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ORNL Data Compilation for the IAEA CRP-6 Coated Particle Fuel Quality Control Benchmark Exercise

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Four TRISO-coated particle samples were subjected to a battery of quality control (QC) measurements typically used at Oak Ridge National Laboratory for characterization and acceptance testing of coated particle fuel. The results of this analysis will be compared to similar analyses performed by the other participants in a QC benchmark exercise as part of the International Atomic Energy Agency (IAEA) Coordinated Research Project number 6 (CRP-6). The samples in this study were all produced on zirconia spheres as a surrogate material for the uranium bearing fuel kernel. Four of the participating organizations supplied these surrogate fuel samples. The sample suppliers were 1) the Babcock and Wilcox Company (B&W) in the USA, 2) the Korea Atomic Energy Research Institute (KAERI) in South Korea, 3) Pebble Bed Modular Reactor Ltd. in South Africa, and 4) the Oak Ridge National Laboratory (ORNL) in the USA. It should be stressed that the purpose of this exercise is for the comparison and benchmarking of the characterization methods and results. These surrogate coated particles are not necessarily representative of the baseline process used by each organization and conclusions should not be drawn regarding fuel performance based on the properties of these samples.

Table 1 lists the average values obtained for the various properties measured by ORNL. Detailed data for each of these measurements can be found in the following sections for each fuel sample.

Table 1. Summary of QC measurements

Property	ORNL	B&W	KAERI	PBMR
SiC sink/float density (Mg/m^3)	3.2033	3.1971	3.2058	3.2073
OPyC sink/float density (Mg/m^3)	2.0266	1.8885	1.4448	1.6336
Particle Diameter (µm)	907.4	881.2	1001.0	1076.2
Particle aspect ratio	1.044	1.039	1.055	1.041
Average buffer thickness (µm)	83.8	61.9	106.0	115.4
Average IPyC thickness (µm)	43.7	40.9	45.3	78.3
Average SiC thickness (µm)	31.7	32.1	35.4	29.6
Average OPyC thickness (µm)	37.1	38.4	46.0	53.1
IPyC anisotropy (BAFo)	1.0335	1.0205	1.0042	1.0058
OPyC anisotropy (BAFo)	1.0127	1.0168	1.0045	1.0056
Average particle weight (mg)	1.1120	1.0270	NM	NM
OPyC open porosity (ml/m^2)	0.0659	0.3690	NM	NM

NM = Not Measured

Limited scanning electron microscopy (SEM) and X-ray tomography was performed in order to demonstrate the capabilities of those methods. Figures 1 and 2 are composites of SEM images from each particle batch. In Figure 1, variation in microstructure such as porosity and interface intercalation can readily be resolved by scanning electron (SE) imaging. In Figure 2, back-scattered electron detection (BSE) can be used to resolve differences in SiC grain structure. Figure 3 is a composite of x-ray tomography images presented as two dimension cross sections through the center of each particle. Dimensional information such as particle size and shape and coating layer thickness can be extracted using this non-destructive method. Microstructural variations such as layer porosity can also be resolved. Additional SEM and X-ray images are included in each data section for the various particle samples.

Acknowledgement

This research was sponsored by the US-DOE Office of Nuclear Energy, Science and Technology's Advanced Gas Reactor Fuel Development and Qualification program and by the Oak Ridge National Laboratory, managed by UT-Battelle, LLC for the U.S. Department of Energy under contract no. DE-ACO5-00OR22725.

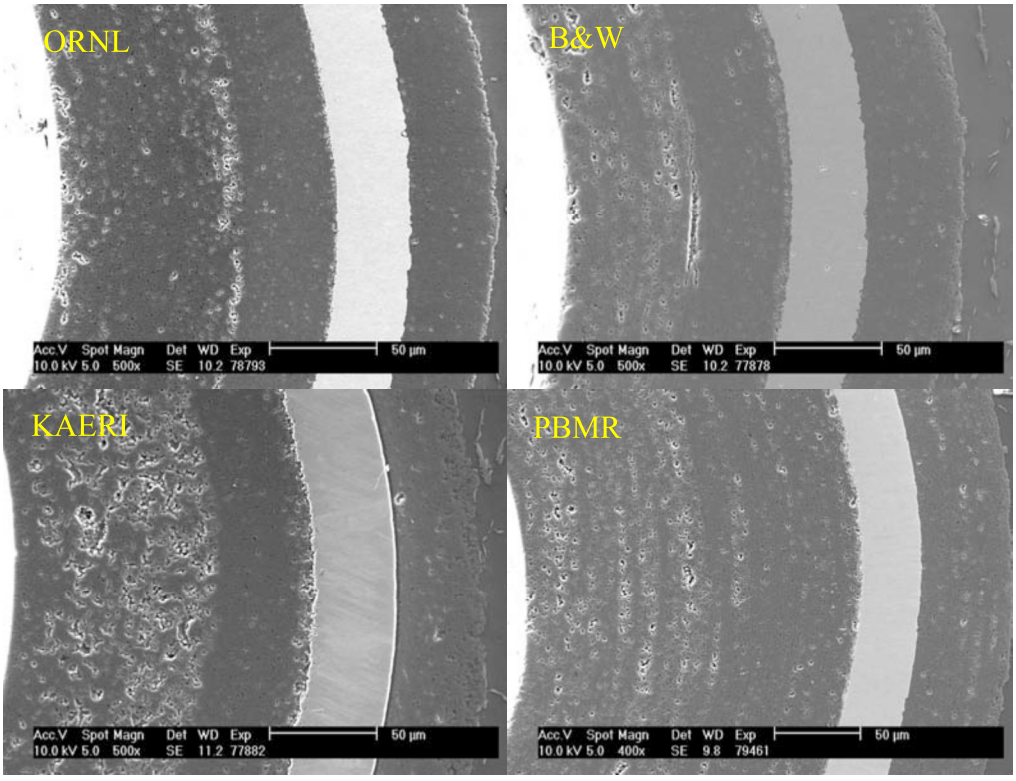


Figure 1. SE images of TRISO coatings.

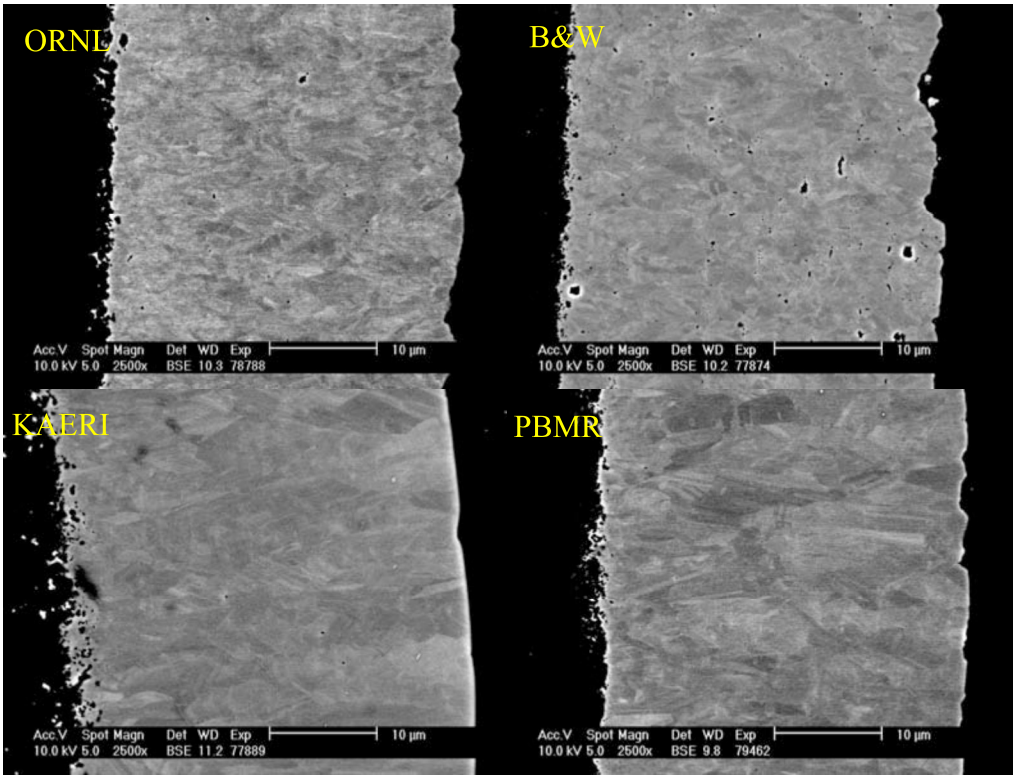


Figure 2. BSE images of SiC grain structure.

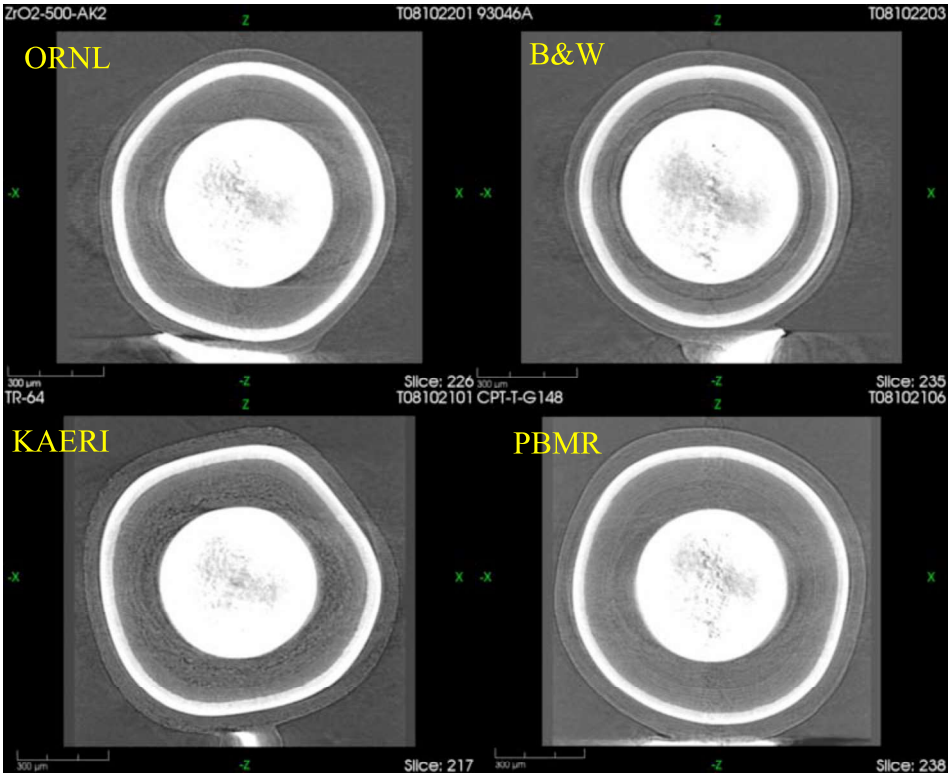


Figure 3. X-ray tomography cross-sections of TRISO particles.

Description of ORNL Characterization Methods

Mercury Porosimetry

A mercury porosimeter is used to determine kernel and buffer density, as well as open porosity in the IPyC and OPyC layers. The mercury porosimeter is essentially a volume measurement device. A sample of a few thousand particles is placed in a penetrometer cell with mercury and the air is evacuated. Because mercury does not wet to the sample, the mercury will tend to surround the particles with void space between the particles. The volume not occupied by the mercury is the called the *bulk volume*. Pressure is applied on the mercury to cause it to surround the individual particles. At some point the majority of void space in between the particles is filled with mercury and under this condition the mercury essentially enshrouds each individual particle in the sample. The volume not occupied by the mercury at this pressure is called the *envelope volume*. As pressure is increased beyond this point, the open pores at the particle surface are gradually filled, where the size of the penetrated opening is inversely related to the applied pressure. When all open porosity is filled, the volume not occupied by the mercury is called the *skeletal volume*. The difference between the envelope volume and the skeletal volume is therefore the open pore volume. These various types of volume and density are defined in ASTM standard D3766.

The envelope density of a sample of kernels is simply the mass of the kernel sample divided by the envelope volume. Note that this method does not resolve envelope volume of an individual kernel, but rather gives an average value for the measured sample. Buffer envelope density is somewhat more complicated to determine because the buffer cannot be isolated. The buffer envelope density is defined as the weight of the buffer divided by the volume of the buffer. This can not be measured directly so it is estimated using the average weight and volume of the buffer-coated particles and kernels. The porosimeter is used to measure the average envelope volume of a sample of buffer-coated particles. The average envelope volume of the kernels inside the buffer coated particles is then subtracted. The difference, being the envelope volume of the buffer, is then divided by the average weight of the buffer to calculate the envelope density. The average weight of the buffer is determined from the difference between the average weight of the buffer-coated particles and the average weight of the kernels inside. As for the kernel measurement, this analysis does not resolve the density of an individual buffer layer. In addition, because it is not feasible to separate the buffer from the IPyC layer in a fully coated particle, this analysis is not performed on the actual coated particle batch, but is instead performed on buffer-coated particles either removed during coating by hot sampling or obtained by interrupting the coating process.

Kernel and buffer envelope density were not measured for this QC Benchmark exercise because samples of bare kernels and buffer-only coated kernels were not available. OPyC open porosity was measured on the samples provided from ORNL and B&W. Sufficient material was not available in the samples from KAERI and PBMR. IPyC open porosity was not measured because this can not be done after deposition of the SiC layer. OPyC open porosity was calculated in units of ml/m^2 from the open pore volume of a sample divided by the surface area of that sample. Surface area was estimated from the approximate number of particles in the sample and the average volume of the particles (with the assumption of a spherical shape). Open pore volume was determined from the mercury intrusion over a pressure range of 250 to 10,000 psi. Note that

this pressure range is only appropriate for OPyC open porosity. IPyC open porosity can not be measured at higher pressures (typically above 3000 psi) because of compression of the IPyC/buffer layers.

Average Particle Weight

Average particle weight is needed for the calculation of open porosity and is also used to determine the number of particles in a large sample for statistical calculations in several of the attribute property inspections not performed for this benchmarking exercise. Average particle weight was determined by weighing and counting several samples containing 100-200 particles each. These particle samples are prepared by rotary riffling. It is very important that these samples be obtained in a random manner. Manual selection of particles tends toward the selection of larger particles which biases the average particle weight determination.

Coating Layer Density by Liquid Density Gradient Column

A liquid density gradient column is used to determine sink-float density of the IPyC, SiC, and OPyC layers. The sink-float density of a material lies in value between the envelope and skeletal densities discussed above. This is because the liquid in the column, which wets the samples, is able to partially penetrate the open porosity. A liquid density gradient column is created by filling a glass column with two liquids of different density, where the ratio of the two liquids is varied during filling in order to create a linear density as a function of the column height. This linear density gradient is determined by measuring the zero buoyancy position of calibrated floats. Different liquid density gradient columns with different density liquids are used to create columns for measuring pyrocarbon (typical range 1.8 – 2.0 g/cc) and SiC (typical range 3.19-3.21 g/cc). The OPyC layer densities of the four samples in this exercise varied beyond the range of the typical ORNL density columns. Different recipes and calibration standards were used for each sample to obtain a range that spanned the OPyC density of that sample.

Samples of the IPyC, SiC, and OPyC layers are obtained by fracturing the coatings on individual coated particles. Free-standing OPyC layer fragments were picked out of the fractured coatings. The OPyC layer could be separated on some particles because of the weak bonding between the SiC and OPyC layers. The IPyC density was not measured for this benchmarking exercise because free-standing IPyC layer fragments usually can not be obtained after deposition of the SiC because of the impregnation of SiC into the open porosity of the IPyC layer. For this reason, IPyC density must be determined using hot sampling or interrupted batches, similar to buffer density analysis discussed above. Free-standing SiC was obtained by picking out multiple layer fragments and heating in air to 850°C for 2 hours to remove the attached pyrocarbon. Care must be exercised to not excessively oxidize the SiC by heating at too high a temperature or for too long a time. The typical burn-off conditions of 850°C could not be used for the PBMR sample due to apparent oxide formation on the inner surface. For this sample the burn-off temperature was lowered to 750°C for 2 hours. Layer fragments were placed in the appropriate liquid density gradient column. The sink-float density of the fragments was determined by measuring the zero buoyancy position in the column. The mean and standard deviation for the measured density of each fragment was calculated.

Particle Dimensions

Optical microscopy was used to measure particle size and shape and coating thickness. Particle size and shape were measured using a shadowgraphic technique. Transmitted light was used to produce silhouettes of the particles. Analysis of these images produces information on mean particle diameter and aspect ratio. Aspect ratio is defined as the maximum diameter divided by the minimum diameter. Particles were mounted in a monolayer in a large transparent tray and imaged by acquiring a series of tiled photographs using an automated microscope.

Coating thickness was measured by preparing polished cross-sections and imaging with reflected light. Samples were prepared by arranging a number of particles in a square planar array and then mounting them in clear thermoplastic epoxy. The mounted particles were then ground down and polished to expose a mid-plane cross-section. These cross-sections rarely present a planar section that passes exactly through the center of the particle. This results in an imaged layer thickness that is greater than the thickness along the radial direction of the particle. The magnitude of the error can be minimized by polishing the particles as close to the mid-plane as possible. However, for maximum accuracy, a simple geometric correction was applied by calculating the offset of the imaged plane using the polish down distance and the measured particle radius in the plane of polish, with the assumption of a spherical geometry.

Imaging of the polished cross-sections was performed using a computer automated optical microscope. The microscope is capable of automatically positioning each particle in the field of view, acquiring a 12 megapixel image, and saving this image to a server for future processing. Quantified analysis of images obtained for size and shape or coating thickness was done using an ORNL developed image analysis program. Manual measurements can introduce error from operator bias and tend to be more limited because of the required effort. Automated image analysis allows for both more samples to be analyzed and for more measurements to be made on each imaged sample. For size and shape determination, the image analysis program identifies approximately 360 points around the outer boundary of each particle silhouette and calculates average diameter and aspect ratio. For coating layer thickness, the image analysis program identifies approximately 720 points on each layer interface and calculates radial coating thickness for each layer.

Pyrocarbon Anisotropy using the ORNL 2-MGEM

The ORNL Two-Modulator Generalized Ellipsometry Microscope (2-MGEM) was designed to determine pyrocarbon anisotropy at much greater accuracy and sensitivity than can be achieved with an optical polarimeter. The 2-MGEM uses two polarizer-photoelastic modulator pairs, oscillating at two different frequencies in the kilohertz range, to generate and analyze elliptically polarized light. The 2-MGEM can determine the optical polarization properties of the pyrolytic carbon by reflecting the polarized light beam at near normal incidence from a polished cross-section of the coated particle. This instrument fully determines the change in the elliptical polarization of the light reflected from the pyrocarbon surface and determines all the elements of the Mueller matrix. From this matrix, the diattenuation (N) can be extracted. The diattenuation is directly related to the optical Bacon anisotropy factor (BAF_o) by the equation,

$$BAF_o = (1+N)/(1-N).$$

Samples are mounted and polished similar to the methods used for imaging with an optical microscope. A computer-controlled stage is used to scan the sample beneath the 2-MGEM. This makes it possible to obtain detailed images of the diattenuation with a resolution as low as 2 µm. Figure 4 shows a particle cross-section imaged for reflected light intensity using an optical microscope and for diattenuation intensity using the 2-MGEM. Note that the kernel has been removed from this particle and replaced with epoxy. The figure shows some diattenuation from the hole in the epoxy, but this is unimportant. Figure 4 shows a relatively high diattenuation in the IPyC layer, moderate diattenuation in the OPyC layer, low diattenuation in the buffer, and none in the SiC. Figure 5 shows the distribution of the diattenuation in each of the carbon layers. Typically, the observed variation in diattenuation within each particle is much larger than the variation of the average diattenuation from particle to particle within a batch. The observed variation in diattenuation within each particle is much larger than the uncertainty in the measurement and has been shown to be very reproducible.

