

FY18Q2 Quarterly Report: Radiation Enhanced Diffusion of Ag, Ag-Pd, Eu, and Sr in Neutron Irradiated PyC/SiC Diffusion Couples



Tyler J. Gerczak
Anne A. Campbell
John D. Hunn
Brian C. Jolly
Austin T. Schumacher

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Fusion and Materials for Nuclear Systems Division

**FY18Q2 QUARTERLY REPORT: RADIATION ENHANCED DIFFUSION
OF AG, AG-PD, EU, AND SR IN NEUTRON IRRADIATED PYC/SIC
DIFFUSION COUPLES**

Tyler J. Gerczak
Anne A. Campbell
John D. Hunn
Brian C. Jolly
Austin T. Schumacher

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ACRONYMS

AGR	Advanced Gas Reactor (Fuel Development and Qualification Program)
AGR-1	First AGR fuel irradiation experiment
CGF	Coating gas fraction
CVD	Chemical vapor deposition
FBCVD	Fluidized-bed chemical vapor deposition
LIBS	Laser-induced-breakdown spectroscopy
MS	Methylsilane
MTS	Methyltrichlorosilane
PyC	Pyrolytic carbon or pyrocarbon
S-PyC	Support pyrocarbon
scm	Standard cubic centimeters per minute
SiC	Silicon carbide (TRISO layer)
TRISO	Tristructural-isotropic (coated particles)

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ABSTRACT

Obtaining accurate diffusion kinetics in materials representative of those found in tristructural-isotopic (TRISO) coated particle fuel is needed to predict the diffusive release of fission products in reactor. Planar diffusion couples with representative pyrocarbon (PyC) and silicon carbide (SiC) layers are being produced using the same fluidized-bed chemical vapor deposition (FBCVD) technology used to produce TRISO particles from the first irradiation experiment of the Advanced Gas Reactor (AGR) Fuel Qualification and Development Program. The layer properties of the planar diffusion couples are tailored to meet the specified PyC density and microstructure of the SiC layer as defined by the AGR program. The influence of these variables on diffusion is also being explored by producing PyC and SiC variants. The pathway to producing the diffusion couples is discussed and builds upon efforts described in previous reports [1,2].

1. INTRODUCTION

Planar pyrocarbon (PyC) and silicon carbide (SiC) diffusion couples are being fabricated to explore diffusion in various exposure conditions including high temperature and high temperature neutron irradiation environments. The PyC/SiC construction serves to represent the diffusion pathway for fission product species in tristructural-isotropic (TRISO) coated particle fuel and allows for diffusion analysis in representative systems to be obtained. Previous efforts focused on producing representative pyrocarbon (PyC) and silicon carbide (SiC) layered substrates for diffusion analysis. A summary of the previous results can be found in prior progress reports; ORNL/TM-2017/704 [1] and ORNL/TM-2018/766 [2].

The development of PyC/SiC layers used a FBCVD reactor which was identical to the system used to produce TRISO particles from the first AGR fuel irradiation experiment (AGR-1). The prior work resulted in identification of diffusion couple design consisting of a layered PyC/SiC/support-PyC (S-PyC). Here, the PyC and SiC layers represent the layers with tailored properties. The conditions to produce samples with targeted layer properties were confirmed for the three identified variants (Baseline, SiC Variant, PyC Variant) [2]. This report summarizes the work completed in FY18Q2 which focused on completing the ion implantation steps and developing an appropriate seal coating approach.

2. ION IMPLANTATION OF PYC/SIC/S-PYC SAMPLES

Three variants with unique properties were produced in prior efforts to study the influence of layer properties on the diffusion of select fission product species. The three variants were Baseline, SiC Variant, and PyC Variant. The planned test conditions are shown in Table 1. Two primary thrusts are planned to pursue the impact of neutron irradiation on diffusion and diffusion at high temperature conditions. This serves to better understand both in-pile behavior and diffusion during safety-testing as varying diffusion behaviors are likely at the different conditions [3,4]. Also, included in the test matrix are commercial SiC samples. The commercial SiC samples are polycrystalline 3C-SiC from Rohm and Haas and 4H-SiC single crystal samples from MSE Supplies. The commercial SiC samples are directly implanted and serve as a comparison to prior studies reported in the literature [5-8].

Table 1. Planned diffusion couple test matrix

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

The fission product species are introduced in the system by ion implantation into the representative PyC layer prior to deposition of a seal coating layer. The seal coating layer serves to isolate the diffusion system during thermal exposure. Figure 1 shows a cross-section view identifying the location of the implantation relative to the construction of the PyC/SiC/S-PyC diffusion couples.

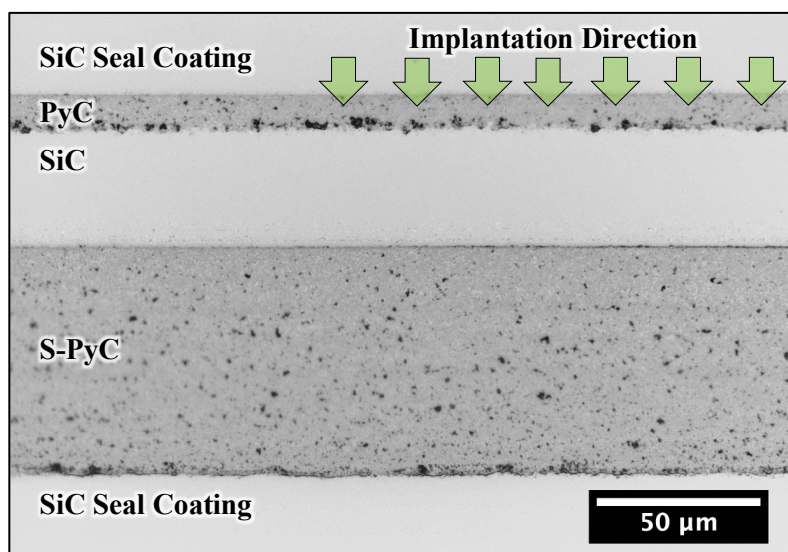


Figure 1. Optical micrograph of the PyC/SiC/S-PyC construction after SiC seal coating showing layer construction and location of ion implantation.

