] which explored Pd interactions with SiC and other material systems up to 1600 ºC. A schematic of the design is shown in Figure 9. The design is composed of an outer graphite sample holder which is utilized to ensure a constant contact on the planar diffusion couple system. The interior sample holder is also composed of graphite and has a recessed area cut out for the planar samples. The samples to be explored are excess non-implanted, non-seal-coated PyC/SiC/S-PyC samples which were produced in previous efforts. In the design the PyC/SiC interface of two identical samples face each other with a Pd-Ag foil between the two samples. The Pd-Ag foil is the diffusion source and a 75at%-Pd, 25at%-Ag foil ~250 µm thick has been purchased from Alfa Aesar.

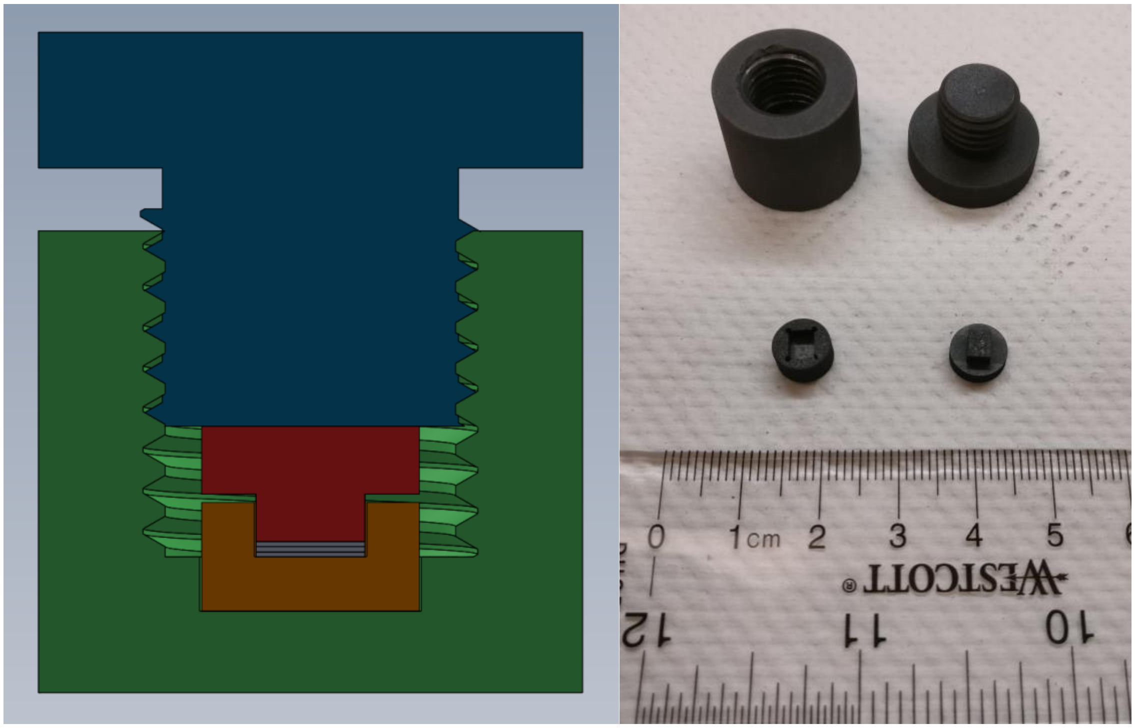

**Figure 9. Schematic of constant source PyC/SiC diffusion couple (left) and image of graphite components (right).**

Significant interaction between Pd and SiC was observed at 1000 ºC, 10 h in the interfacial reaction study while testing occurred up to 1200 ºC for Pd/SiC and up to 1400 ºC for Pd/TiN and Pd/TiC [15]. The addition of 25at% Ag lowers the melting temperature of the Pd foil to ~1400 ºC from ~1555 ºC which limits the ultimate operational temperature. A simple test matrix is shown Table 5. The test matrix will define the role of the PyC layer on protecting the SiC from Pd attack through a direct comparison of Baseline with a Baseline sample with no PyC. The role of varying PyC properties will be define by the PyC and Baseline comparison at 50 hours while the multiple time exposures will provide insight on penetration depths of Ag and Pd – these results will support and supplement the seal-coated study observations. The planned analysis is cross-sectional scanning electron microscopy analysis.

**Table 5. Simple test matrix for Pd-Ag foil study.**

| Sample | Temperature (ºC) | Exposure Time (h) |
|---|---|---|
| Baseline | 1200 | 50, 100, 150 |
| PyC | 1200 | 50 |
| Baseline - No PyC | 1200 | 50 |

## 5. SUMMARY

The past quarter focused on establishing and initiating sample exposures. The neutron irradiations have been approved and the DC01 and DC02 capsule irradiations have been completed. The capsules are currently awaiting disassembly. The thermal equivalent samples will be exposed after the capsule irradiation temperature is established. The high-temperature thermal testing of the diffusion couples is in progress. Initial GD-DOS results demonstrate the diffusion couple design behaves as planned, however, sensitivity issues have complicated the analysis approach. Alternative approaches have been identified and are being pursued to explore the impact of different variants.

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