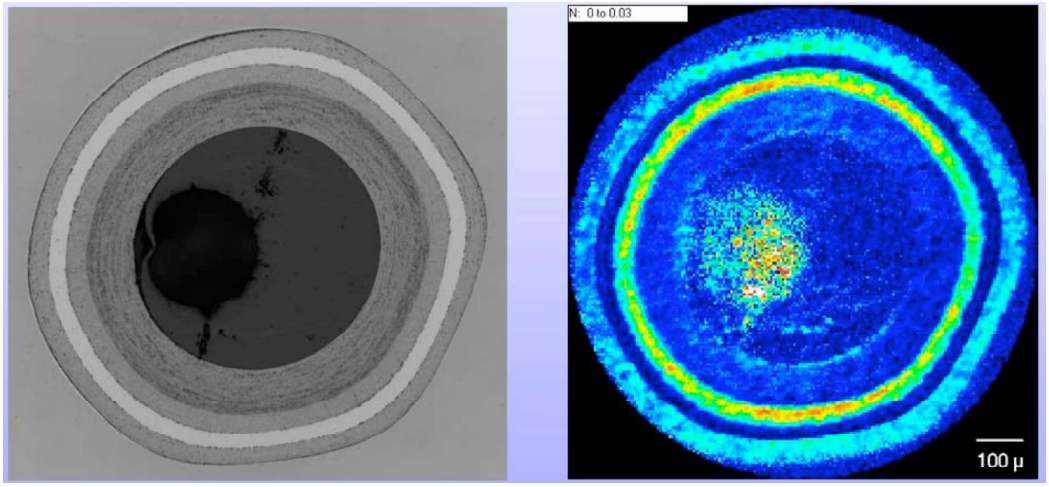


Figure 4. On left, image using intensity of reflected light. On right, image using diattenuation of reflected light with color temperature scaled to the diattenuation.

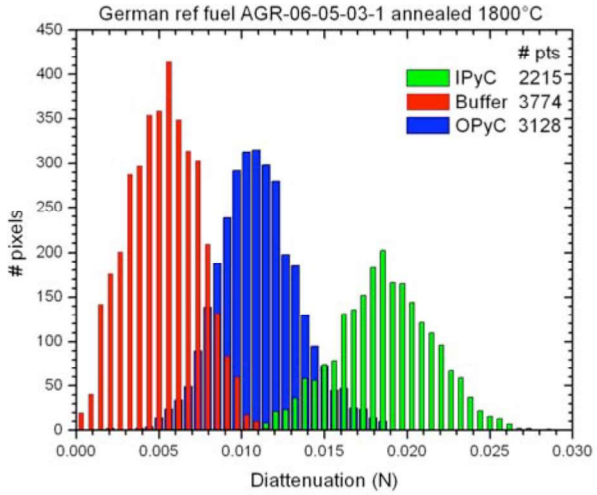


Figure 5. Histogram of diattenuation in Figure 4.

Scanning Electron Microscopy

The scanning electron microscope is another standard tool used at ORNL for characterization of coated particles. In many cases, it offers enhanced microstructural analysis beyond the capability of the optical microscope. In addition to standard secondary electron imaging to image layer and interface structure, backscattered electron imaging was used to take advantage of the variation in electron stopping power with grain orientation to image the individual SiC grains. At ORNL, this approach is used instead of acid etching to reveal the SiC grain boundaries.

X-ray Analysis

X-ray radiography is a powerful non-destructive analysis tool that is also applied at ORNL to characterize coated particle fuel. X-ray imaging allows the interior microstructure of a coated particle to be viewed without the need for materialographic cross-sectioning. A benchtop X-ray microtomograph system capable of 1-2 μm resolution was used to image one particle from each sample.

The x-ray source was at 40kV and 8W. Source and detector positions were 1 m and 1 cm, respectively. Tomographs were taken using a 20x objective, which corresponds to a field of view of approximately one cubic millimeter. Each particle mount was made by gluing an individual particle to a fine stainless steel wire with cyanoacrylate adhesive. The tomographs were performed from -90° to 90° with 451 images and with 15 seconds per image. X-ray images were taken at full resolution (0.61 μm/pixel), but pixels were binned by a factor of 4 to reduce the necessary computations for tomographic reconstruction.

X-ray microtomography of TRISO particles has associated imaging artifacts. The x-ray beam cannot penetrate the kernel due to the high atomic number of the cations of the ceramic kernels used in this study. Therefore, regions of the particle are blocked from the x-ray beam in certain angular orientations during the imaging. Tomographic reconstruction inherently assumes no x-ray blockage, so artifacts, such as faint arcs and blurring, are created in the reconstruction, although this only occurs in the z (height) range of the kernel, where the layers are partially shadowed by the kernel. In addition, the rotational stage motor produces a small wobble; even with computational corrections, this wobble causes a slight blurring in the tomographic reconstruction near the rotation axis through the particle. Tomographs also typically show some faint concentric rings and arcs (which computational methods can partially correct); however, careful study of an image can distinguish concentric artifacts from circumferential features in TRISO particles.

QC of ORNL TRISO Sample

This section contains the detailed data for the characterization measurements performed on ORNL TRISO sample ZrO2-500-AK2-S01.

On the following page is an inspection report form that summarizes the results of each measurement. The mean and standard deviation for each measurement is listed in the table, where appropriate. Also provided on this form are the statistically calculated 95% confidence limits for the mean and dispersion of the parent batch of material.

A and B are the minimum and maximum predicted values for the mean of the parent batch up to the 95% confidence level.

$$A = \bar{x} - \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$$B = \bar{x} + \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$\bar{x}$ and σ_x are the mean and the standard deviation obtained from the measurement performed on a sample of n items. t_{n-1} is the one-tailed Student's-t value for n-1 degrees of freedom.

C is the minimum value for which all but 1% of the population is above (at 95% confidence). D is the maximum value for which all but 1% of the population is below (at 95% confidence).

$$C = \bar{x} - k_n\sigma_x$$

$$D = \bar{x} + k_n\sigma_x$$

k_n is the one-tailed tolerance factor for a sample of n items, given a 1% tolerance limit and a 95% confidence level.

Following the inspection report form are the individual data report forms for each measurement. These forms provide more detail of the measurement performed and include either data for each measured item or histograms of this data.

At the end of this section are images of a few particles obtained using scanning electron microscopy and x-ray tomography. These images show the typical microstructure of the coating layers and interfaces.

Inspection Report Form IRF-CRP6: QC Benchmark Measurements on Coated Particles

Coated particle composite ID:	ZrO2-500-AK2-S01
Coated particle composite description:	ORNL CRP-6 QC Benchmark TRISO

Property	Measured Data				95% Confidence Limits for Particle Lot			Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value				
SiC sink/float density (Mg/m³)	3.2033	0.0063	23	1.717	mean ≥ A	A = x - ts/√n	3.201	DRF-02
					mean ≤ B	B = x + ts/√n	3.206	
				3.206	99% of population > C	C = x - ks	3.183	
					99% of population < D	D = x + ks	3.223	
OPyC sink/float density (Mg/m³)	2.0266	0.0040	46	1.679	mean ≥ A	A = x - ts/√n	2.026	DRF-03
					mean ≤ B	B = x + ts/√n	2.028	
				2.882	99% of population > C	C = x - ks	2.015	
					99% of population < D	D = x + ks	2.038	
Particle Diameter (μm)	907.4	16.0	1449	1.646	mean ≥ A	A = x - ts/√n	906.7	DRF-07 DRF-10
					mean ≤ B	B = x + ts/√n	908.1	
				2.412	99% of population > C	C = x - ks	868.8	
					99% of population < D	D = x + ks	946.0	
Particle aspect ratio	1.044		1449		number of particles > 1.14		0	DRF-07 DRF-10
Average buffer thickness for each particle (μm)	83.8	5.5	124	1.657	mean ≥ A	A = x - ts/√n	83.0	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	84.6	
				2.640	99% of population > C	C = x - ks	69.3	
					99% of population < D	D = x + ks	98.3	
Average IPyC thickness for each particle (μm)	43.7	2.5	110	1.659	mean ≥ A	A = x - ts/√n	43.3	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	44.1	
				2.662	99% of population > C	C = x - ks	37.0	
					99% of population < D	D = x + ks	50.4	
Average SiC thickness for each particle (μm)	31.7	1.1	141	1.656	mean ≥ A	A = x - ts/√n	31.5	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	31.9	
				2.619	99% of population > C	C = x - ks	28.8	
					99% of population < D	D = x + ks	34.6	
Average OPyC thickness for each particle (μm)	37.1	1.9	164	1.654	mean ≥ A	A = x - ts/√n	36.9	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	37.3	
				2.595	99% of population > C	C = x - ks	32.2	
					99% of population < D	D = x + ks	42.0	
IPyC anisotropy (BAFo)	1.0335	0.0023	10	1.833	mean ≥ A	A = x - ts/√n	1.0322	DRF-18
					mean ≤ B	B = x + ts/√n	1.0348	
				3.981	99% of population > C	C = x - ks	1.0243	
					99% of population < D	D = x + ks	1.0427	
OPyC anisotropy (BAFo)	1.0127	0.0004	10	1.833	mean ≥ A	A = x - ts/√n	1.0125	DRF-18
					mean ≤ B	B = x + ts/√n	1.0129	
				3.981	99% of population > C	C = x - ks	1.0111	
					99% of population < D	D = x + ks	1.0143	
Average particle weight (mg)	1.1120	0.0029	5 measurements	2.132	mean ≥ A	A = x - ts/√n	1.1093	DRF-22
					mean ≤ B	B = x + ts/√n	1.1147	
			828 particles	5.741	99% of population > C	C = x - ks	1.0955	
					99% of population < D	D = x + ks	1.1285	
OPyC open porosity (ml/m²)	0.0659		1 measurement					DRF-31
			2949 particles					

Comments
Sample was analyzed by SEM for SiC grain structure and general microstructure of layers and interfaces. A particle was imaged using high resolution x-ray tomography.

John Kim — QC Supervisor

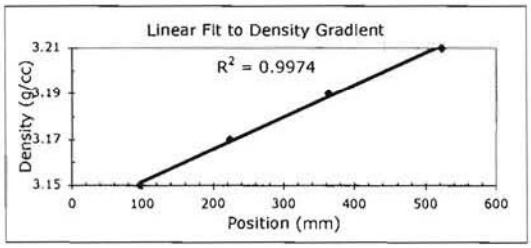
10-30-08 — Date

Data Report Form DRF-02: Measurement of SiC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-02 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08062001_DRF02R3.xls
Sample ID:	ZR02-500-AK2-S01
Sample description:	ORNL CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.4 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
3.150	82.96	103.17	96.43
3.170	208.23	231.89	224.00
3.190	347.99	370.86	363.24
3.210	506.92	530.74	522.80

Linear Fit			
slope	StDev	intercept	StDev
1.41E-04	2.37E-06	3.14E+00	7.15E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	377.25	3.1907	26			51		
2	367.78	3.1893	27			52		
3	387.39	3.1921	28			53		
4	398.98	3.1937	29			54		
5	406.39	3.1948	30			55		
6	456.93	3.2019	31			56		
7	465.14	3.2030	32			57		
8	475.83	3.2046	33			58		
9	483.66	3.2057	34			59		
10	484.40	3.2058	35			60		
11	486.66	3.2061	36			61		
12	490.42	3.2066	37			62		
13	490.42	3.2066	38			63		
14	490.86	3.2067	39			64		
15	492.18	3.2069	40			65		
16	493.70	3.2071	41			66		
17	497.45	3.2076	42			67		
18	494.12	3.2071	43			68		
19	496.82	3.2075	44			69		
20	501.38	3.2082	45			70		
21	503.30	3.2084	46			71		
22	501.28	3.2081	47			72		
23	498.95	3.2078	48			73		
24			49			74		
25			50			75		

Average density of SiC fragments:	3.2033
Standard deviation in density of SiC fragments:	0.0063
Uncertainty in calculated density of SiC fragments:	0.0014

Dixie Barker
Operator

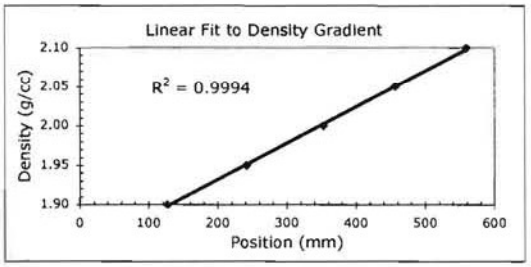
6-20-08
Date

Data Report Form DRF-03: Measurement of PyC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08061101_DRF03R3_high range.xls
Sample ID:	ZrO2-500-AK2-S01
Sample description:	ORNL CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.0 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.900	124.18	129.88	127.03
1.950	237.74	245.15	241.45
2.000	349.34	356.83	353.09
2.050	452.66	459.61	456.13
2.100	555.97	562.38	559.18

Linear Fit			
slope	StDev	intercept	StDev
4.64E-04	2.80E-06	1.84E+00	1.05E-03



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	384.07	2.0171	26	406.50	2.0275	51		
2	384.17	2.0172	27	407.90	2.0282	52		
3	385.53	2.0178	28	407.90	2.0282	53		
4	397.12	2.0232	29	405.11	2.0269	54		
5	397.73	2.0235	30	405.58	2.0271	55		
6	398.60	2.0239	31	406.40	2.0275	56		
7	399.31	2.0242	32	408.03	2.0282	57		
8	398.35	2.0237	33	408.03	2.0282	58		
9	399.30	2.0242	34	408.03	2.0282	59		
10	400.15	2.0246	35	408.10	2.0283	60		
11	401.39	2.0252	36	411.12	2.0297	61		
12	401.59	2.0253	37	414.54	2.0313	62		
13	402.19	2.0255	38	416.33	2.0321	63		
14	403.19	2.0260	39	418.82	2.0332	64		
15	403.19	2.0260	40	423.61	2.0355	65		
16	403.23	2.0260	41	433.94	2.0403	66		
17	403.61	2.0262	42	399.48	2.0243	67		
18	403.87	2.0263	43	403.18	2.0260	68		
19	403.87	2.0263	44	404.00	2.0264	69		
20	403.87	2.0263	45	406.03	2.0273	70		
21	400.14	2.0246	46	408.01	2.0282	71		
22	405.54	2.0271	47			72		
23	404.04	2.0264	48			73		
24	405.45	2.0270	49			74		
25	405.45	2.0270	50			75		

Average density of PyC fragments:	2.0266
Standard deviation in density of PyC fragments:	0.0040
Uncertainty in calculated density of PyC fragments:	0.0016

Dixie Barker
Operator

6-11-08
Date

Data Report Form DRF-07: Imaging of Particle Diameter and Aspect Ratio Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-07 Rev. 1
Operator:	Cliff Davisson
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP6 QC Benchmark TRISO Sample
Folder name containing images:	\\Mc-agr\AGR\ImageProcessing\P08042202\

DMR Calibration Expiration Date:	9/20/2008
Stage Micrometer Calibration Expiration Date:	2/13/2009
Measured Value for 1200 μm in Stage Micrometer Image:	1200. μm

Clifford C. Davisson
Operator

4/22/08
Date

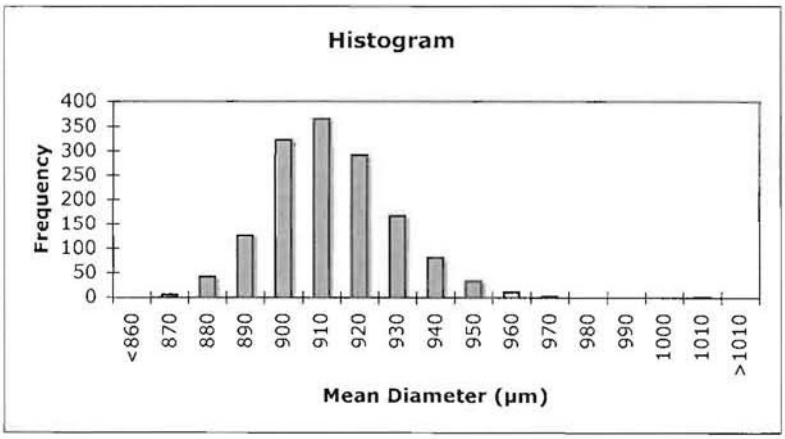
Data Report Form DRF-10A: Measurement of Particle Diameter

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Cliff Davisson / Andy Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08042202\
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP6 QC Benchmark TRISO Sample
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08042202_output\

Number of particles analyzed:	1449
Mean of the average diameter of each particle (μm):	907.4
Standard deviation in the average diameter of each particle (μm):	16

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<860	0
870	7
880	42
890	126
900	322
910	365
920	291
930	168
940	81
950	33
960	11
970	2
980	0
990	0
1000	0
1010	1
>1010	0



Clifford C. Davisson / Andy Kercher
Operator

5/05/08
Date

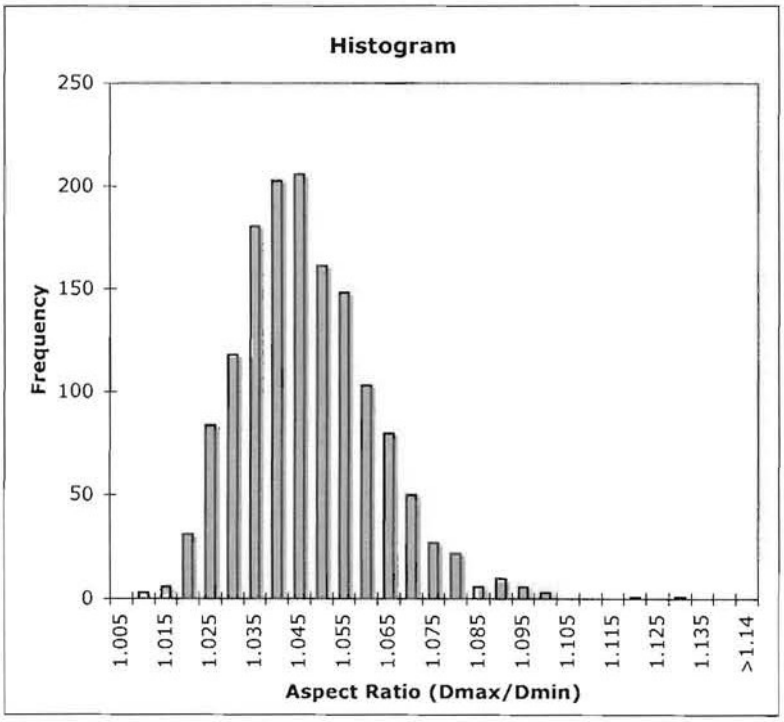
Data Report Form DRF-10B: Measurement of Particle Aspect Ratio (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Cliff Davisson / Andy Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08042202\
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP6 QC Benchmark TRISO Sample
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08042202_output\

Number of particles analyzed:	1449
Number of particles with aspect ratio ≥ 1.14	0
Average particle aspect ratio:	1.044

Distribution of the aspect ratio (top binned)

Aspect Ratio (D)	*Frequency*
1.005	0
1.010	3
1.015	6
1.020	31
1.025	84
1.030	118
1.035	180
1.040	203
1.045	206
1.050	161
1.055	148
1.060	103
1.065	80
1.070	50
1.075	27
1.080	22
1.085	6
1.090	10
1.095	6
1.100	3
1.105	0
1.110	0
1.115	0
1.120	1
1.125	0
1.130	1
1.135	0
1.140	0
>1.14	0



Clifford C. Davisson Robert K. Kercher — 5/05/08

Operator — Date

Data Report Form DRF-08-10x: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	ZrO2-500-AK2-S01
Sample description:	ORNL CRP-6 QC Benchmark TRISO
Mount ID number:	M08071502
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08102801\P0810280101\

DMR calibration expiration date:	10/28/09
Calibrated pixels/micron:	2.2617
Stage micrometer calibration expiration date:	2/13/09
Measured value for 600 µm in stage micrometer image (µm):	599.6

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
396	401	399	403

Approximate layer width in polish plane (µm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
248	85	42	36	37

Comments
The procedure was modified for this sample. 10x magnification was used rather than 12.5x.

Operator

October 28, 2008

Date

Data Report Form DRF-08-10x: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

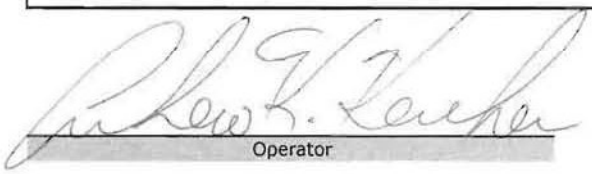
Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	ZrO2-500-AK2-S01
Sample description:	ORNL CRP-6 QC Benchmark TRISO
Mount ID number:	M08071503
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08102801\P0810280102\

DMR calibration expiration date:	10/28/09
Calibrated pixels/micron:	2.2617
Stage micrometer calibration expiration date:	2/13/09
Measured value for 600 µm in stage micrometer image (µm):	599.6

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
390	380	387	406

Approximate layer width in polish plane (µm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
242	87	42	36	38

Comments
The procedure was modified for this sample. 10x magnification was used rather than 12.5x.



Operator

October 28, 2008

Date

Data Report Form DRF-08-10x: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

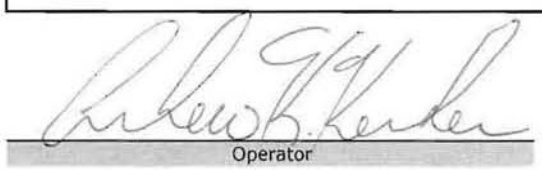
Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	ZrO2-500-AK2-S01
Sample description:	ORNL CRP-6 QC Benchmark TRISO
Mount ID number:	M08071504
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08102801\P0810280103\

DMR calibration expiration date:	10/28/09
Calibrated pixels/micron:	2.2617
Stage micrometer calibration expiration date:	2/13/09
Measured value for 600 μm in stage micrometer image (μm):	599.6

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
358	386	368	382

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
235	87	44	36	38

Comments
The procedure was modified for this sample. 10x magnification was used rather than 12.5x to accommodate larger particle diameter.