The ion implantations were conducted by the Michigan Ion Beam Laboratory at the University of Michigan using the 400-kV inline implanter known as “Blue”. Prior to implantation the samples were

sectioned to size. All Baseline samples were cut to 3-mm \times 5-mm, SiC Variant to 3-mm \times 4.5-mm, and PyC Variant to 3-mm \times 4-mm. A total of 120 PyC/SiC samples were sectioned for ion implantation. By sectioning to different lengths, the samples can be more easily identified after irradiation if positioning is lost during disassembly. The different sample geometries required the fabrication of a new implantation holder to accommodate the larger samples. The prior design was successful in producing shakedown samples [1] and as such the design was modified to accommodate the larger geometry samples. A total of 30 samples can be implanted simultaneously.

Table 2 lists the ion implantation conditions for each planned sample condition. The initial targeted fluences were selected based on insights from the AGR-1 end-of-life inventories [9]. The targeted fluences were selected to represent the total inventory of each fission product species in a TRISO particle at end-of-life and was normalized to areal density of the PyC/SiC interface in the new geometry. This approach has a number of assumptions namely the total inventory at end-of-life is available to diffuse through the PyC/SiC layer. This does not account for the kernel's ability to stabilize and retain fission products. The motivation to targeting the normalized end-of-life inventory was to be near the potential conditions expected in TRISO fuel operation. Over estimating the fraction of the inventory able to interact with the SiC layer also possibly improves analysis due to possible concerns of detection limits. The targeted fluence for the direct implantation of commercial-SiC samples (3C-SiC and 4H-SiC) was selected to avoid amorphization of the SiC in the implanted layer [10].

Various constraints to the diffusion couple development process required deviation from the targeted fluences. First, during seal coating development it was observed that significant loss of silver from the implantation surface was occurring [2]. Various seal coating approaches are being pursued to mitigate this issue, however, increasing the total silver fluence can aid in retaining a satisfactory fraction of the implantation dose as such a larger silver fluence was sought. A total fluence of 8.4×10^{16} ions/cm² was selected to increase the total fluences and remain within an order of magnitude of the targeted dose. Instrument time and material consumption were other constraints for conditions 2 through 4. For the palladium implantations, the amount of material consumed to reach the targeted fluence of 7.8×10^{17} ions/cm² would require multiple targets be purchased. This was cost prohibitive and not feasible for the experiment. The europium and strontium targets produced low current relative to silver and palladium. The low currents did not allow for the targeted fluences to be reached and a lower dose was accepted at 5.7×10^{16} ions/cm². The variations to the targeted fluences were acceptable based on the inherent assumptions of fraction of fission product inventory available to diffuse in the system. No change in the targeted fluence for the commercial-SiC implants was required.

Table 2. Ion implantation conditions for diffusion analysis.

Condition	Energy	Fluence (ions/cm ²) Targeted	Fluence (ions/cm ²) Actual	Temperature
1: Ag	400 kV	4.2×10 ¹⁶	8.4×10 ¹⁶	RT
2: Ag+Pd	400 kV	(Ag) 4.2×10 ¹⁶ , (Pd) 7.8×10 ¹⁷	(Ag) 8.4×10 ¹⁶ , (Pd) 8.4×10 ¹⁶	RT
3: Eu	400 kV	7.98×10 ¹⁶	5.7×10 ¹⁶	RT
4: Sr	400 kV	1.36×10 ¹⁸	5.7×10 ¹⁶	RT
5: Ag*	400 kV	1×10 ¹⁵	1×10 ¹⁵	300 °C

*Commercial-SiC samples (CVD-SiC and 4H-SiC)

Successful completion of the ion implantation step yielded 120 total samples. The breakdown of each implanted variant by coating run (DCCD-##) [2] is shown Table 3. An excess of samples exists required to investigate the conditions described in Table 1. This provides for duplicate samples to be investigated at select conditions.

Table 3. Breakdown of implantation by Variant and coating run.

	Baseline			PyC		SiC		CVD-SiC	4H-SiC
	DCCD-30	DCCD-33	DCCD-34	DCCD-31	DCCD-28	DCCD-32	DCCD-35		
1: Ag	18	-	-	6	-	14	-	-	-
2: Ag+Pd	2	10	-	10	-	-	-	-	-
3: Eu	-	10	5	-	10	5	-	-	-
4: Sr	-	-	15	-	-	-	15	-	-
5: Ag	-	-	-	-	-	-	-	30	30

3. SEAL COATING

Seal coating of the PyC/SiC/S-PyC samples is required to maintain an isolated system during neutron and high temperature exposure. An iterative approach has been undertaken to develop an optimal seal coat. An optimal seal coat is one that retains a majority of the implanted dose and presents a hermetic seal. To test the hermeticity of the seal coat samples are burned in air at 1000 °C for 5 hours and the mass change is measured to determine if the underlying PyC layers were compromised. The retention of the implantation dose is determined by glow discharge optical emission spectroscopy (GD-OES) at the Oak Ridge National Laboratory (ORNL). The GD-OES technique provides nanometer depth resolution and potential parts-per-billion sensitivity. The technique can profile 10's of microns of depth which is required for the buried surfaces of interest. For these reasons the GD-OES technique is preferred over laser-induced-breakdown spectroscopy (LIBS) which was first used to profile the implantation species.