Operator

October 28, 2008

Date

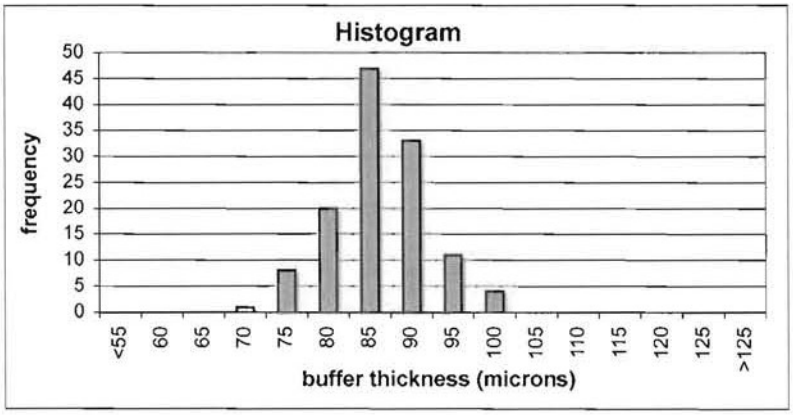
Data Report Form DRF-11A: Measurement of Buffer Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801\
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801_output\

Number of buffer layers analyzed:	124
Mean of the average buffer thickness of each particle (μm):	83.8
Standard deviation in the average buffer thickness of each particle (μm):	5.5

Distribution of the average buffer layer thickness (top binned)

Buffer Thickness (μm)	*Frequency*
<55	0
60	0
65	0
70	1
75	8
80	20
85	47
90	33
95	11
100	4
105	0
110	0
115	0
120	0
125	0
>125	0



Andrew K. Kercher
Operator

October 30, 2008
Date

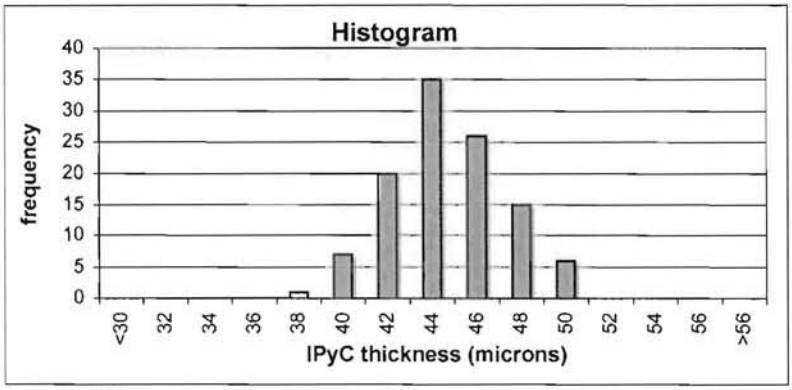
Data Report Form DRF-11B: Measurement of Inner Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801\
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801_output\

Number of inner pyrocarbon layers analyzed:	110
Mean of the average IPyC thickness of each particle (μm):	43.7
Standard deviation in the average IPyC thickness of each particle (μm):	2.5

Distribution of the average IPyC layer thickness (top binned)

IPyC Thickness (μm)	*Frequency*
<30	0
32	0
34	0
36	0
38	1
40	7
42	20
44	35
46	26
48	15
50	6
52	0
54	0
56	0
>56	0



Operator

October 30, 2008

Date

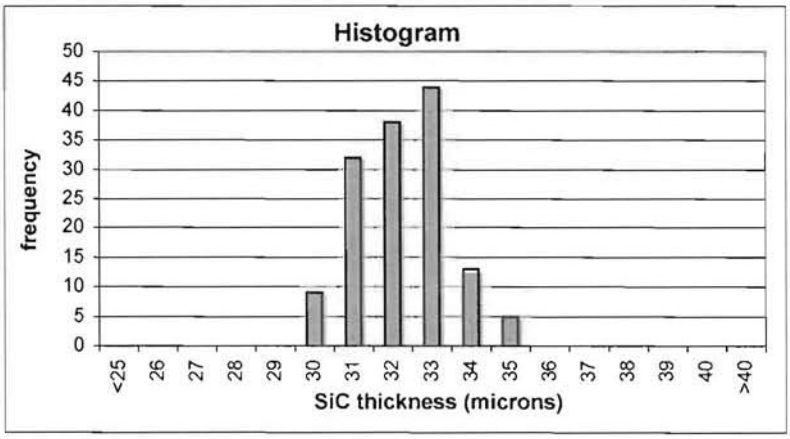
Data Report Form DRF-11C: Measurement of Silicon Carbide Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801\
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801_output\

Number of silicon carbide layers analyzed:	141
Mean of the average SiC thickness of each particle (μm):	31.7
Standard deviation in the average SiC thickness of each particle (μm):	1.1

Distribution of the average SiC layer thickness (top binned)

SiC Thickness (μm)	*Frequency*
<25	0
26	0
27	0
28	0
29	0
30	9
31	32
32	38
33	44
34	13
35	5
36	0
37	0
38	0
39	0
40	0
>40	0



Richard K. Kercher
Operator

October 30, 2008
Date

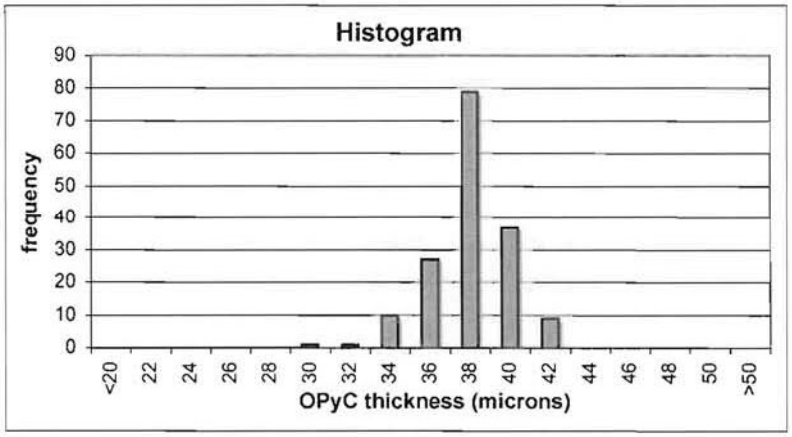
Data Report Form DRF-11D: Measurement of Outer Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801\
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08102801_output\

Number of outer pyrocarbon layers analyzed:	164
Mean of the average OPyC thickness of each particle (μm):	37.1
Standard deviation in the average OPyC thickness of each particle (μm):	1.9

Distribution of the average OPyC layer thickness (top binned)

OPyC Thickness (μm)	*Frequency*
<20	0
22	0
24	0
26	0
28	0
30	1
32	1
34	10
36	27
38	79
40	37
42	9
44	0
46	0
48	0
50	0
>50	0



Operator

October 30, 2008

Date

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08071601
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08072101\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0158	0.0036	0.0006	1.0321	0.0074	0.0012
2	4,5	0.0189	0.0039	0.0006	1.0385	0.0081	0.0012
3	4,6	0.0150	0.0034	0.0006	1.0305	0.0070	0.0012
4	5,4	0.0166	0.0039	0.0007	1.0338	0.0081	0.0014
5	5,5	0.0154	0.0039	0.0007	1.0313	0.0080	0.0014
6	5,6	0.0174	0.0041	0.0007	1.0354	0.0085	0.0015
7	6,4	0.0167	0.0041	0.0008	1.0340	0.0085	0.0017
8	6,5	0.0161	0.0037	0.0008	1.0327	0.0076	0.0017
9	6,6	0.0164	0.0041	0.0008	1.0333	0.0085	0.0017
10	5,7	0.0163	0.0036	0.0008	1.0331	0.0074	0.0017
Average		0.0165	0.0038	0.0007	1.0335	0.0079	0.0015

Mean of average BAFo per particle:	1.0335
Standard deviation of average BAFo per particle:	0.0023

Comments

G. E. Jellison — Operator

7/21/08 — Date

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08071601
Sample ID:	ZrO2-500-AK2-S01
Sample Description:	ORNL CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08072101\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0066	0.0035	0.0007	1.0133	0.0071	0.0014
2	4,5	0.0063	0.0034	0.0007	1.0127	0.0069	0.0014
3	4,6	0.0063	0.0034	0.0007	1.0127	0.0069	0.0014
4	5,4	0.0065	0.0033	0.0007	1.0131	0.0067	0.0014
5	5,5	0.0063	0.0031	0.0008	1.0127	0.0063	0.0016
6	5,6	0.0065	0.0034	0.0008	1.0131	0.0069	0.0016
7	6,4	0.0061	0.0031	0.0009	1.0123	0.0063	0.0018
8	6,5	0.0064	0.0034	0.0009	1.0129	0.0069	0.0018
9	6,6	0.0059	0.0031	0.0009	1.0119	0.0063	0.0018
10	5,7	0.0062	0.0031	0.0009	1.0125	0.0063	0.0018
Average		0.0063	0.0033	0.0008	1.0127	0.0066	0.0016

Mean of average BAFo per particle:	1.0127
Standard deviation of average BAFo per particle:	0.0004

Comments

G. E. Jellison — Operator

7/21/08 — Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	Zr02-500-AK2-S01
Particle Lot Description:	ORNL CRP-6 QC Benchmark TRISO
Filename:	\\mc-agr\AGR\ParticleWeight\W08061701_DRF22R1 xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1495	0.1520	0.1982	0.2278	0.1930
Number of particles:	134	137	177	205	175
Average weight/particle (g):	1.116E-03	1.109E-03	1.120E-03	1.111E-03	1.103E-03

Mean average weight/particle (g):	1.112E-03
Standard error in mean average weight/particle (g):	2.87E-06

Dixie Barker
Operator

6-19-08
Date

Data Report Form DRF-31: Measurement of Open Porosity using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-31 Rev. 1
Operator:	S. D. Nunn
Coated particle batch ID:	ZrO2-500-AK2-S01-I01
Batch Description:	ORNL CRP-6 QC Benchmark TRISO
Thermocouple Expiration Date:	5/15/09
Penetrometer Expiration Date:	7/12/08
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S08061901\S08061901_DRF31R1.xls

Mean average weight/particle (g):	1.11E-03
Standard error in mean average weight/particle (g):	2.87E-06

Weight of particles (g):	3.2794
Approximate number of particles:	2949
Uncertainty in number of particles:	8
Total envelope volume of sample (cc):	1.145
Average envelope volume/particle (cc):	3.88E-04
Sample envelope density (g/cc):	2.864

Average particle diameter (microns):	9.05E+02
Average surface area/particle (cm2):	2.57E-02
Total sample surface area (cm2):	7.59E+01
Intruded mercury volume from 250-10,000 psia (cc):	5.00E-04
Open porosity (ml/m2):	6.59E-02

Comments

S. D. Nunn
Operator

6/19/08
Date

A scanning electron microscope (SEM) was used to image particles polished close to midplane. The following 4 secondary electron (SE) images show the TRISO coatings and interfaces.

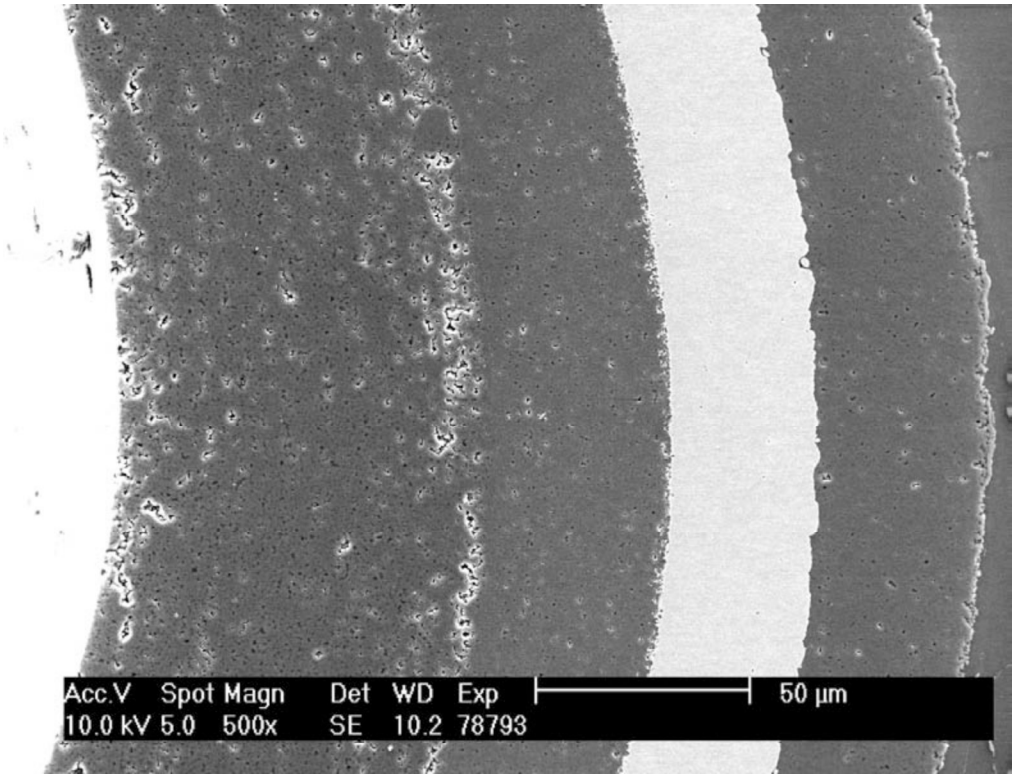


Figure 6. SE image of TRISO coatings on a typical particle.

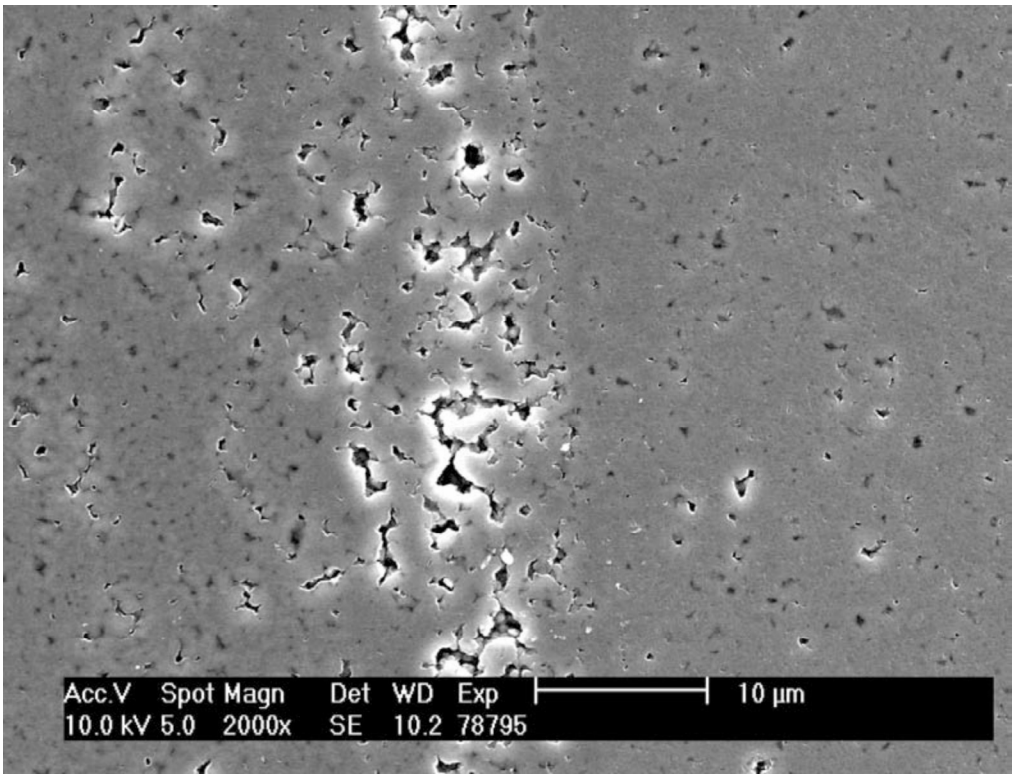


Figure 7. SE image of Buffer/IPyC interface.

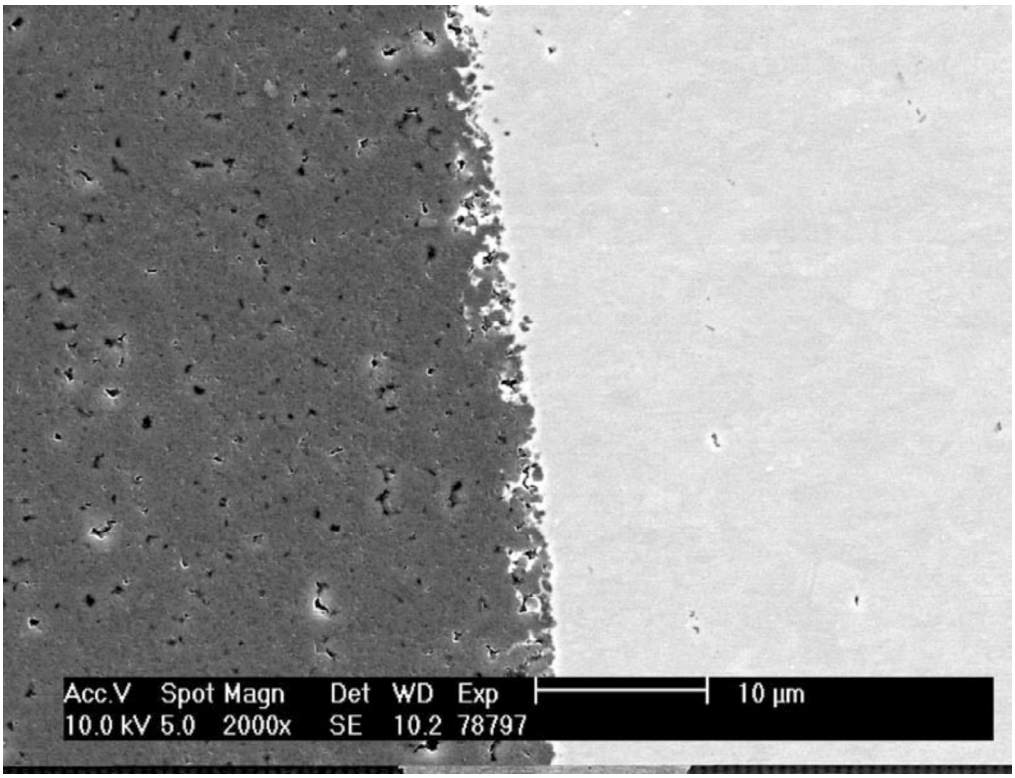


Figure 8. SE image of IPyC/SiC interface.

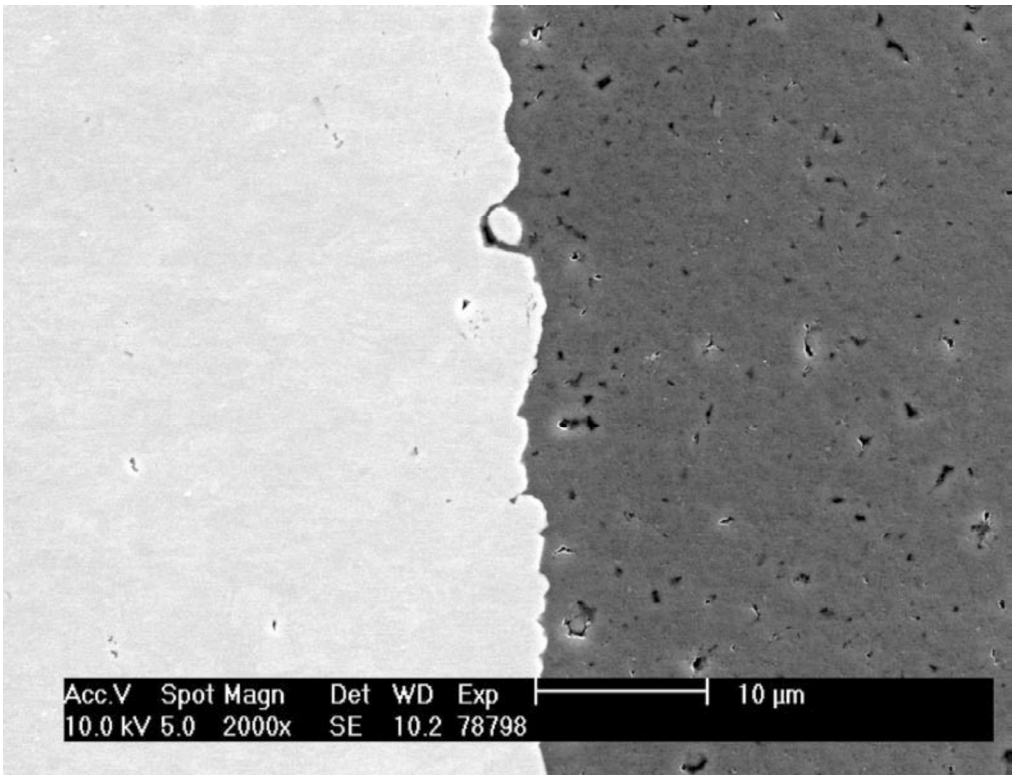


Figure 9. SE image of SiC/OPyC interface.