3.1 GD-OES DEPTH PROFILING

The GD-OES analysis technique requires flat samples greater than 10-mm diameter to maintain vacuum and a consistent plasma over the surface. The as-fabricated samples (3-mm × 4-5-mm × ~0.2-mm) did not satisfy this requirement. The sample must also be centered over the 2-mm diameter anode to facilitate analysis of the small samples. Initial attempts to embed the samples did not prove successful. A new approach utilizing an aluminum centering ring and machined aluminum mount was developed to facilitate GD-OES depth profiling analysis of the seal coated PyC/SiC/S-PyC samples. Figure 2 shows the sample mount, centering ring, anode and positioning of the sample on the anode.

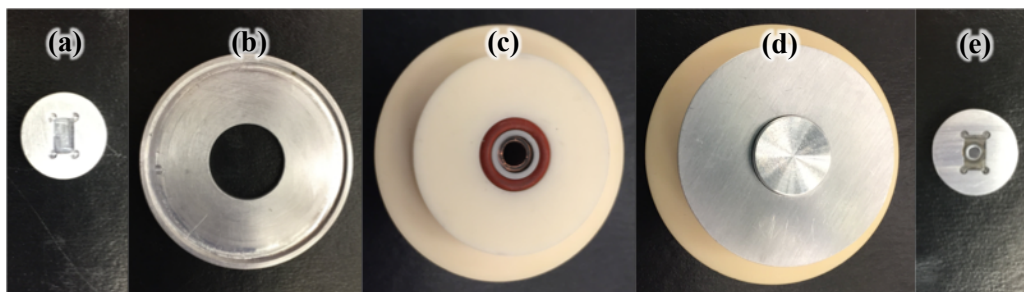


Figure 2. Example of (a) sample mount, (b) centering ring, (c) anode, (d) positioning of sample on the anode and (e) analyzed sample.

To create a flush surface the diffusion couple sample is secured to the bottom of the aluminum mount with adhesive glue. The void space surrounding the flat diffusion couple sample is filled with colloidal graphite to maintain a flat surface and conductive pathway from sample to aluminum mount. The sample is then thinned using an Allied Multiprep® system to control the amount of material removed. The aluminum mount is brought near flush or polished slightly into the SiC seal coat to ensure an appropriately flat surface is present to limit disruptions in the plasma during GD-OES analysis. Through application of this process repeatable GD-OES analysis has been demonstrated providing a high sensitivity depth profiling technique for confirmation of seal coating development and diffusion analysis.

3.2 DEVELOPMENT OF MS DERIVED SiC SEAL COATING PROCESS

The initial seal coating deposition conditions attempted to overcome the kinetics of silver release from the implantation surface by limiting time at temperature prior to initiating methyltrichlorosilane (MTS) derived SiC deposition. The MTS derived SiC was deemed optimal as it is similar to the base SiC layer and remains intact at safety testing temperature. The initial runs on non-implanted samples demonstrated the robustness of this design, however, initial scoping runs of the MTS derived SiC seal coating showed no measureable retained silver after coating [1,2]. This is likely due to rapid diffusion kinetics of silver in PyC at 1425 °C [11]. To avoid loss of silver, a low temperature diffusion barrier is being pursued. The use of low temperature diffusion barriers prior to high temperature MTS SiC deposition has been shown to be successful in retaining silver in prior experiments by López-Honorato, *et al.* [12]. Silicon carbide has been shown to be deposited below 1000 °C using methylsilane (MS) as a precursor gas in a FB-CVD system [13]. The use of MS to deposit SiC provides an opportunity to deposit a low temperature SiC barrier prior to the robust MTS derived SiC layer. In this work both MS and MTS SiC depositions occur in the FB-CVD reactor.

Table 4 lists the seal coating approaches pursued during this study. The first MS depositions (MS-Trial 1 through 4) were used to determine the feasibility of low temperature SiC deposition with subsequent runs pursued to determine optimal seal coating routes to obtain hermeticity and silver retention. Sample cross-sectioning and optical microscopy was routinely utilized to investigate the nature of the seal coating layer.

Table 4. SiC seal coating conditions.

Run	Precursor Gas	Run Time (min)	Temp. (°C)	Ar	H ₂	MS	CGF ^a	MTS used (g)
General MTS SiC Seal Coating	MTS	157	1425	3500	3500	-	0.023	175
MS-Trial-1	MS	120	900	6000	-	60	0.0099	-
MS-Trial-2	MS	120	600	6000	-	120	0.0196	-
MS-Trial-3	MS	120	700	6000	-	120	0.0196	-
MS-Trial-4	MS	121	700	6000	-	240	0.0385	-
MS-MTS-SC01-U	MS	33	700	6000	-	120	0.0196	-
	MTS	57	1425	3500	3500	-	0.022	60
MS-MTS-SC02-I	MS	60	700	6000	-	120	0.0196	-
	MTS	57	1425	3500	3500	-	0.022	60
MS-SC03-Ramp	MS	120	700-1000	6000	-	60-120	Var.	-
MS-SC04	MS	120	900	6000	-	60	0.0099	-
MS-SC05-I	MS	45	700	6000	-	60	0.0099	-
	MS	120	900	6000	-	60	0.0099	-
MS-SC06	MS	120	800	6000	-	60	0.0099	-

^aCGF is coating gas fraction, all gas flow rates are in standard cubic centimeters per minute (sccm).

The MS-Trial-# samples demonstrated low temperature SiC deposition was feasible. These runs were deposited on sapphire disks coated with PyC, with the exception of MS-Trial-4 which included a Ag⁺ ion implanted test sample to test for silver retention. The initial MS coating conditions were derived from the literature which served as a starting point for low temperature SiC deposition [13]. The first run was set at 900 °C which was the lowest the temperature could be monitored using the optical pyrometer. The following runs operated at a lower temperature after the bed temperature was calibrated using a physical thermocouple embedded in the furnace. Figure 3 shows examples of the coating for MS-Trial-1, MS-Trial-3 and MS-Trial-4. No apparent coating was observed for MS-Trial-2. The apparent thickness as measured from cross-section for the three trial runs are 12–13 μm, 9–13 μm, and 15–21 μm for MS-Trial-1, MS-Trial-3 and MS-Trial-4 respectively. The coating for MS-Trial -1 is more uniform over the surface of the sample while MS-Trial-3 and MS-Trial-4 show signs of cracking and irregular growth. Heating tests showed good stability from the 900 °C MS-Trial-1 sample while the 700 °C trials for the different coating gas fractions (CGF) showed a degree of cracking.

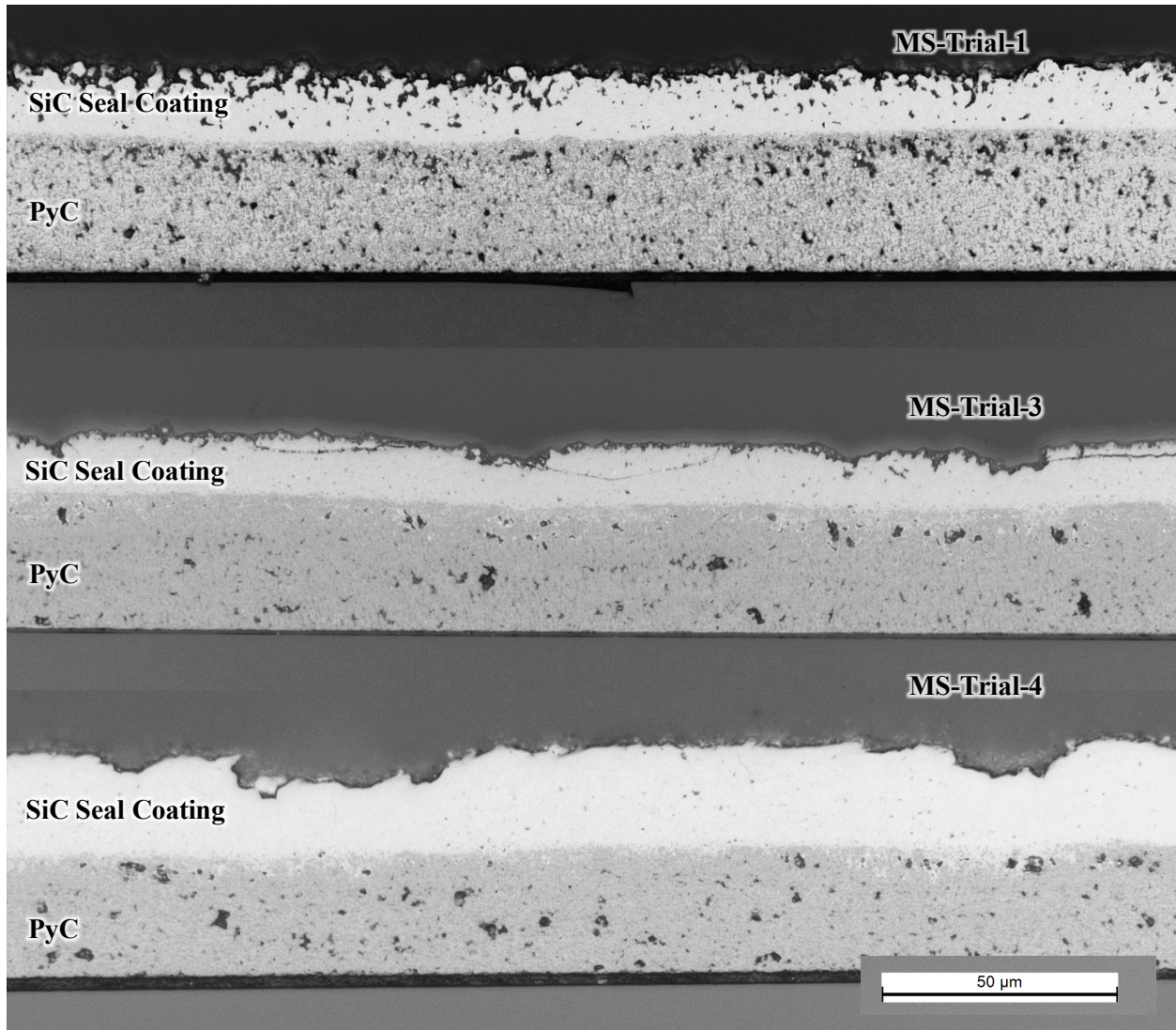


Figure 3. Optical images of cross-sections from MS-Trial-1, MS-Trial-3, and MS-Trial-4.

The GD-OES depth profile obtained for MS-Trial-4 is shown in Figure 4. The profile shows a clear peak associated with the implanted silver profile. A shoulder into the MS-SiC coating layer is observed and expected to be due to redistribution during deposition. The clear presence of silver is promising for the ability to retain silver (and subsequently palladium, europium, and strontium), however, the challenge still exists that a secondary seal coat must be applied to maintain structural integrity at elevated temperatures as the 700 °C sample does not perform adequately.