Backscattered electron imaging (BSE) was used to view the SiC grain structure. The following images show 3 different particles, each at two magnifications.

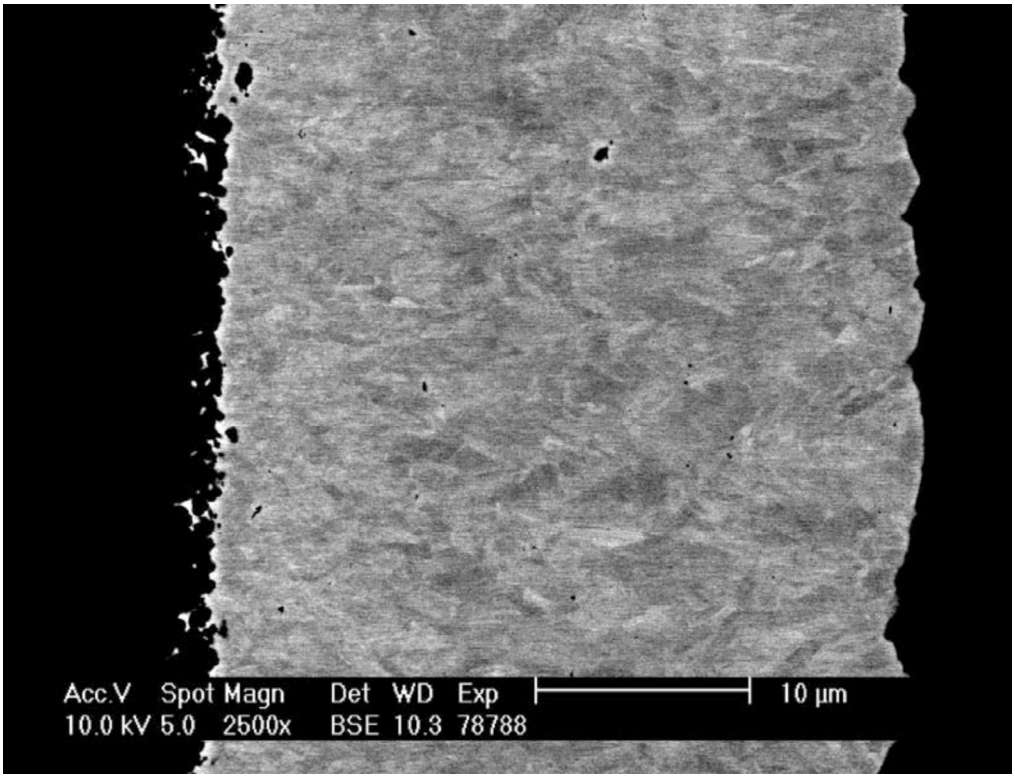


Figure 10. Particle 1 SiC grain structure shown by BSE.

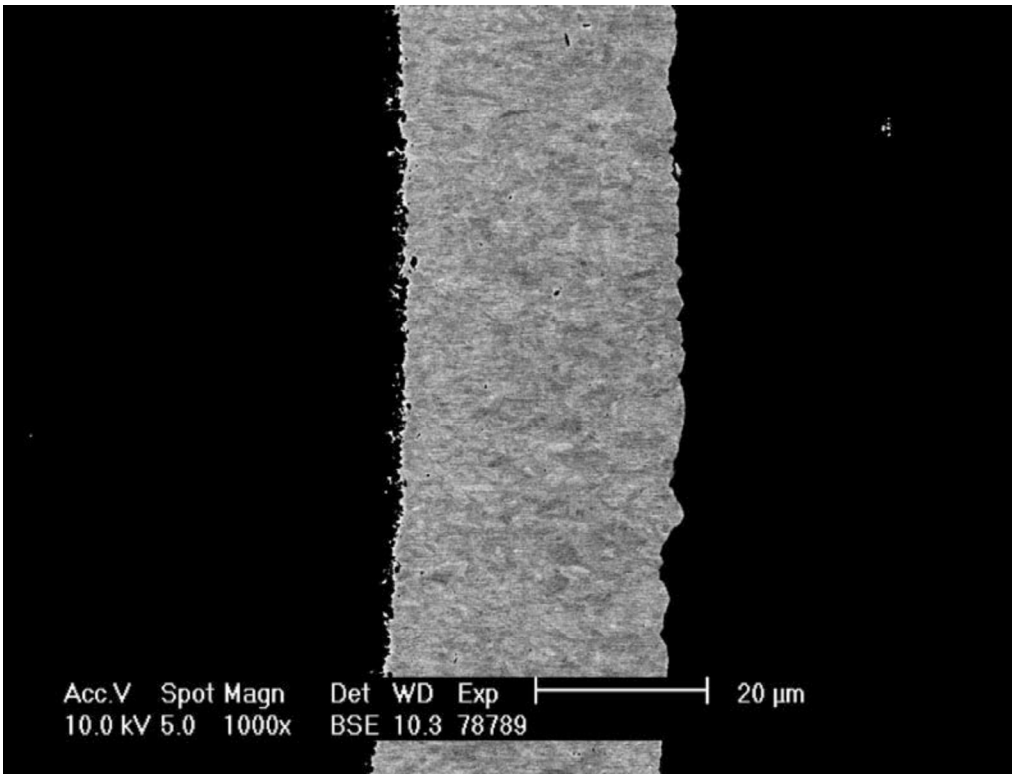


Figure 11. Particle 1 SiC grain structure shown by BSE.

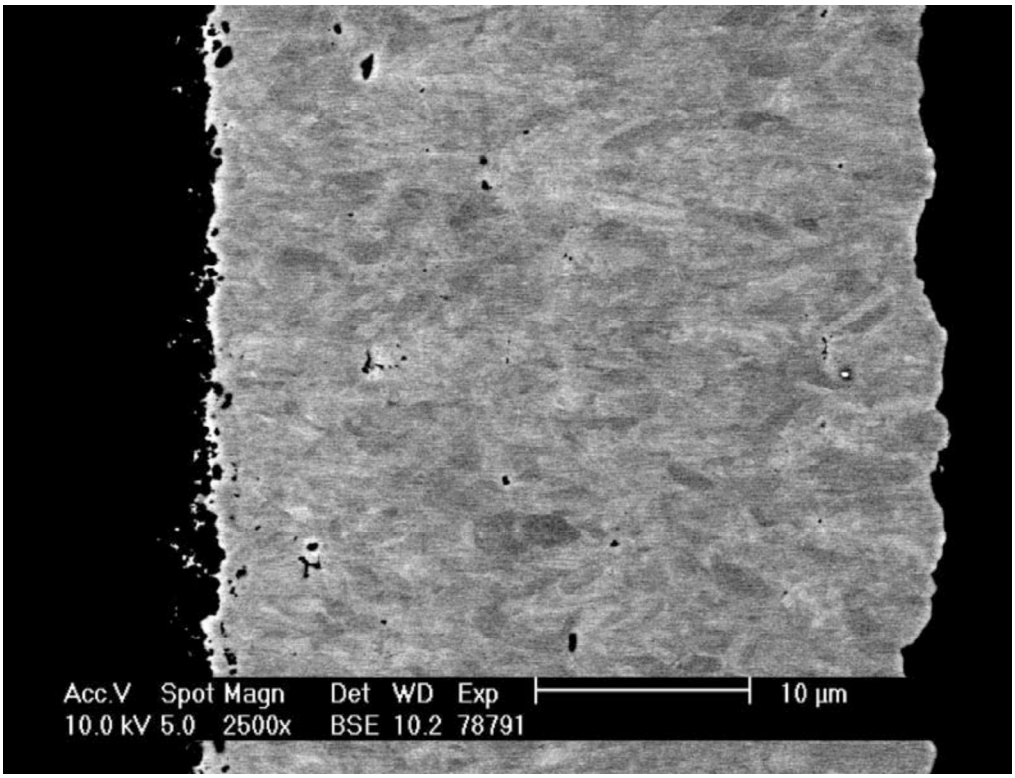


Figure 12. Particle 2 SiC grain structure shown by BSE.

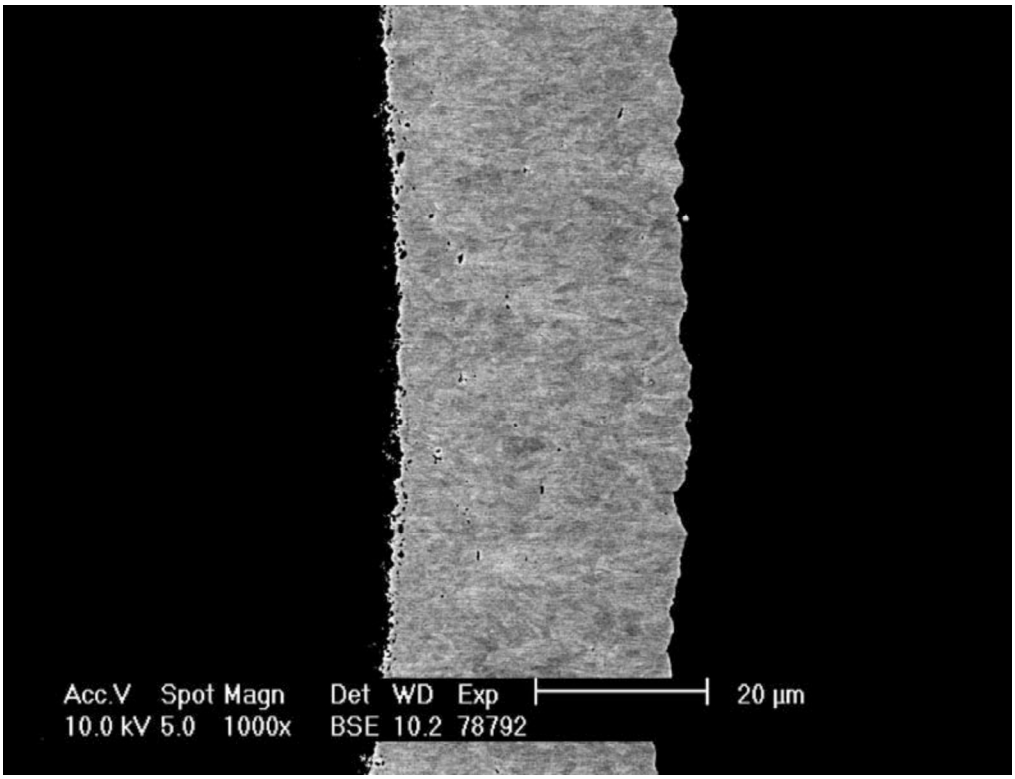


Figure 13. Particle 2 SiC grain structure shown by BSE.

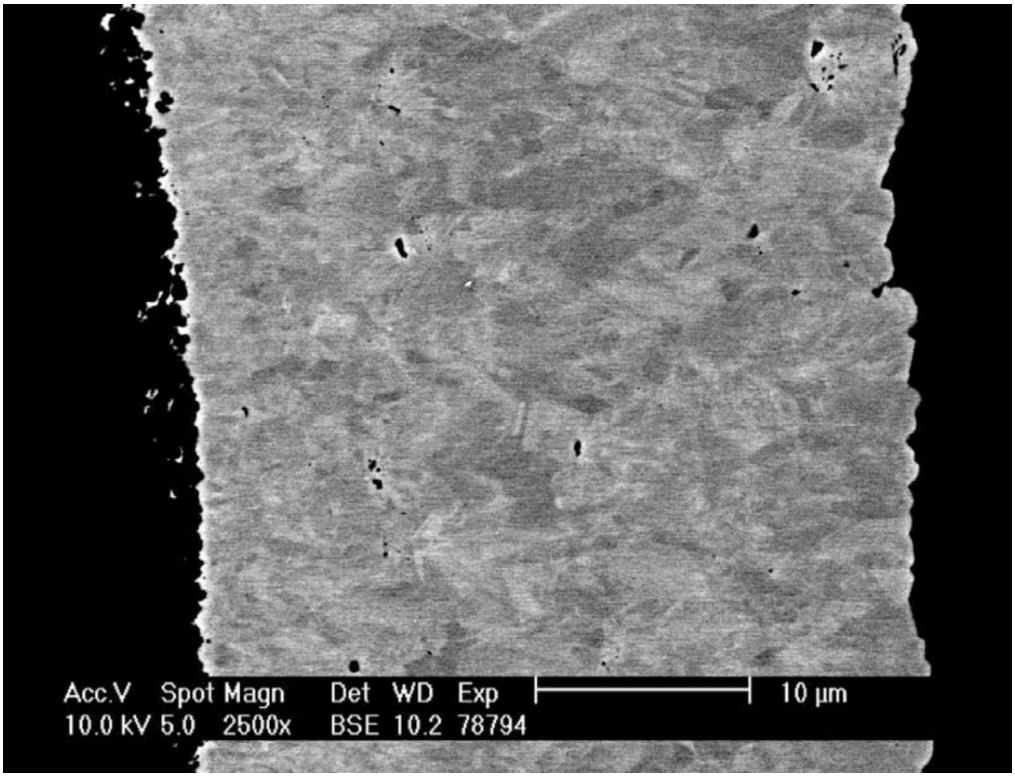


Figure 14. Particle 3 SiC grain structure shown by BSE.

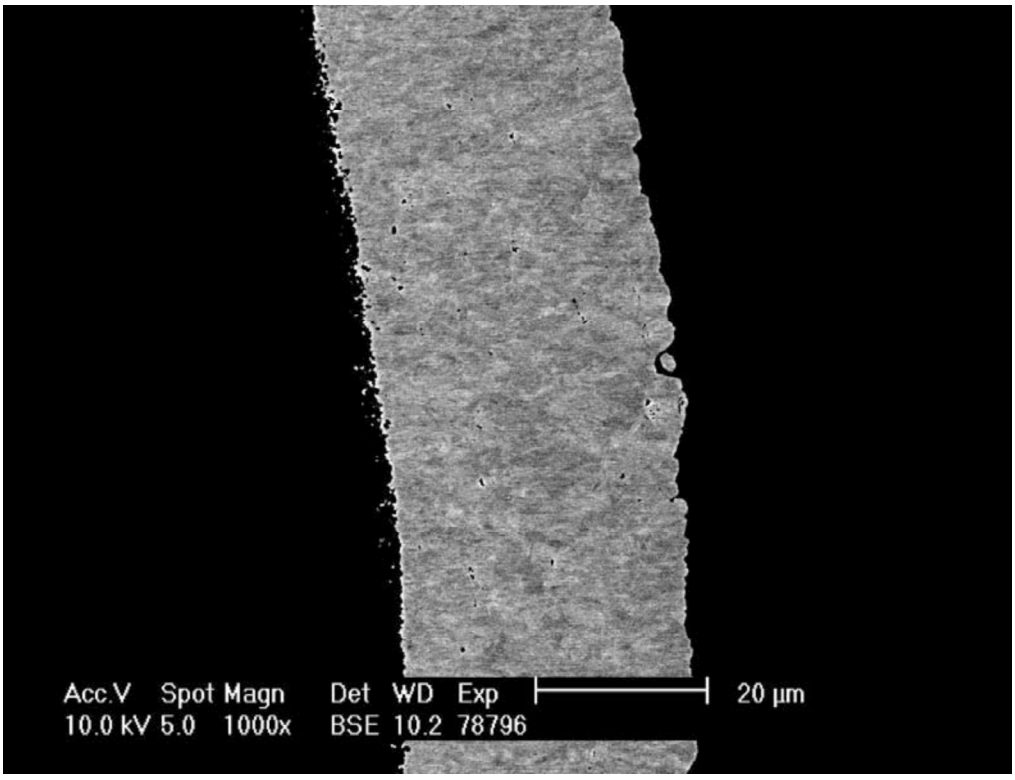


Figure 15. Particle 3 SiC grain structure shown by BSE.

One particle was selected at random and imaged using a high resolution x-ray tomograph.

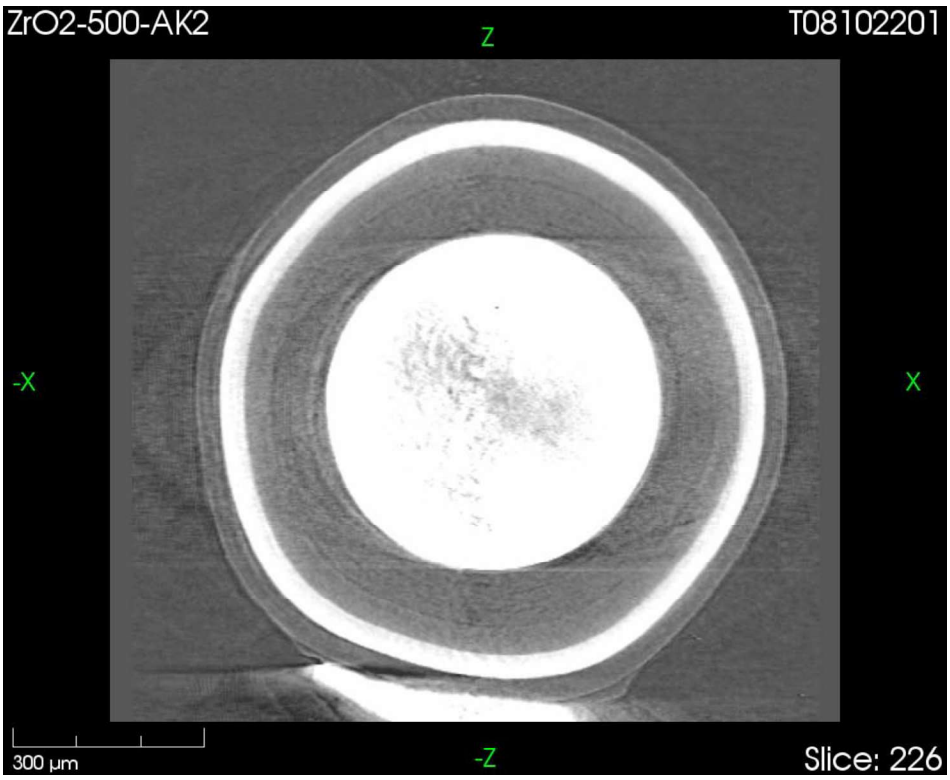


Figure 16. Tomographic cross-section of particle with rotation axis (z) in plane.

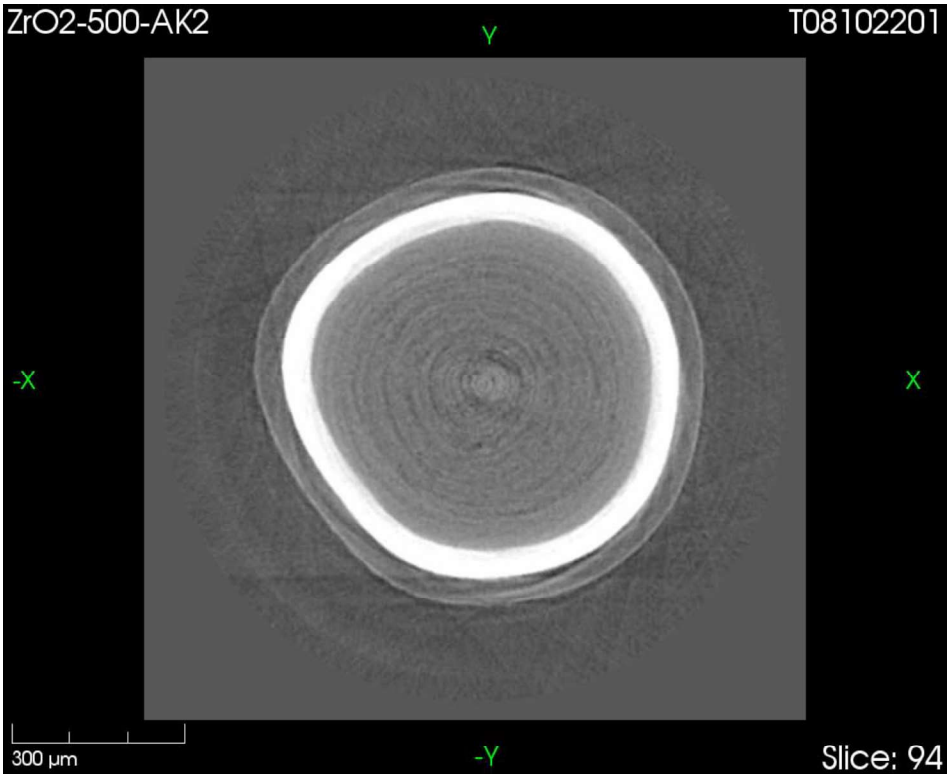


Figure 17. Tomographic cross-section perpendicular to rotation axis (z) showing a plane above kernel, where kernel effects on tomographic reconstruction are not present.