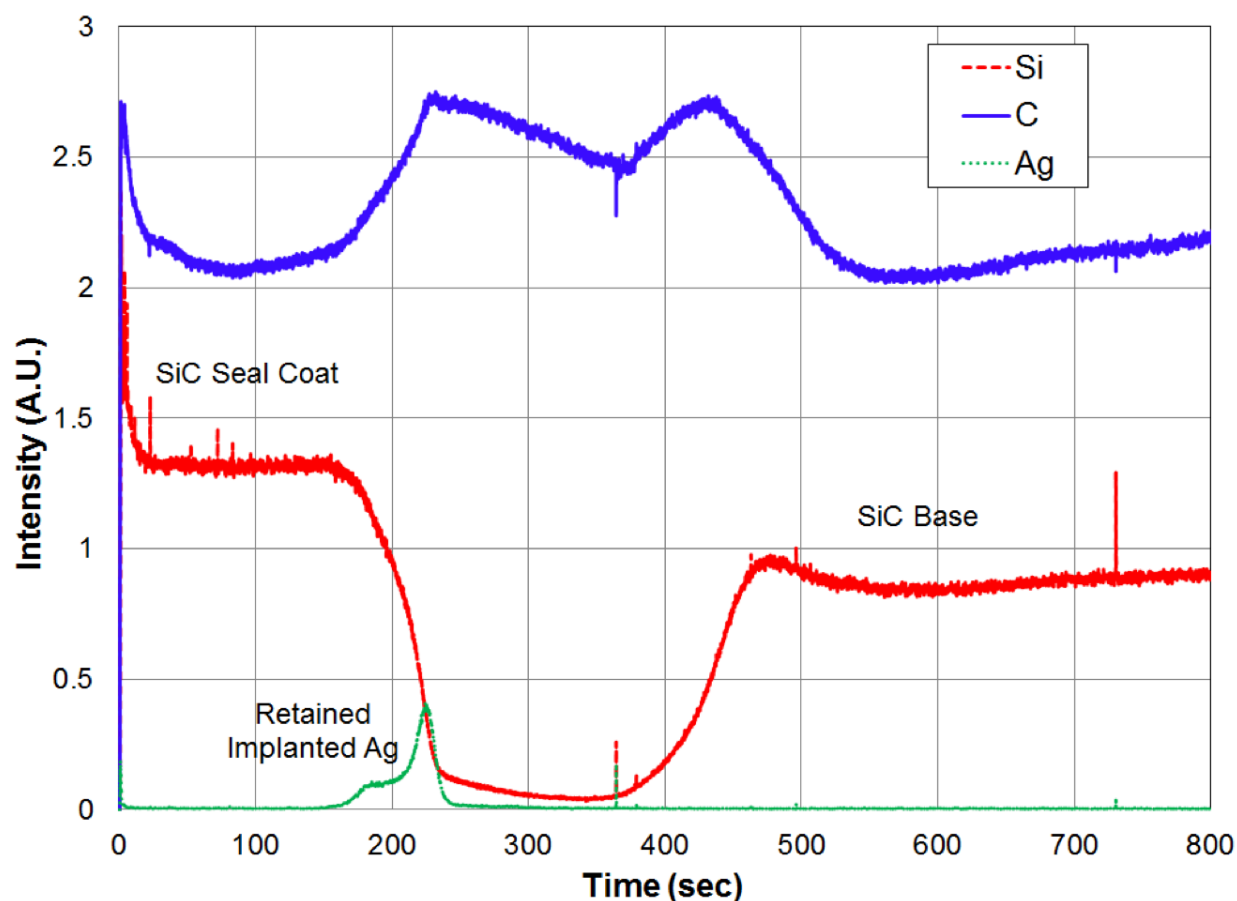


Figure 4. GD-OES depth profile showing retained implanted Ag for MS-Trial-4.

The subsequent runs attempted to first deposit the low temperature MS derived SiC followed by a high temperature MTS derived SiC as the MTS derived SiC is robust and stable at elevated temperatures. Two approaches were pursued which involved an uninterrupted coating process (MS-MTS-SC01-U) and interrupted coating process (MS-MTS-SC02-I). For the uninterrupted coating run the 700 °C MS derived SiC was deposited for 33 min to obtain a thin SiC layer followed by a 15 min ramp to 1425 °C before depositing MTS SiC. Cross section analysis showed (Figure 5) a dense SiC layer surrounding the entire sample, no clear distinction between the MS and MTS SiC was observed suggesting the MS SiC may have been lost during the ramp to MTS deposition conditions. Corresponding GD-OES analysis indicated no silver was retained in the coating layers. The interrupted coating run, MS-MTS-SC02-I, was attempted to overcome perceived loss of the MS coating during the ramp to MTS deposition. Here the 700 °C MS coated samples were allowed to cool down and removed from the system. They were inserted into the FB-CVD reactor at temperature (1425 °C) and MTS deposition was initiated after ~10 seconds. Cross-section analysis shows irregular coating behavior over the PyC surface (Figure 6) while a continuous coating is observed over the S-PyC surface. The GD-OES analysis (Figure 7) of MS-MTS-SC02-I showed limited silver at the PyC/seal coating interface suggesting an improvement in the process.

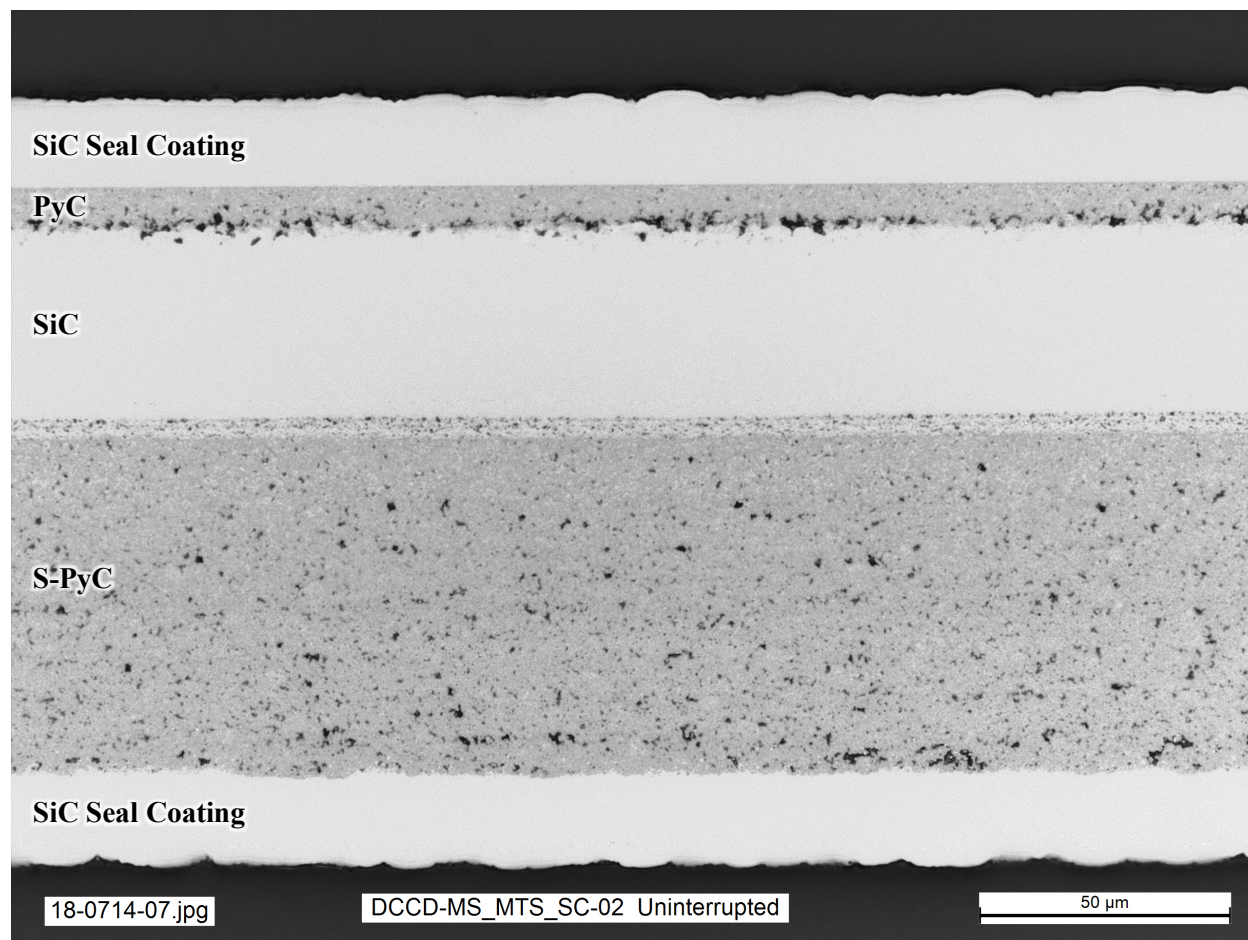


Figure 5. Cross-section optical image of MS-MTS-SC01-U, showing a dense, homogeneous coating layer over all surfaces.