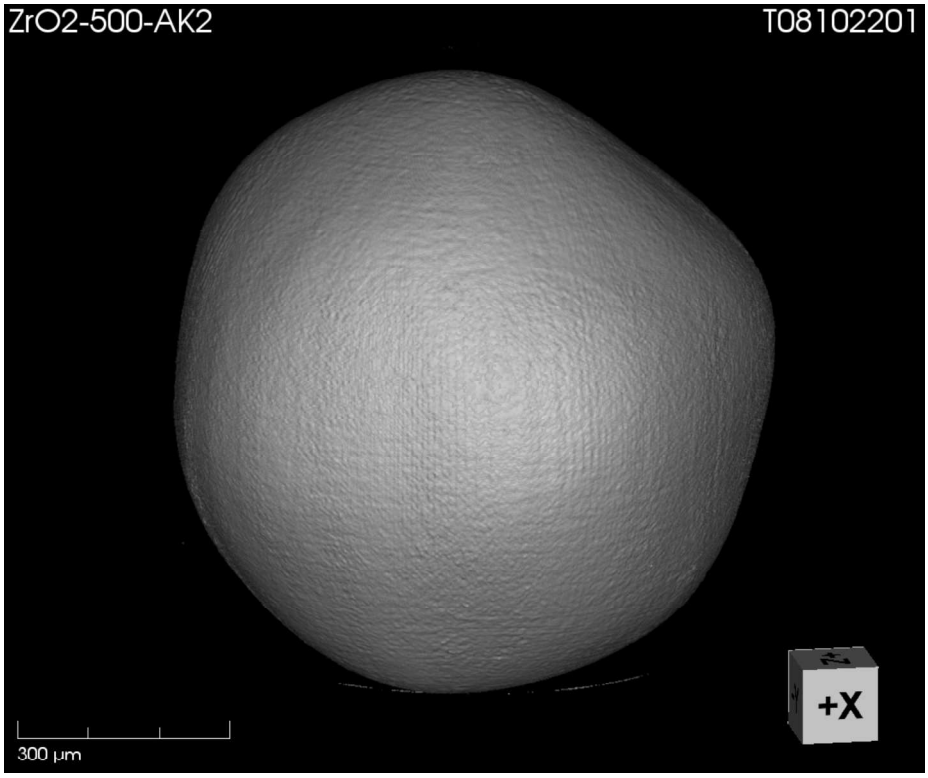


Figure 18. Tomographic 3-D visualization of SiC surface, oriented to show maximum faceting.

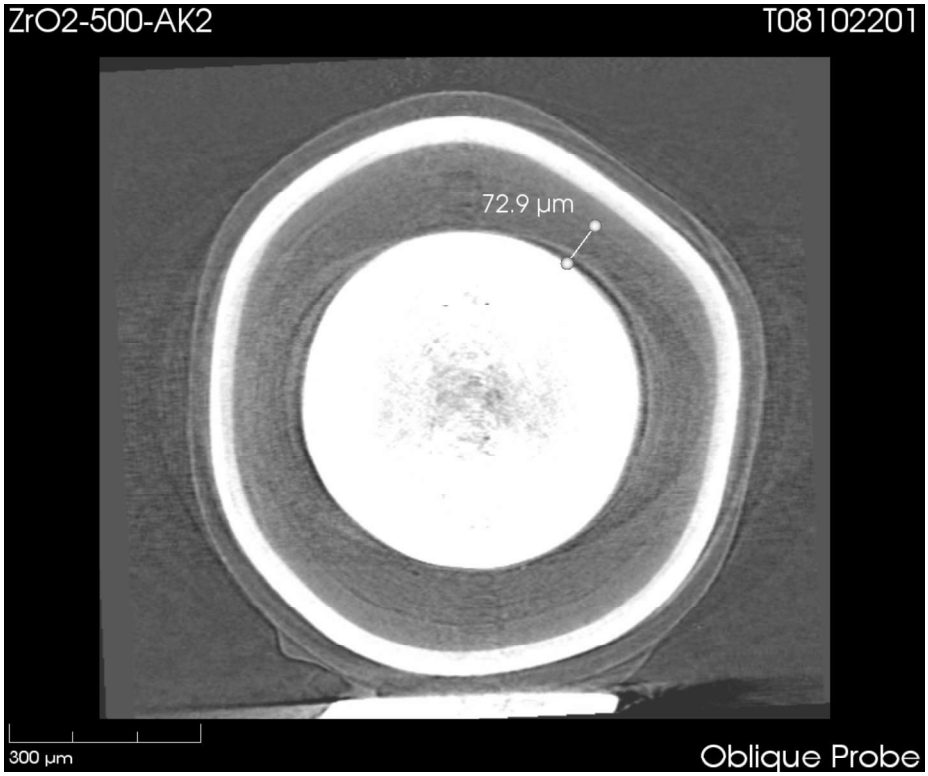


Figure 19. Tomographic cross-section rotated off axis to visualize facet caused by thin region in buffer.

QC of B&W TRISO Sample

This section contains the detailed data for the characterization measurements performed on B&W TRISO sample 93046A.

On the following page is an inspection report form that summarizes the results of each measurement. The mean and standard deviation for each measurement is listed in the table, where appropriate. Also provided on this form are the statistically calculated 95% confidence limits for the mean and dispersion of the parent batch of material.

A and B are the minimum and maximum predicted values for the mean of the parent batch up to the 95% confidence level.

$$A = \bar{x} - \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$$B = \bar{x} + \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$\bar{x}$ and σ_x are the mean and the standard deviation obtained from the measurement performed on a sample of n items. t_{n-1} is the one-tailed Student's-t value for n-1 degrees of freedom.

C is the minimum value for which all but 1% of the population is above (at 95% confidence). D is the maximum value for which all but 1% of the population is below (at 95% confidence).

$$C = \bar{x} - k_n\sigma_x$$

$$D = \bar{x} + k_n\sigma_x$$

k_n is the one-tailed tolerance factor for a sample of n items, given a 1% tolerance limit and a 95% confidence level.

Following the inspection report form are the individual data report forms for each measurement. These forms provide more detail of the measurement performed and include either data for each measured item or histograms of this data.

At the end of this section are images of a few particles obtained using scanning electron microscopy and x-ray tomography. These images show the typical microstructure of the coating layers and interfaces.

Inspection Report Form IRF-CRP6: QC Benchmark Measurements on Coated Particles

Coated particle composite ID:	93046A
Coated particle composite description:	B&W CRP-6 QC Benchmark TRISO

Property	Measured Data				95% Confidence Limits for Particle Lot			Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value				
SiC sink/float density (Mg/m³)	3.1971	0.0018	40	1.685	mean ≥ A	A = x - ts/√n	3.197	DRF-02
					mean ≤ B	B = x + ts/√n	3.198	
				2.941	99% of population > C	C = x - ks	3.192	
					99% of population < D	D = x + ks	3.202	
OPyC sink/float density (Mg/m³)	1.8885	0.0201	40	1.685	mean ≥ A	A = x - ts/√n	1.883	DRF-03
					mean ≤ B	B = x + ts/√n	1.894	
				2.941	99% of population > C	C = x - ks	1.829	
					99% of population < D	D = x + ks	1.948	
Particle Diameter (µm)	881.2	24.0	2083	1.646	mean ≥ A	A = x - ts/√n	880.3	DRF-07 DRF-10
					mean ≤ B	B = x + ts/√n	882.1	
				2.397	99% of population > C	C = x - ks	823.7	
					99% of population < D	D = x + ks	938.7	
Particle aspect ratio	1.039		2083		number of particles > 1.14		0	DRF-07 DRF-10
Average buffer thickness for each particle (µm)	61.9	6.9	187	1.653	mean ≥ A	A = x - ts/√n	61.1	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	62.7	
				2.577	99% of population > C	C = x - ks	44.1	
					99% of population < D	D = x + ks	79.7	
Average IPyC thickness for each particle (µm)	40.9	2.5	214	1.652	mean ≥ A	A = x - ts/√n	40.6	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	41.2	
				2.559	99% of population > C	C = x - ks	34.5	
					99% of population < D	D = x + ks	47.3	
Average SiC thickness for each particle (µm)	32.1	0.8	216	1.652	mean ≥ A	A = x - ts/√n	32.0	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	32.2	
				2.558	99% of population > C	C = x - ks	30.1	
					99% of population < D	D = x + ks	34.1	
Average OPyC thickness for each particle (µm)	38.4	2.5	216	1.652	mean ≥ A	A = x - ts/√n	38.1	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	38.7	
				2.558	99% of population > C	C = x - ks	32.0	
					99% of population < D	D = x + ks	44.8	
IPyC anisotropy (BAFo)	1.0205	0.0008	10	1.833	mean ≥ A	A = x - ts/√n	1.0200	DRF-18
					mean ≤ B	B = x + ts/√n	1.0210	
				3.981	99% of population > C	C = x - ks	1.0173	
					99% of population < D	D = x + ks	1.0237	
OPyC anisotropy (BAFo)	1.0168	0.0006	10	1.833	mean ≥ A	A = x - ts/√n	1.0165	DRF-18
					mean ≤ B	B = x + ts/√n	1.0171	
				3.981	99% of population > C	C = x - ks	1.0144	
					99% of population < D	D = x + ks	1.0192	
Average particle weight (mg)	1.0270	0.0029	5 measurements	2.132	mean ≥ A	A = x - ts/√n	1.0243	DRF-22
					mean ≤ B	B = x + ts/√n	1.0297	
			644 particles	5.741	99% of population > C	C = x - ks	1.0106	
					99% of population < D	D = x + ks	1.0434	
OPyC open porosity (ml/m²)	0.3690		1 measurement					DRF-31
			3025 particles					

Comments

Sample was analyzed by SEM for SiC grain structure and general microstructure of layers and interfaces.
A particle was imaged using high resolution x-ray tomography.

John Hunn
QC Supervisor

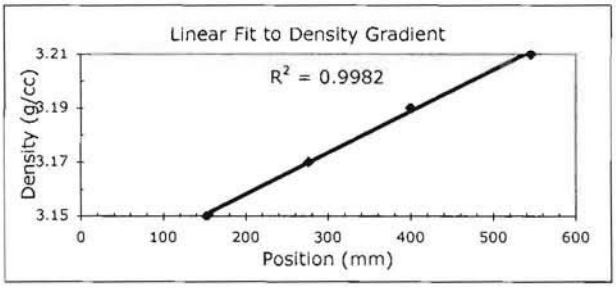
10-30-08
Date

Data Report Form DRF-02: Measurement of SiC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-02 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08021901_DRF02R3.xls
Sample ID:	93046A-E01
Sample description:	B&W CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.4 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
3.150	138.98	159.57	152.71
3.170	260.15	283.86	275.96
3.190	384.20	406.94	399.36
3.210	529.69	553.37	545.48

Linear Fit			
slope	StDev	intercept	StDev
1.54E-04	2.66E-06	3.13E+00	9.13E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	421.46	3.1920	26	464.48	3.1986	51		
2	437.48	3.1945	27	466.10	3.1988	52		
3	430.51	3.1934	28	466.94	3.1990	53		
4	436.06	3.1942	29	468.69	3.1992	54		
5	439.65	3.1948	30	470.38	3.1995	55		
6	442.55	3.1952	31	470.99	3.1996	56		
7	442.42	3.1952	32	474.73	3.2002	57		
8	443.10	3.1953	33	471.79	3.1997	58		
9	445.90	3.1957	34	467.79	3.1991	59		
10	447.15	3.1959	35	465.94	3.1988	60		
11	447.98	3.1961	36	458.61	3.1977	61		
12	448.76	3.1962	37	459.11	3.1978	62		
13	449.30	3.1963	38	456.32	3.1973	63		
14	450.49	3.1965	39	455.59	3.1972	64		
15	451.70	3.1966	40	455.90	3.1973	65		
16	451.70	3.1966	41			66		
17	453.22	3.1969	42			67		
18	455.45	3.1972	43			68		
19	456.10	3.1973	44			69		
20	456.25	3.1973	45			70		
21	458.57	3.1977	46			71		
22	460.06	3.1979	47			72		
23	460.32	3.1980	48			73		
24	461.80	3.1982	49			74		
25	461.34	3.1981	50			75		

Average density of SiC fragments:	3.1971
Standard deviation in density of SiC fragments:	0.0018
Uncertainty in calculated density of SiC fragments:	0.0016

Dixie Barker
Operator

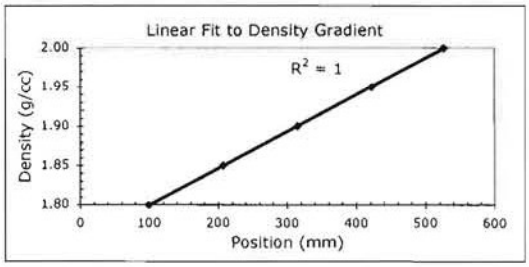
2-15-08
Date

Data Report Form DRF-03: Measurement of PyC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08021701_DRF03R3.xls
Sample ID:	93046A
Sample description:	B&W CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.5 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.800	94.55	104.43	99.49
1.850	203.11	211.57	207.34
1.900	312.20	318.09	315.15
1.950	418.34	425.40	421.87
2.000	523.01	529.48	526.25

Linear Fit			
slope	StDev	intercept	StDev
4.68E-04	2.74E-06	1.75E+00	8.66E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	276.58	1.8825	26	300.28	1.8936	51		
2	278.18	1.8832	27	300.94	1.8939	52		
3	281.14	1.8846	28	301.71	1.8942	53		
4	285.13	1.8865	29	302.70	1.8947	54		
5	286.50	1.8871	30	302.70	1.8947	55		
6	286.50	1.8871	31	302.88	1.8948	56		
7	288.60	1.8881	32	303.40	1.8950	57		
8	29.76	1.7669	33	303.82	1.8952	58		
9	289.76	1.8886	34	304.39	1.8955	59		
10	291.96	1.8897	35	305.11	1.8958	60		
11	291.86	1.8896	36	306.96	1.8967	61		
12	287.41	1.8875	37	308.99	1.8976	62		
13	288.76	1.8882	38	308.99	1.8976	63		
14	289.75	1.8886	39	310.32	1.8983	64		
15	290.95	1.8892	40	310.65	1.8984	65		
16	291.72	1.8896	41			66		
17	293.76	1.8905	42			67		
18	294.27	1.8908	43			68		
19	295.45	1.8913	44			69		
20	295.89	1.8915	45			70		
21	295.89	1.8915	46			71		
22	297.82	1.8924	47			72		
23	298.52	1.8927	48			73		
24	299.06	1.8930	49			74		
25	299.65	1.8933	50			75		

Average density of PyC fragments:	1.8885
Standard deviation in density of PyC fragments:	0.0201
Uncertainty in calculated density of PyC fragments:	0.0012

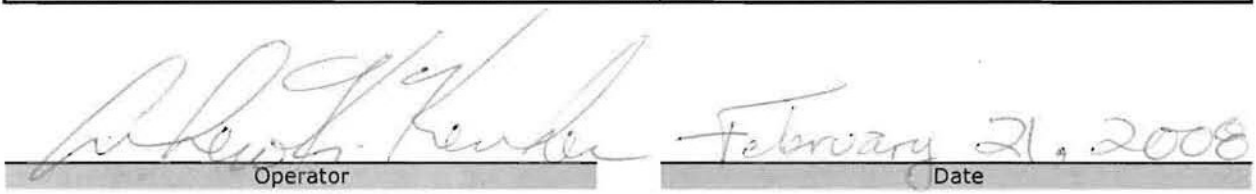
Dixie Barker
Operator

2-17-08
Date

Data Report Form DRF-07: Imaging of Particle Diameter and Aspect Ratio Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-07 Rev. 1
Operator:	Andrew K. Kercher
Sample ID:	93046A-C01
Sample Description:	BWXT surrogate ZrO2 TRISO G73-NF-93046A
Folder name containing images:	\\Mc-agr\agr\ImageProcessing\P08022101\

DMR Calibration Expiration Date:	9/20/2008
Stage Micrometer Calibration Expiration Date:	2/13/2009
Measured Value for 1200 µm in Stage Micrometer Image:	1200.9 µm



Operator | Date

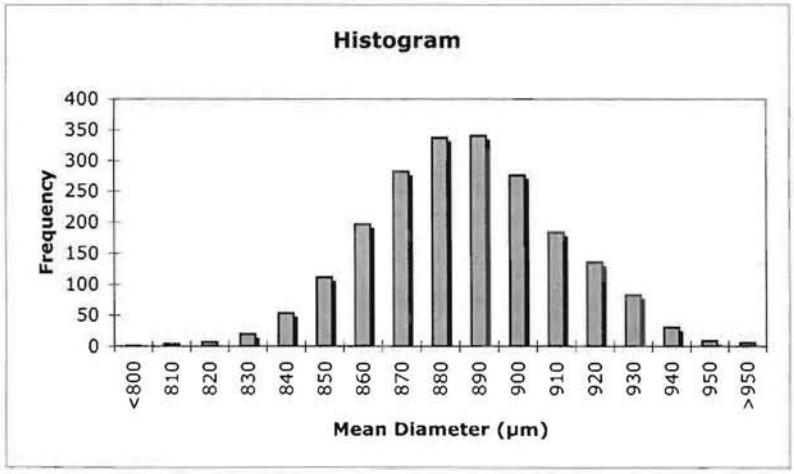
Data Report Form DRF-10A: Measurement of Particle Diameter

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08022101\
Sample ID:	93046A-C01
Sample Description:	BWXT Surrogate ZrO2 TRISO G73-NF-93046A
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08022101_output\

Number of particles analyzed:	2083
Mean of the average diameter of each particle (μm):	881.2
Standard deviation in the average diameter of each particle (μm):	24

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<800	1
810	4
820	7
830	20
840	54
850	112
860	197
870	283
880	338
890	341
900	277
910	184
920	136
930	83
940	31
950	9
>950	6



Andrew K. Kercher — Operator

February 26, 2008 — Date

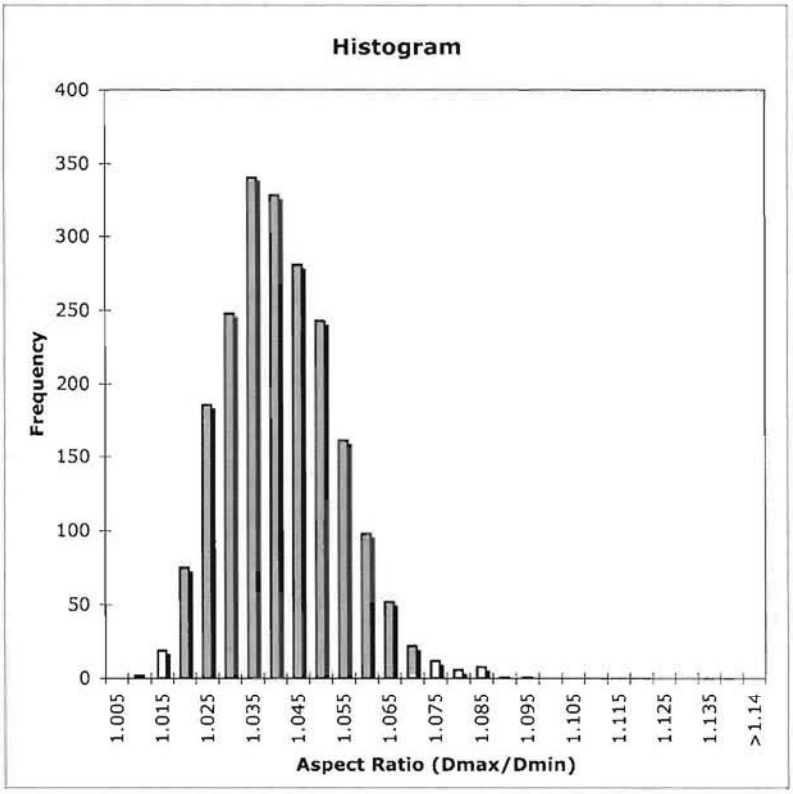
Data Report Form DRF-10B: Measurement of Particle Aspect Ratio (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08022101\
Sample ID:	93046A-C01
Sample Description:	BWXT Surrogate ZrO2 TRISO G73-NF-93046A
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08022101_output\