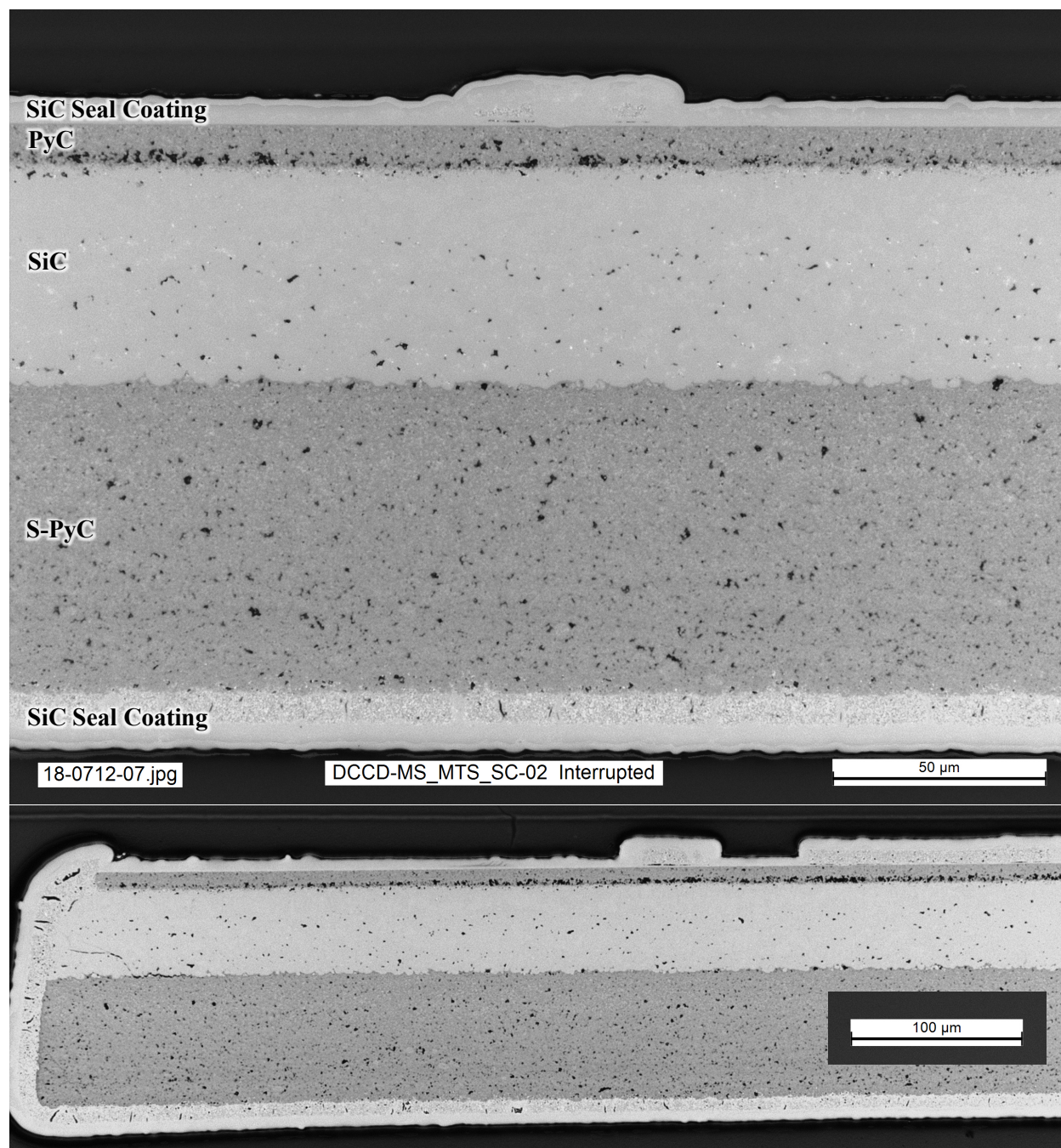


Figure 6. Cross-section optical image of MS-MTS-SC02-I, showing an irregular coating layer over the PyC surface.

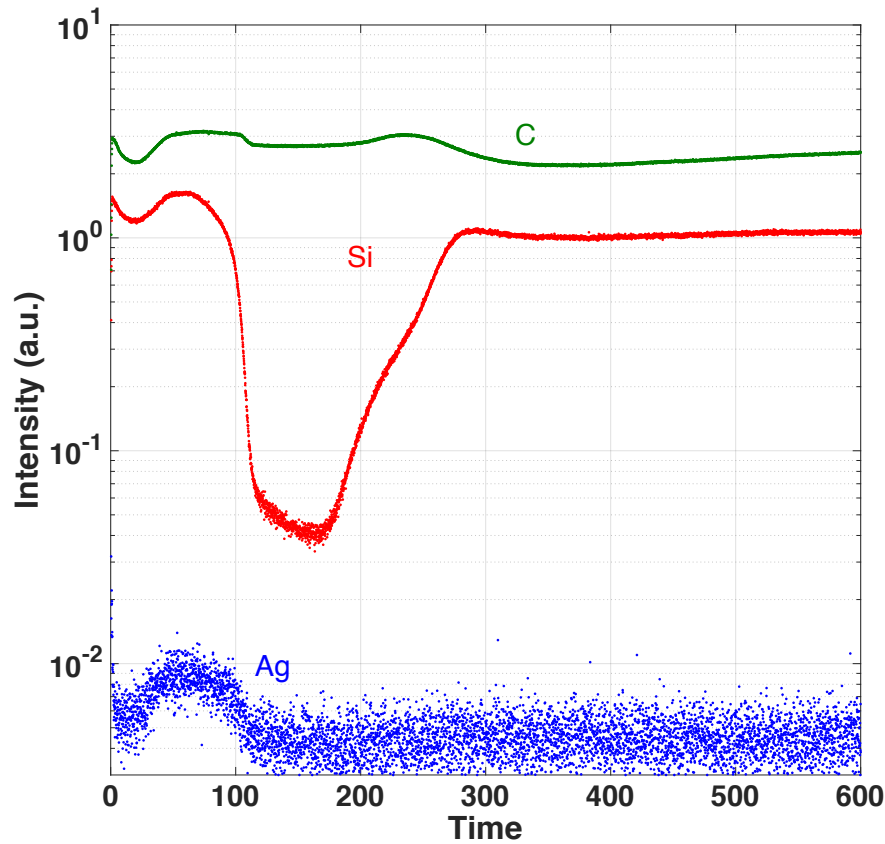


Figure 7. GD-OES analysis of MS-MTS-SC02-I.

The MS-SC03-Ramp run was performed to overcome the cracking and spallation associated with the 700 °C coating conditions over the PyC surface. With this run an initial 700 °C MS SiC layer was deposited for 60 min, a gradual ramp to 1000 °C and hold was performed during which MS SiC deposition was ongoing. The temperature ramp with simultaneous deposition was intended to avoid spallation due to thermal shock and provide continuous coating to heal potential cracks. A cross section of the MS-SC03-Ramp sample is shown in Figure 8. The surface over the PyC layer is irregular suggesting some degree of spallation of the initial coating layers. This is more evident when comparing the coating over the S-PyC layer which shows a more consistent coating however, numerous cracks are apparent in the coating layer. While this coating approach did not maintain its integrity during coating, as indicated by no silver retention and significant cracking, it does show some improvements in coverage can be gained by modifying the coating approach.