Number of particles analyzed:	2083
Number of particles with aspect ratio ≥ 1.14	0
Average particle aspect ratio:	1.039

Distribution of the aspect ratio (top binned)

Aspect Ratio (D)	*Frequency*
1.005	0
1.010	2
1.015	19
1.020	75
1.025	186
1.030	248
1.035	340
1.040	328
1.045	281
1.050	243
1.055	161
1.060	98
1.065	52
1.070	22
1.075	12
1.080	6
1.085	8
1.090	1
1.095	1
1.100	0
1.105	0
1.110	0
1.115	0
1.120	0
1.125	0
1.130	0
1.135	0
1.140	0
>1.14	0



Operator

February 26, 2008

Date

Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	93046A-B01
Sample description:	B&W CRP-6 QC Benchmark Sample
Mount ID number:	M08021801
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08031101\P0803110101\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 μm in stage micrometer image (μm):	500.0

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
399	395	409	412

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
257	70	41	33	42

Operator

March 11, 2008

Date

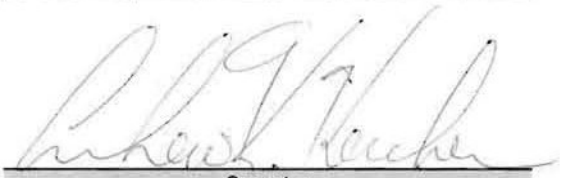
Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	93046A-B01
Sample description:	B&W CRP-6 QC Benchmark Sample
Mount ID number:	M08021802
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08031101\P0803110102\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 µm in stage micrometer image (µm):	500.0

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
386	381	385	383

Approximate layer width in polish plane (µm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
261	69	39	34	39



Operator

March 11, 2008

Date

Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	93046A-B01
Sample description:	B&W CRP-6 QC Benchmark Sample
Mount ID number:	M08021803
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08031101\P0803110103\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 μm in stage micrometer image (μm):	500.0

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
402	406	404	407

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
259	66	43	33	39

Operator

March 11, 2008

Date

Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	93046A-B01
Sample description:	B&W CRP-6 QC Benchmark Sample
Mount ID number:	M08031901
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08032701\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 µm in stage micrometer image (µm):	500.0

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
412	411	403	397

Approximate layer width in polish plane (µm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
262	58	42	33	38

Operator

March 27, 2008

Date

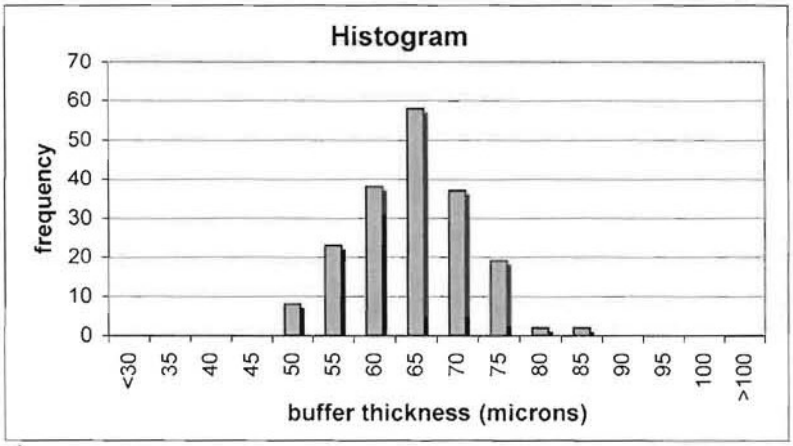
Data Report Form DRF-11A: Measurement of Buffer Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101\
Sample ID:	93046A-B01
Sample Description:	B&W CRP-6 QC Benchmark Sample
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101_output\

Number of buffer layers analyzed:	187
Mean of the average buffer thickness of each particle (μm):	61.9
Standard deviation in the average buffer thickness of each particle (μm):	6.9

Distribution of the average buffer layer thickness (top binned)

Buffer Thickness (μm)	*Frequency*
<30	0
35	0
40	0
45	0
50	8
55	23
60	38
65	58
70	37
75	19
80	2
85	2
90	0
95	0
100	0
>100	0



Andrew K. Kercher — April 8, 2008

Operator — Date

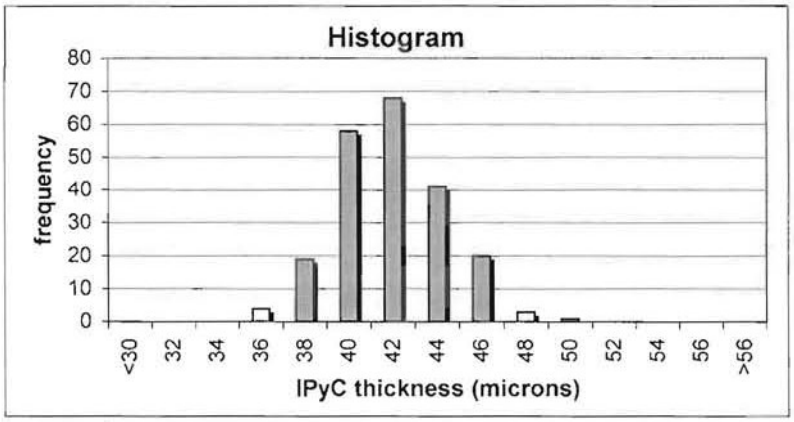
Data Report Form DRF-11B: Measurement of Inner Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101\
Sample ID:	93046A-B01
Sample Description:	B&W CRP-6 QC Benchmark Sample
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101_output\

Number of inner pyrocarbon layers analyzed:	214
Mean of the average IPyC thickness of each particle (μm):	40.9
Standard deviation in the average IPyC thickness of each particle (μm):	2.5

Distribution of the average IPyC layer thickness (top binned)

IPyC Thickness (μm)	*Frequency*
<30	0
32	0
34	0
36	4
38	19
40	58
42	68
44	41
46	20
48	3
50	1
52	0
54	0
56	0
>56	0



Andrew K. Kercher
Operator

April 8, 2008
Date

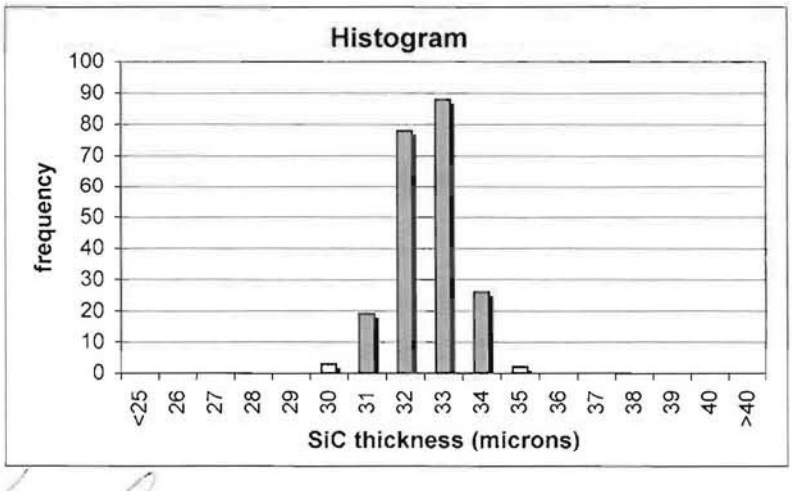
Data Report Form DRF-11C: Measurement of Silicon Carbide Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101\
Sample ID:	93046A-B01
Sample Description:	B&W CRP-6 QC Benchmark Sample
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101_output\

Number of silicon carbide layers analyzed:	216
Mean of the average SiC thickness of each particle (μm):	32.1
Standard deviation in the average SiC thickness of each particle (μm):	0.8

Distribution of the average SiC layer thickness (top binned)

SiC Thickness (μm)	*Frequency*
<25	0
26	0
27	0
28	0
29	0
30	3
31	19
32	78
33	88
34	26
35	2
36	0
37	0
38	0
39	0
40	0
>40	0



Andrew K. Kercher
Operator

April 8, 2008
Date

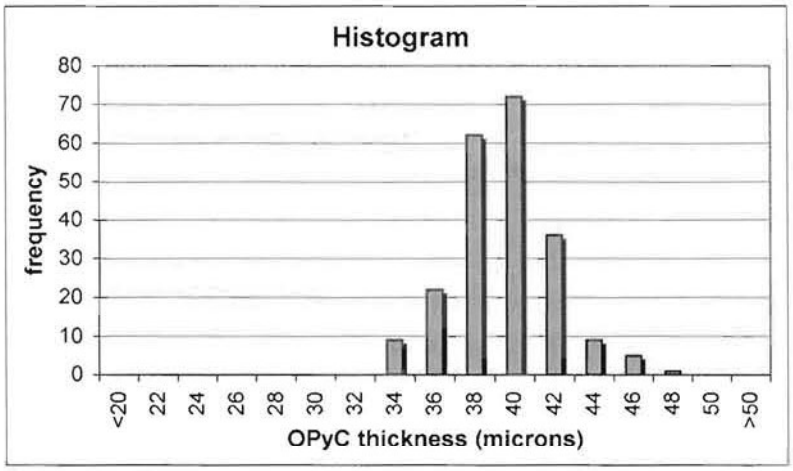
Data Report Form DRF-11D: Measurement of Outer Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101\
Sample ID:	93046A-B01
Sample Description:	B&W CRP-6 QC Benchmark Sample
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08031101_output\

Number of outer pyrocarbon layers analyzed:	216
Mean of the average OPyC thickness of each particle (μm):	38.4
Standard deviation in the average OPyC thickness of each particle (μm):	2.5

Distribution of the average OPyC layer thickness (top binned)

OPyC Thickness (μm)	*Frequency*
<20	0
22	0
24	0
26	0
28	0
30	0
32	0
34	9
36	22
38	62
40	72
42	36
44	9
46	5
48	1
50	0
>50	0



Operator

April 8, 2008

Date

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M07121802
Sample ID:	93046A
Sample Description:	B&W CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08010901\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	3,3	0.0103	0.0027	0.0009	1.0208	0.0055	0.0018
2	3,4	0.0099	0.0023	0.0009	1.0200	0.0047	0.0018
3	3,5	0.0102	0.0022	0.0009	1.0206	0.0045	0.0018
4	4,3	0.0107	0.0024	0.0009	1.0216	0.0049	0.0018
5	4,4	0.0101	0.0024	0.0009	1.0204	0.0049	0.0018
6	4,5	0.0107	0.0022	0.0009	1.0216	0.0045	0.0018
7	5,3	0.0100	0.0026	0.0010	1.0202	0.0053	0.0020
8	5,4	0.0099	0.0024	0.0009	1.0200	0.0049	0.0018
9	5,5	0.0104	0.0024	0.0010	1.0210	0.0049	0.0020
10	4,6	0.0094	0.0025	0.0010	1.0190	0.0051	0.0020
Average		0.0102	0.0024	0.0009	1.0205	0.0049	0.0019

Mean of average BAFo per particle:	1.0205
Standard deviation of average BAFo per particle:	0.0008

Comments

A. E. Jellison
Operator

1/09/08
Date

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M07121802
Sample ID:	93046A
Sample Description:	B&W CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08010901\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	3,3	0.0083	0.0027	0.0009	1.0167	0.0055	0.0018
2	3,4	0.0086	0.0023	0.0009	1.0173	0.0047	0.0018
3	3,5	0.0085	0.0025	0.0009	1.0171	0.0051	0.0018
4	4,3	0.0078	0.0024	0.0010	1.0157	0.0049	0.0020
5	4,4	0.0081	0.0022	0.0009	1.0163	0.0045	0.0018
6	4,5	0.0081	0.0021	0.0009	1.0163	0.0043	0.0018
7	5,3	0.0083	0.0024	0.0009	1.0167	0.0049	0.0018
8	5,4	0.0083	0.0024	0.0009	1.0167	0.0049	0.0018
9	5,5	0.0087	0.0027	0.0010	1.0176	0.0055	0.0020
10	4,6	0.0086	0.0024	0.0010	1.0173	0.0049	0.0020
Average		0.0083	0.0024	0.0009	1.0168	0.0049	0.0019

Mean of average BAFo per particle:	1.0168
Standard deviation of average BAFo per particle:	0.0006

Comments

G. E. Jellison
Operator

1/09/08
Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	93046A-H01
Particle Lot Description:	B&W CRP-6 QC Benchmark TRISO
Filename:	\\mc-agr\AGR\ParticleWeight\W08021501_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1487	0.1215	0.1455	0.1214	0.1244
Number of particles:	144	118	141	119	122
Average weight/particle (g):	1.033E-03	1.030E-03	1.032E-03	1.020E-03	1.020E-03

Mean average weight/particle (g):	1.027E-03
Standard error in mean average weight/particle (g):	2.86E-06

Dixie Barker
Operator

2-15-08
Date

Data Report Form DRF-31: Measurement of Open Porosity using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-31 Rev. 1
Operator:	S. D. Nunn
Coated particle batch ID:	93046A-I01
Batch Description:	B&W CRP-6 QC Benchmark TRISO
Thermocouple Expiration Date:	5/29/08
Penetrometer Expiration Date:	7/12/08
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S08021501\S08021501_DRF31R1.xls

Mean average weight/particle (g):	1.03E-03
Standard error in mean average weight/particle (g):	2.86E-06

Weight of particles (g):	3.1067
Approximate number of particles:	3025
Uncertainty in number of particles:	8
Total envelope volume of sample (cc):	1.069
Average envelope volume/particle (cc):	3.53E-04
Sample envelope density (g/cc):	2.907

Average particle diameter (microns):	8.77E+02
Average surface area/particle (cm2):	2.42E-02
Total sample surface area (cm2):	7.31E+01
Intruded mercury volume from 250-10,000 psia (cc):	2.70E-03
Open porosity (ml/m2):	3.69E-01

Comments

S. D. Nunn
Operator

2/15/08
Date

A scanning electron microscope (SEM) was used to image particles polished close to midplane. The following 4 secondary electron (SE) images show the TRISO coatings and interfaces.

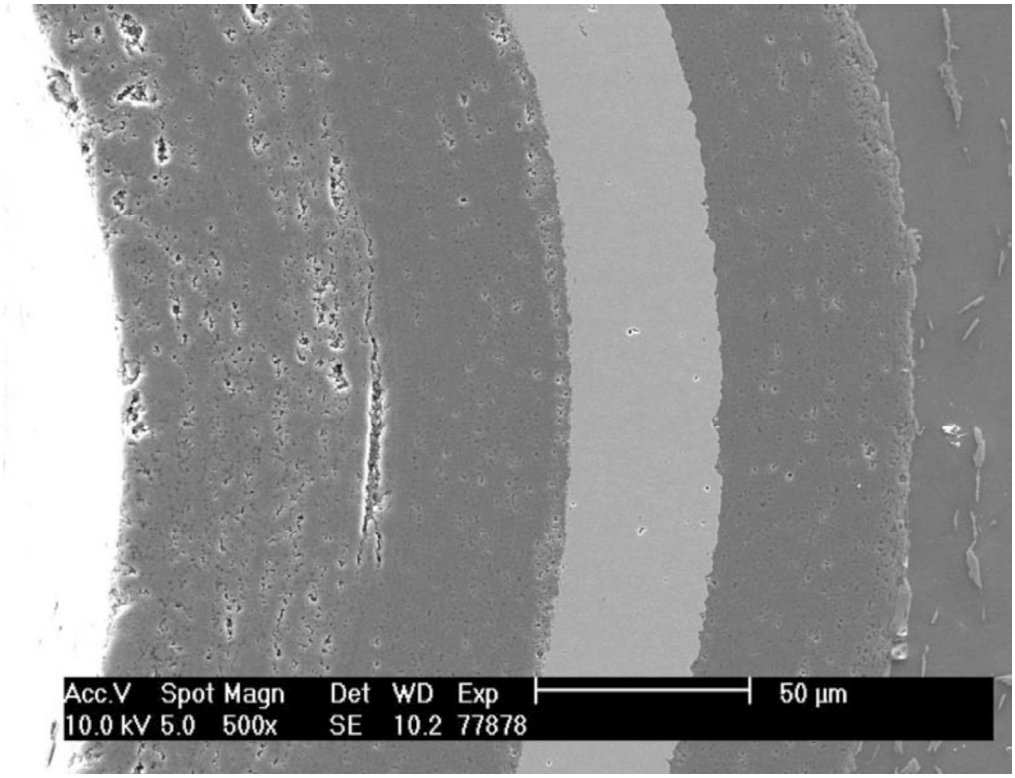


Figure 20. SE image of TRISO coatings on a typical particle.

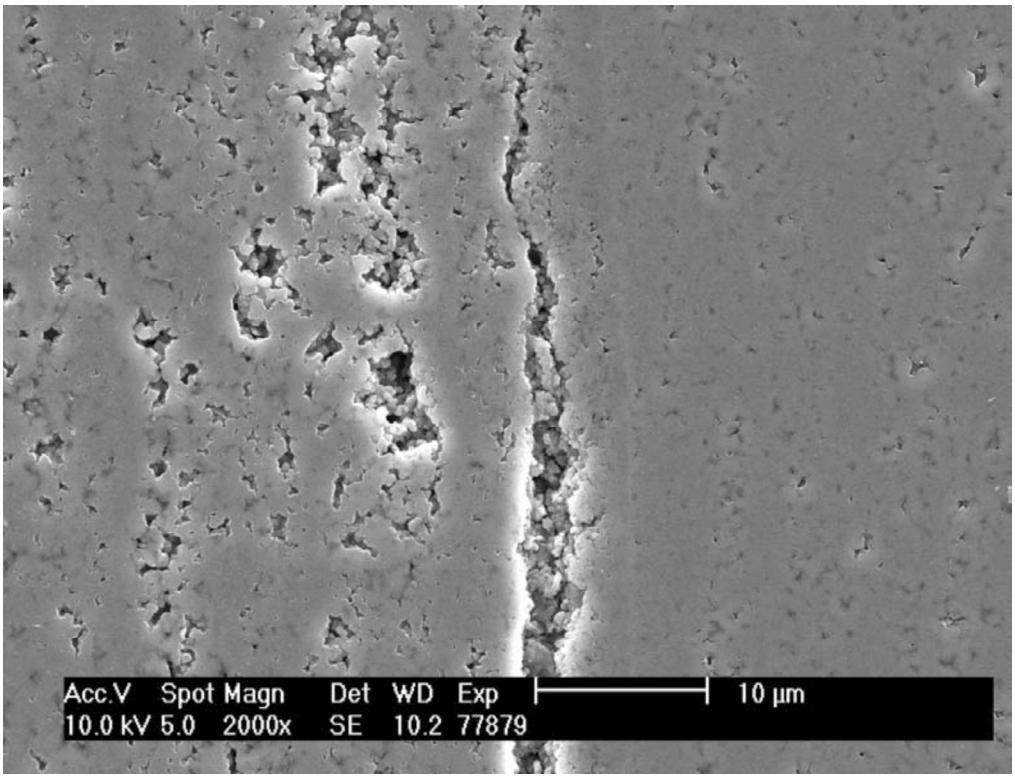


Figure 21. SE image of Buffer/IPyC interface.

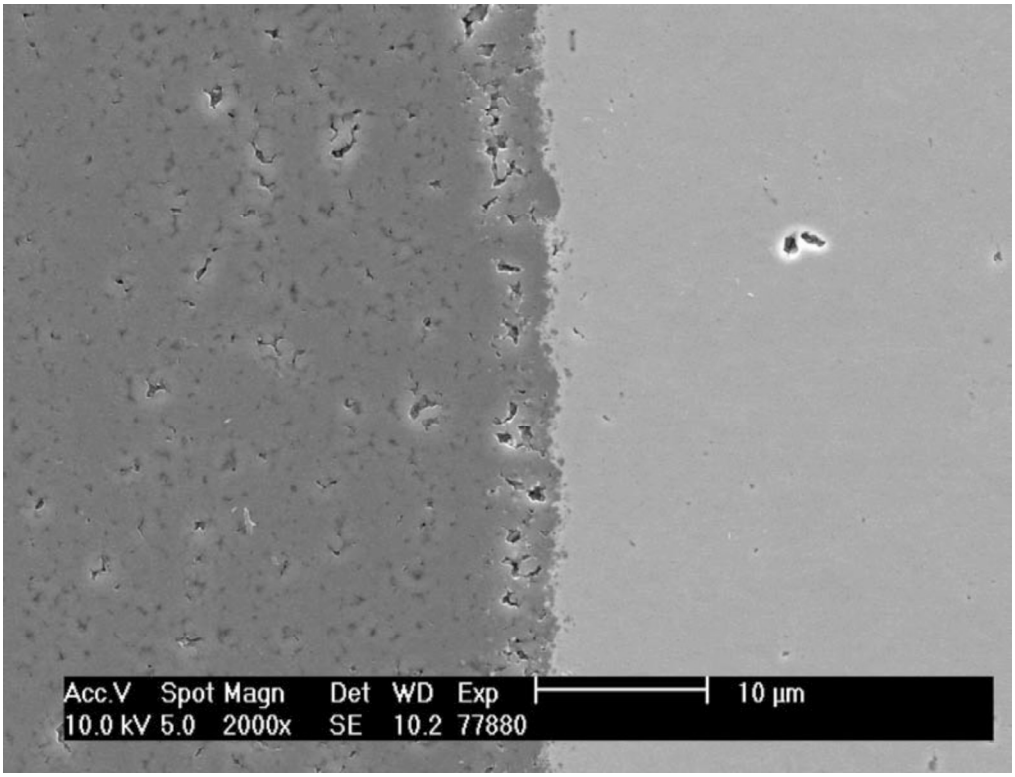


Figure 22. SE image of IPyC/SiC interface.

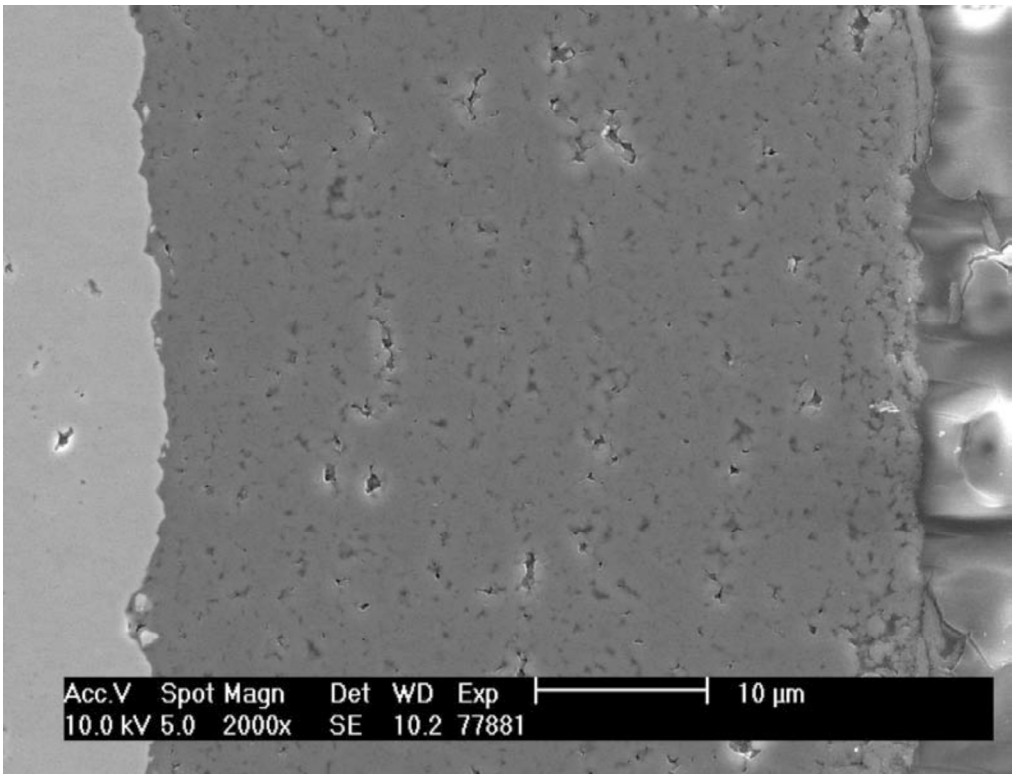


Figure 23. SE image of SiC/OPyC interface.

Backscattered electron imaging (BSE) was used to view the SiC grain structure. The following images show 2 different particles, each at two magnifications.

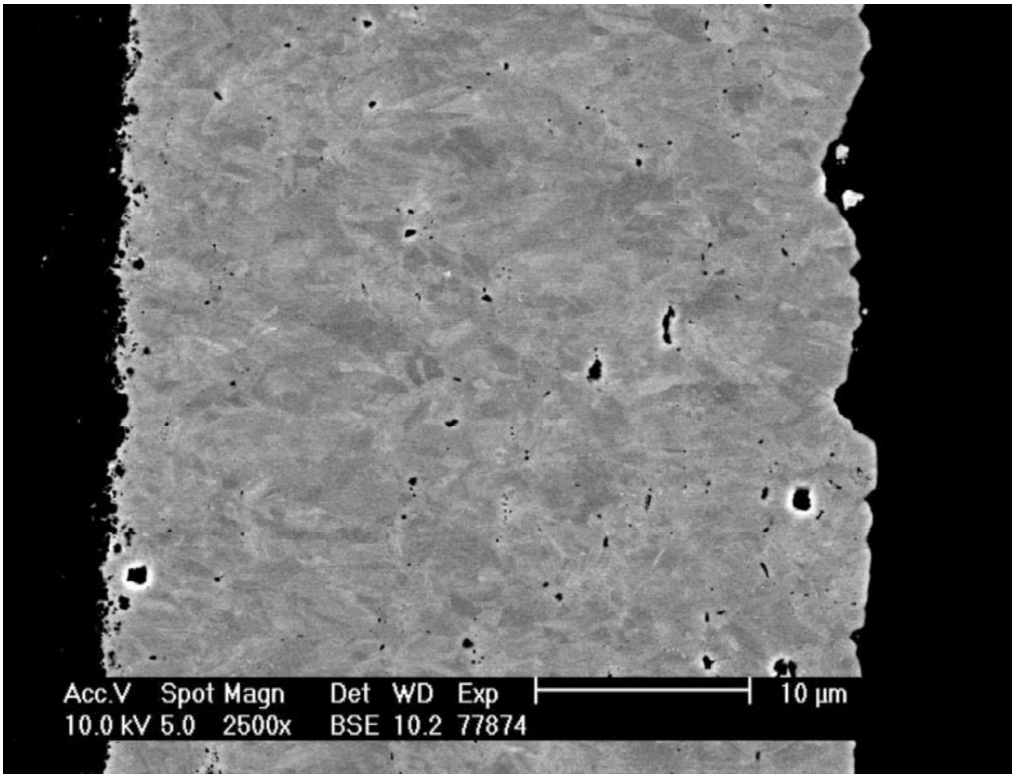


Figure 24. Particle 1 SiC grain structure shown by BSE.

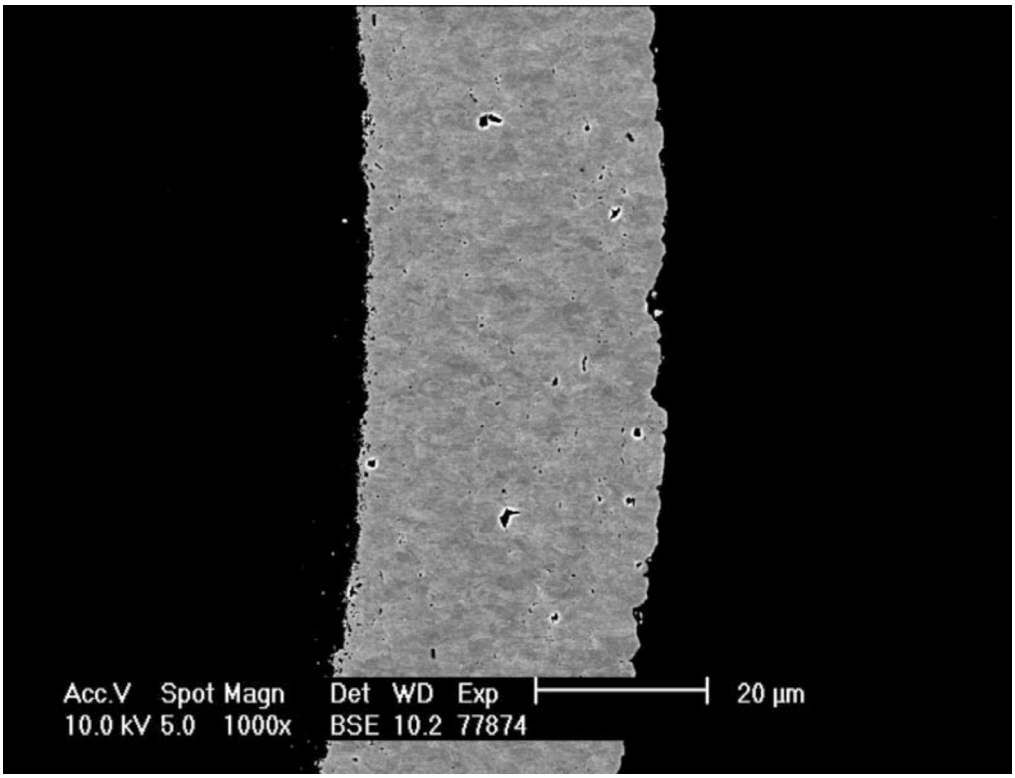


Figure 25. Particle 1 SiC grain structure shown by BSE.

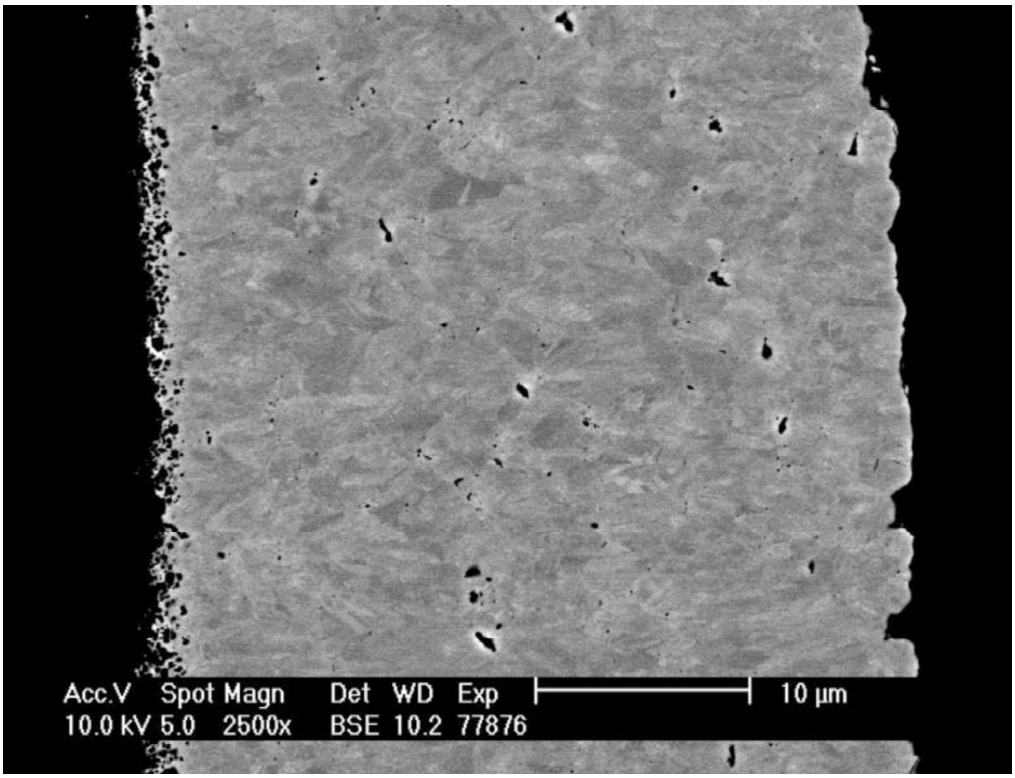


Figure 26. Particle 2 SiC grain structure shown by BSE.

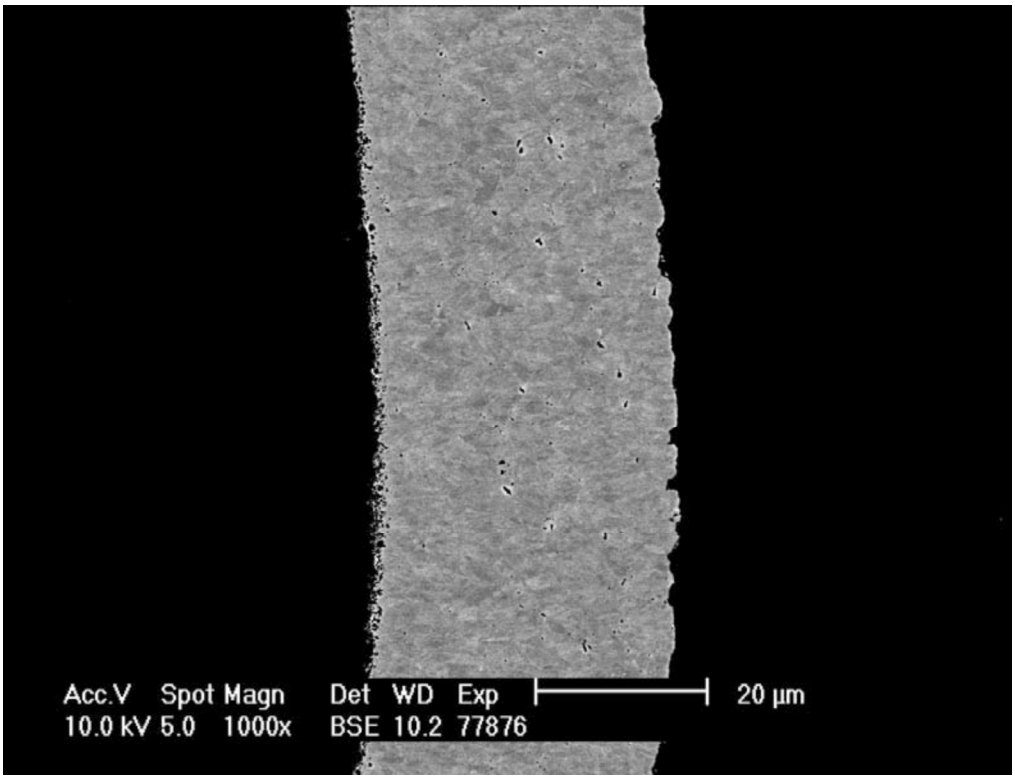


Figure 27. Particle 2 SiC grain structure shown by BSE.

One particle was selected at random and imaged using a high resolution x-ray tomograph.

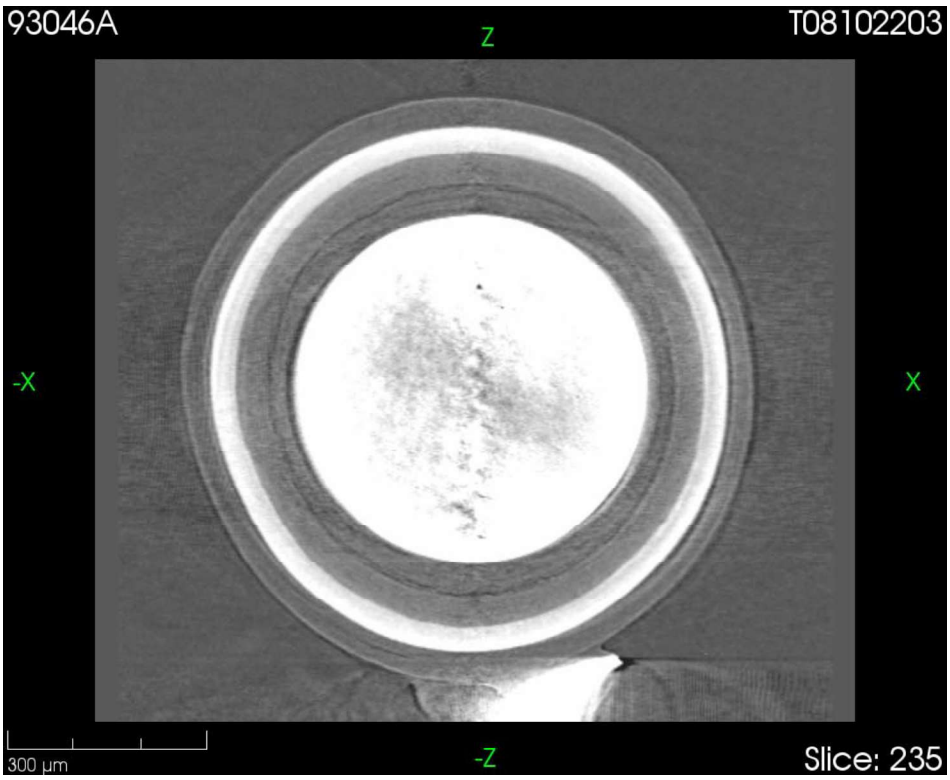


Figure 28. Tomographic cross-section of particle with rotation axis (z) in plane.

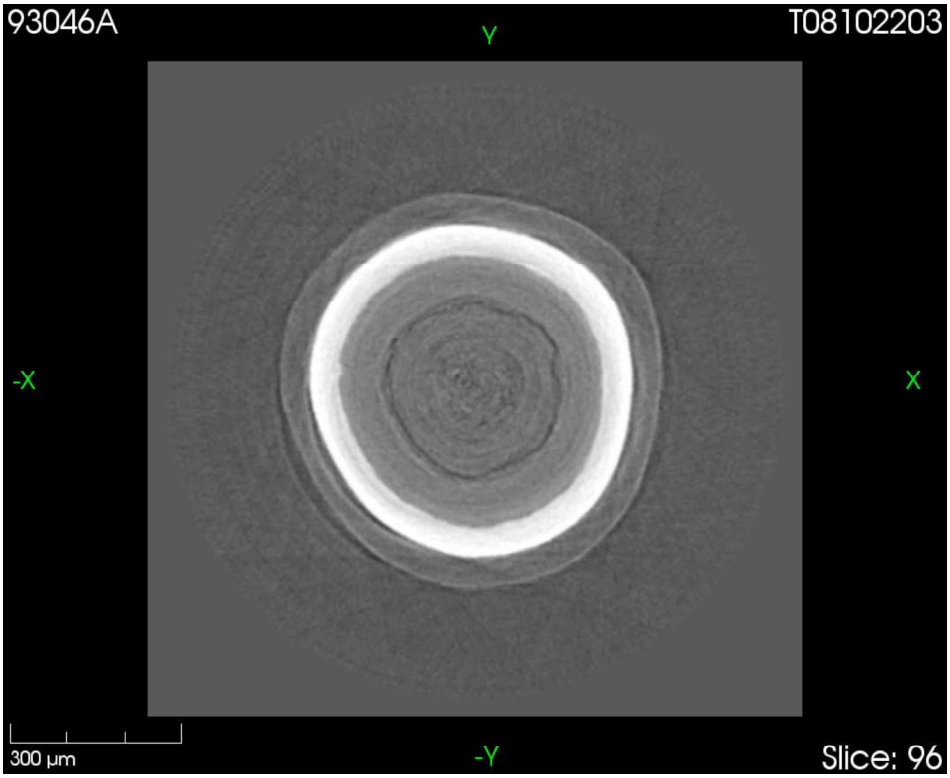


Figure 29. Tomographic cross-section perpendicular to rotation axis (z) showing a plane above kernel, where kernel effects on tomographic reconstruction are not present.

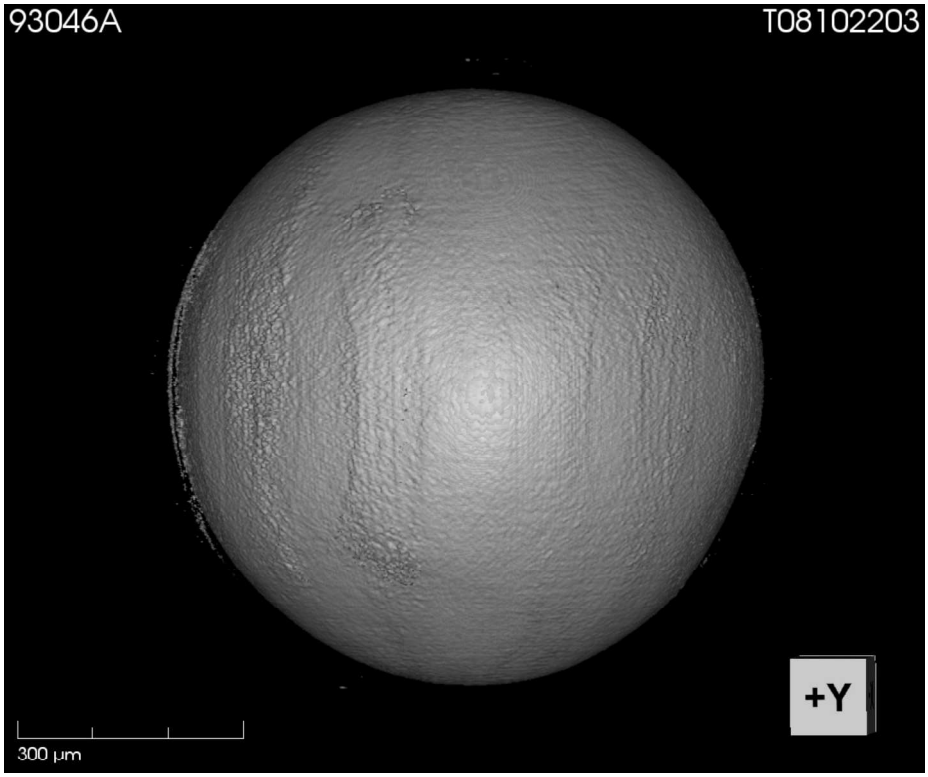


Figure 30. Tomographic 3-D visualization of SiC surface, random orientation.