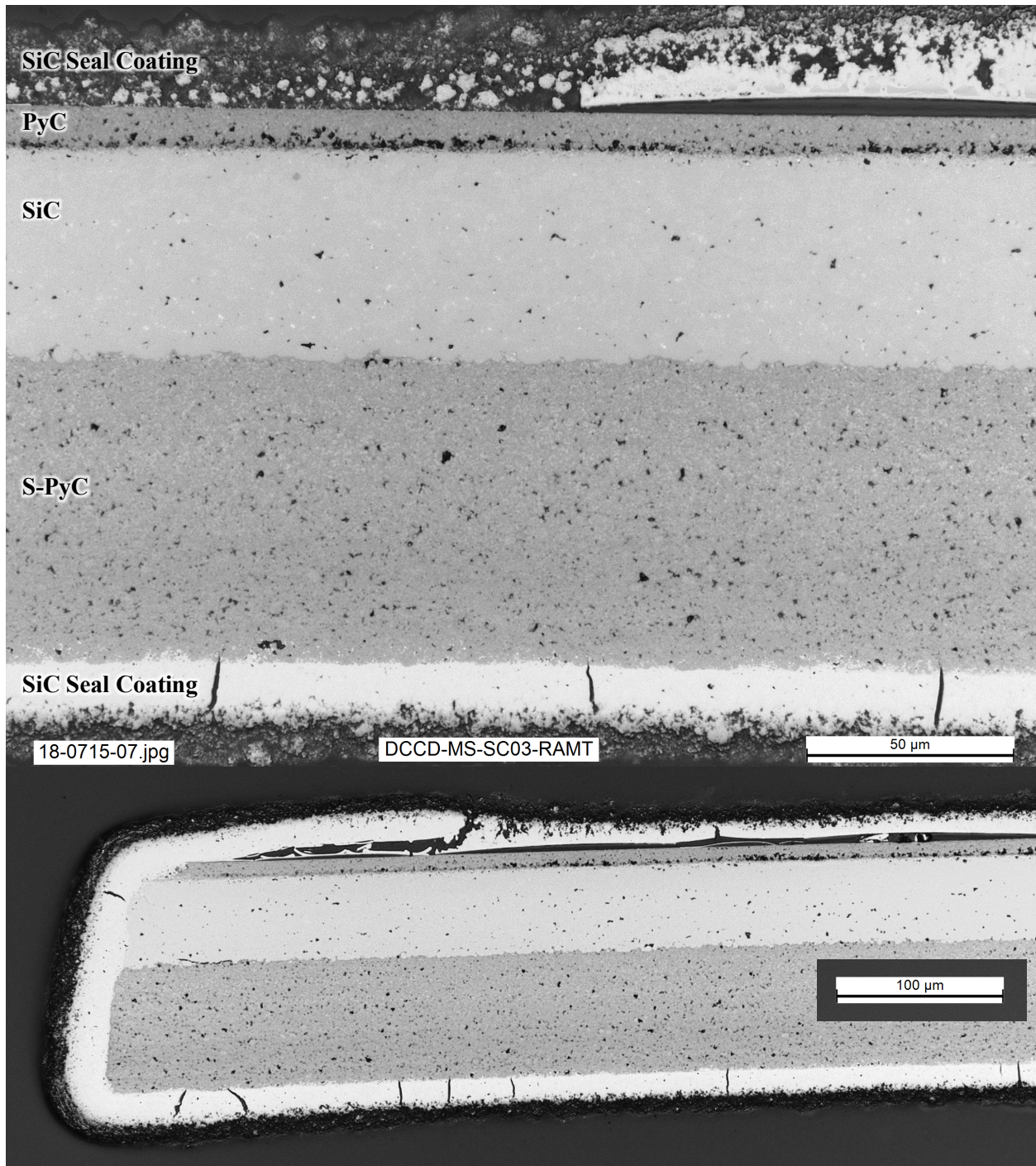


Figure 8. Cross section optical image of MS-SC03-Ramp, showing an irregular coating layer over the PyC surface and cracking in the coating layer over the S-PyC.

The most recent runs MS-SC04 and MS-SC06 were performed to confirm whether silver is retained at 900 °C and 800 °C coatings respectively as the coating properties appear to improve as a function of deposition temperature. The MS-SC05-I run was performed to determine if intact coatings can be

maintained in an intermediate step as the 900 °C conditions showed appropriate integrity. Cross-sectional analysis and GD-OES is ongoing.

3.3 SEAL COATING NEXT STEPS

Significant insight of the seal coating process has been gained in this last quarter, however, the current process is not sufficient to retain silver during the thermal exposures. The primary issue is poor adherence of the coating layer to the implanted PyC surface. Various coating methods are being pursued to deposit a retentive layer which possess stability. The ramp approach and interrupted approach at various temperatures appears to have merit to facilitate a 900 °C final coating. In particular, insight on the interrupted approach will be gained from MS-SC05-I. An alternative approach is to modify the implantation surface as the 700 °C coating showed good adherence to the more “rough” surface of the S-PyC in the interrupted run of MS-MTS-SC02-I. One such approach is to perform controlled low temperature oxidation of the PyC surface prior to coating. Care must be taken as the implantation peak is 180 nm into the surface. Oxidation can be used to introduce minor porosity on the surface which can aid in improving the adherence of the MS derived SiC seal coat.

4. SUMMARY

Samples from all three Variants and commercial SiC samples were sectioned, and ion implanted to facilitate the development of the representative PyC/SiC diffusion couples. To create an isolated diffusion system a seal coating around the PyC/SiC/S-PyC structure is required. Significant development has been undertaken to accomplish this. One positive observation is that silver was shown to be retained after a 700 °C coating providing a minimum condition in which silver can be retained.

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