QC of KAERI TRISO Sample

This section contains the detailed data for the characterization measurements performed on KAERI TRISO sample TR-64.

On the following page is an inspection report form that summarizes the results of each measurement. The mean and standard deviation for each measurement is listed in the table, where appropriate. Also provided on this form are the statistically calculated 95% confidence limits for the mean and dispersion of the parent batch of material.

A and B are the minimum and maximum predicted values for the mean of the parent batch up to the 95% confidence level.

$$A = \bar{x} - \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$$B = \bar{x} + \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$\bar{x}$ and σ_x are the mean and the standard deviation obtained from the measurement performed on a sample of n items. t_{n-1} is the one-tailed Student's-t value for n-1 degrees of freedom.

C is the minimum value for which all but 1% of the population is above (at 95% confidence). D is the maximum value for which all but 1% of the population is below (at 95% confidence).

$$C = \bar{x} - k_n\sigma_x$$

$$D = \bar{x} + k_n\sigma_x$$

k_n is the one-tailed tolerance factor for a sample of n items, given a 1% tolerance limit and a 95% confidence level.

Following the inspection report form are the individual data report forms for each measurement. These forms provide more detail of the measurement performed and include either data for each measured item or histograms of this data.

At the end of this section are images of a few particles obtained using scanning electron microscopy and x-ray tomography. These images show the typical microstructure of the coating layers and interfaces.

Inspection Report Form IRF-CRP6: QC Benchmark Measurements on Coated Particles

Coated particle composite ID:	TR-64
Coated particle composite description:	KAERI CRP-6 QC Benchmark TRISO

Property	Measured Data				95% Confidence Limits for Particle Lot			Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value				
SiC sink/float density (Mg/m³)	3.2058	0.0023	40	1.685	mean ≥ A	A = x - ts/√n	3.205	DRF-02
					mean ≤ B	B = x + ts/√n	3.206	
				2.941	99% of population > C	C = x - ks	3.199	
					99% of population < D	D = x + ks	3.213	
OPyC sink/float density (Mg/m³)	1.4448	0.0068	48	1.678	mean ≥ A	A = x - ts/√n	1.443	DRF-03
					mean ≤ B	B = x + ts/√n	1.446	
				2.868	99% of population > C	C = x - ks	1.425	
					99% of population < D	D = x + ks	1.464	
Particle Diameter (µm)	1001.0	23.0	566	1.648	mean ≥ A	A = x - ts/√n	999.4	DRF-07 DRF-10
					mean ≤ B	B = x + ts/√n	1002.6	
				2.465	99% of population > C	C = x - ks	944.3	
					99% of population < D	D = x + ks	1057.7	
Particle aspect ratio	1.055		566		number of particles > 1.14		0	DRF-07 DRF-10
Average buffer thickness for each particle (µm)	106.0	7.8	180	1.653	mean ≥ A	A = x - ts/√n	105.0	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	107.0	
				2.582	99% of population > C	C = x - ks	85.9	
					99% of population < D	D = x + ks	126.1	
Average IPyC thickness for each particle (µm)	45.3	2.0	194	1.653	mean ≥ A	A = x - ts/√n	45.1	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	45.5	
				2.572	99% of population > C	C = x - ks	40.2	
					99% of population < D	D = x + ks	50.4	
Average SiC thickness for each particle (µm)	35.4	2.1	222	1.652	mean ≥ A	A = x - ts/√n	35.2	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	35.6	
				2.555	99% of population > C	C = x - ks	30.0	
					99% of population < D	D = x + ks	40.8	
Average OPyC thickness for each particle (µm)	46.0	4.5	95	1.661	mean ≥ A	A = x - ts/√n	45.2	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	46.8	
				2.691	99% of population > C	C = x - ks	33.9	
					99% of population < D	D = x + ks	58.1	
IPyC anisotropy (BAFo)	1.0042	0.0002	10	1.833	mean ≥ A	A = x - ts/√n	1.0041	DRF-18
					mean ≤ B	B = x + ts/√n	1.0043	
				3.981	99% of population > C	C = x - ks	1.0034	
					99% of population < D	D = x + ks	1.0050	
OPyC anisotropy (BAFo)	1.0045	0.0002	10	1.833	mean ≥ A	A = x - ts/√n	1.0044	DRF-18
					mean ≤ B	B = x + ts/√n	1.0046	
				3.981	99% of population > C	C = x - ks	1.0037	
					99% of population < D	D = x + ks	1.0053	
Average particle weight (mg)					mean ≥ A	A = x - ts/√n		DRF-22
					mean ≤ B	B = x + ts/√n		
					99% of population > C	C = x - ks		
					99% of population < D	D = x + ks		
OPyC open porosity (ml/m²)								DRF-31

Comments

Sample was analyzed by SEM for SiC grain structure and general microstructure of layers and interfaces.
A particle was imaged using high resolution x-ray tomography.
Difficulty in identifying outer OPyC boundary due to high porosity may have affected accuracy of coating thickness measurement.
Particle weight and open porosity were not measured because the available sample size was insufficient for these tasks.

John Kim
QC Supervisor

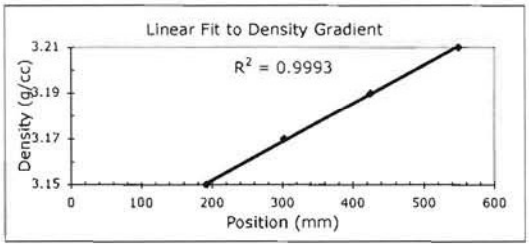
11-14-08
Date

Data Report Form DRF-02: Measurement of SiC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-02 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08040801_DRF02R3.xls
Sample ID:	TR-64
Sample description:	KAERI CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.4 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
3.150	178.45	199.06	192.19
3.170	286.79	310.27	302.44
3.190	410.38	433.10	425.53
3.210	533.44	556.94	549.11

Linear Fit			
slope	StDev	intercept	StDev
1.67E-04	3.09E-06	3.12E+00	1.13E-03



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	491.63	3.2008	26	525.50	3.2065	51		
2	492.91	3.2010	27	526.18	3.2066	52		
3	493.62	3.2011	28	527.83	3.2069	53		
4	499.83	3.2022	29	527.83	3.2069	54		
5	503.85	3.2029	30	527.83	3.2069	55		
6	512.35	3.2043	31	519.96	3.2056	56		
7	515.00	3.2047	32	514.58	3.2047	57		
8	516.93	3.2050	33	512.32	3.2043	58		
9	519.84	3.2055	34	503.89	3.2029	59		
10	521.00	3.2057	35	491.92	3.2009	60		
11	524.78	3.2064	36	524.60	3.2063	61		
12	523.60	3.2062	37	526.07	3.2066	62		
13	524.77	3.2064	38	527.36	3.2068	63		
14	525.65	3.2065	39	530.51	3.2073	64		
15	526.79	3.2067	40	532.47	3.2076	65		
16	529.27	3.2071	41			66		
17	530.00	3.2072	42			67		
18	531.97	3.2076	43			68		
19	531.85	3.2075	44			69		
20	533.79	3.2079	45			70		
21	535.80	3.2082	46			71		
22	535.80	3.2082	47			72		
23	536.15	3.2083	48			73		
24	539.50	3.2088	49			74		
25	543.95	3.2096	50			75		

Average density of SiC fragments:	3.2058
Standard deviation in density of SiC fragments:	0.0023
Uncertainty in calculated density of SiC fragments:	0.0020

Dixie Barker
Operator

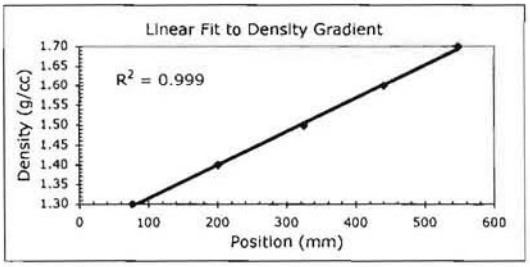
4-8-08
Date

Data Report Form DRF-03: Measurement of PyC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08111301_DRF03R3_1,3-1,7 range.xls
Sample ID:	TR-64
Sample description:	KAERI CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	10/2009
Bath temperature:	23.7 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.300	73.51	80.10	76.81
1.400	197.14	205.58	201.36
1.500	322.31	328.21	325.26
1.600	438.44	443.79	441.12
1.700	544.49	551.14	547.82

Linear Fit			
slope	StDev	intercept	StDev
8.46E-04	4.77E-06	1.23E+00	1.53E-03



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	235.16	1.4295	26	255.56	1.4468	51		
2	236.71	1.4309	27	256.01	1.4472	52		
3	238.61	1.4325	28	256.01	1.4472	53		
4	239.36	1.4331	29	256.64	1.4477	54		
5	240.43	1.4340	30	257.12	1.4481	55		
6	243.09	1.4363	31	258.51	1.4493	56		
7	243.25	1.4364	32	258.51	1.4493	57		
8	244.29	1.4373	33	255.32	1.4466	58		
9	245.51	1.4383	34	256.16	1.4473	59		
10	246.92	1.4395	35	256.16	1.4473	60		
11	247.81	1.4402	36	257.68	1.4486	61		
12	248.08	1.4405	37	258.59	1.4494	62		
13	249.15	1.4414	38	258.91	1.4496	63		
14	249.15	1.4414	39	259.76	1.4504	64		
15	249.76	1.4419	40	261.41	1.4517	65		
16	250.29	1.4423	41	262.29	1.4525	66		
17	250.06	1.4421	42	262.84	1.4530	67		
18	251.67	1.4435	43	262.84	1.4530	68		
19	251.67	1.4435	44	263.68	1.4537	69		
20	251.67	1.4435	45	265.06	1.4548	70		
21	252.01	1.4438	46	265.06	1.4548	71		
22	250.18	1.4422	47	267.88	1.4572	72		
23	253.92	1.4454	48	265.57	1.4553	73		
24	253.92	1.4454	49			74		
25	255.16	1.4465	50			75		

Average density of PyC fragments:	1.4448
Standard deviation in density of PyC fragments:	0.0068
Uncertainty in calculated density of PyC fragments:	0.0020

Dixie Barker
Operator

11-13-08
Date

Data Report Form DRF-29: Imaging of Overcoated Particle Diameter and Aspect Ratio Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-29 Rev. 1
Operator:	John Hunn
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing images:	\\Mc-agr\agr\ImageProcessing\P08031802\

DMR Calibration Expiration Date:	9/20/08
Stage Micrometer Calibration Expiration Date:	2/13/09
Measured Value for 2500 μm in Stage Micrometer Image:	2500. μm

John Hunn
Operator

3-18-08
Date

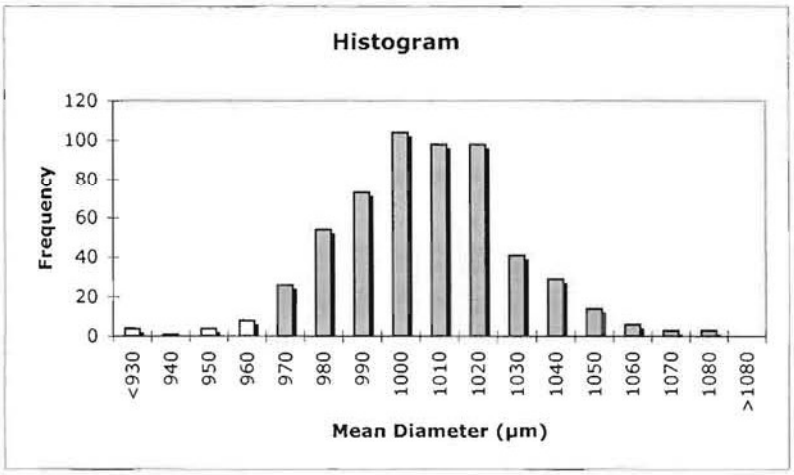
Data Report Form DRF-30A: Measurement of Over-coated Particle Diameter

Procedure:	AGR-CHAR-DAM-30 Rev. 0
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08031802\
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08031802_output\

Number of particles analyzed:	566
Mean of the average diameter of each particle (μm):	1001.0
Standard deviation in the average diameter of each particle (μm):	23

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<930	4
940	1
950	4
960	8
970	26
980	54
990	73
1000	104
1010	98
1020	98
1030	41
1040	29
1050	14
1060	6
1070	3
1080	3
>1080	0



Operator

March 24, 2008
Date

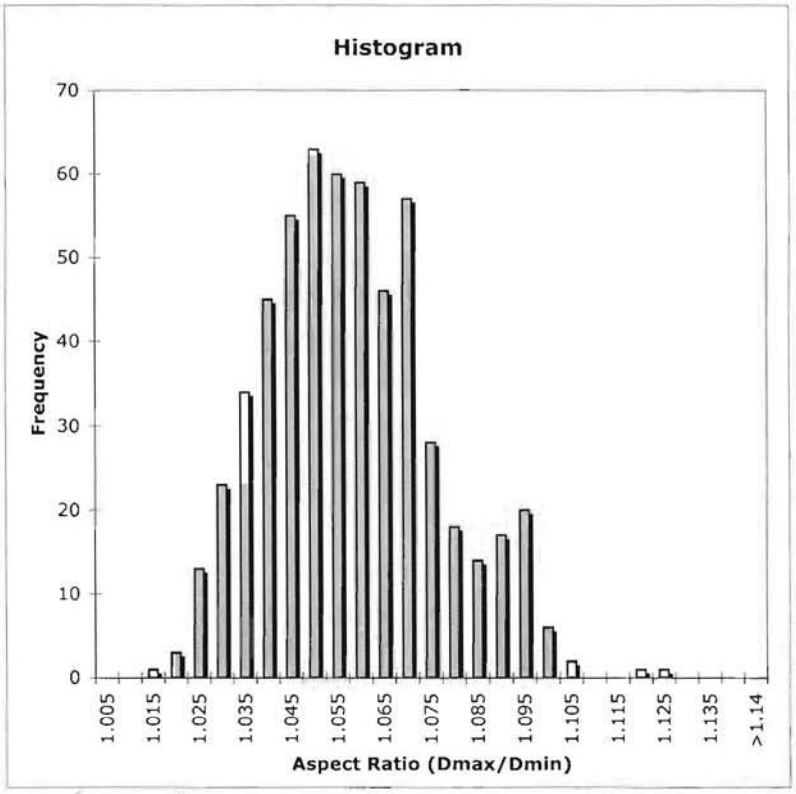
Data Report Form DRF-30B: Measurement of Over-coated Particle Aspect Ratio (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-30 Rev. 0
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08031802\
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08031802_output\

Number of particles analyzed:	566
Average particle aspect ratio:	1.055

Distribution of the aspect ratio (top binned)

Aspect Ratio (D)	*Frequency*
1.005	0
1.010	0
1.015	1
1.020	3
1.025	13
1.030	23
1.035	34
1.040	45
1.045	55
1.050	63
1.055	60
1.060	59
1.065	46
1.070	57
1.075	28
1.080	18
1.085	14
1.090	17
1.095	20
1.100	6
1.105	2
1.110	0
1.115	0
1.120	1
1.125	1
1.130	0
1.135	0
1.140	0
>1.14	0



Operator

March 24, 2008

Date

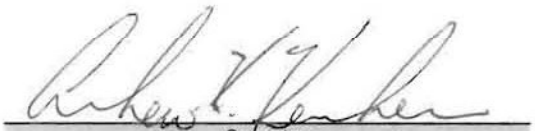
Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	TR-64
Sample description:	KAERI CRP-6 QC Benchmark TRISO
Mount ID number:	M08032601
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08062501\P0806250101\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 μm in stage micrometer image (μm):	500.0

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
455	477	467	482

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
257	105	46	37	51



Operator

June 25, 2008

Date

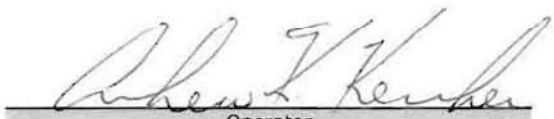
Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	TR-64
Sample description:	KAERI CRP-6 QC Benchmark TRISO
Mount ID number:	M08032602
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08062501\P0806250102\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 µm in stage micrometer image (µm):	500.0

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
493	500	499	507

Approximate layer width in polish plane (µm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
252	115	46	35	52



Operator

June 25, 2008

Date

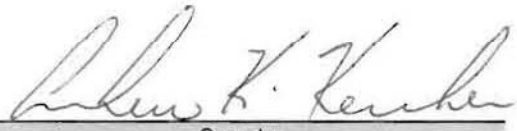
Data Report Form DRF-08: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	TR-64
Sample description:	KAERI CRP-6 QC Benchmark TRISO
Mount ID number:	M08032603
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08062501\P0806250103\

DMR calibration expiration date:	9/20/2008
Calibrated pixels/micron:	2.8280
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 500 µm in stage micrometer image (µm):	500.0

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
461	478	483	493

Approximate layer width in polish plane (µm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
255	113	46	36	52



Operator

June 25, 2008

Date

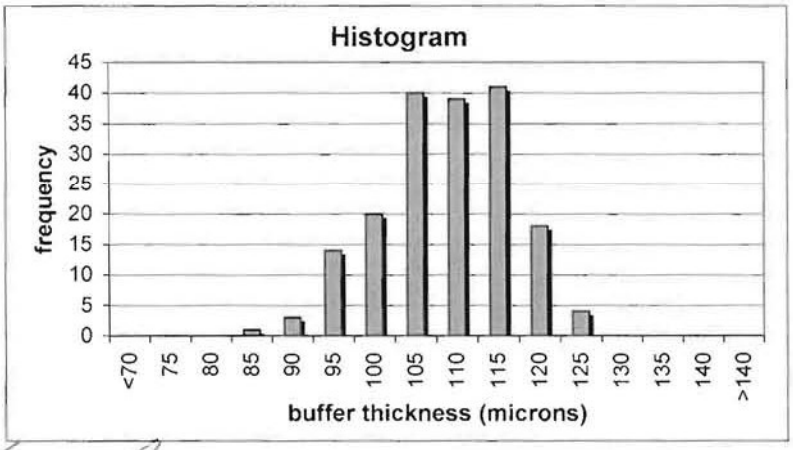
Data Report Form DRF-11A: Measurement of Buffer Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501\
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501_output\

Number of buffer layers analyzed:	180
Mean of the average buffer thickness of each particle (μm):	106.0
Standard deviation in the average buffer thickness of each particle (μm):	7.8

Distribution of the average buffer layer thickness (top binned)

Buffer Thickness (μm)	*Frequency*
<70	0
75	0
80	0
85	1
90	3
95	14
100	20
105	40
110	39
115	41
120	18
125	4
130	0
135	0
140	0
>140	0



Andrew K. Kercher
Operator

June 27, 2008
Date

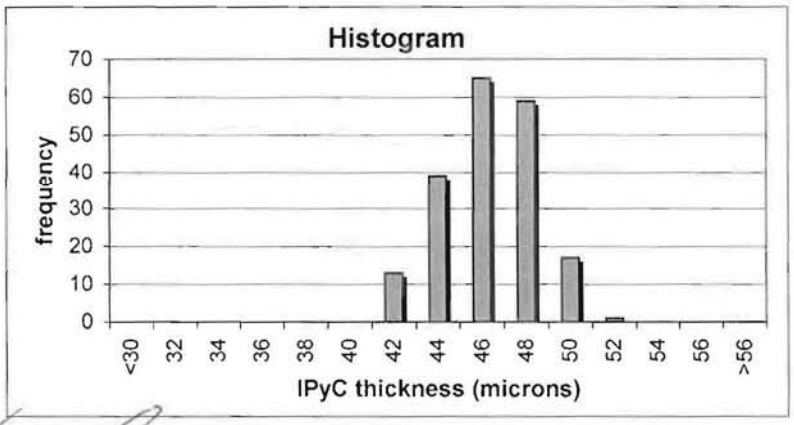
Data Report Form DRF-11B: Measurement of Inner Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501\
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501_output\

Number of inner pyrocarbon layers analyzed:	194
Mean of the average IPyC thickness of each particle (μm):	45.3
Standard deviation in the average IPyC thickness of each particle (μm):	2.0

Distribution of the average IPyC layer thickness (top binned)

IPyC Thickness (μm)	*Frequency*
<30	0
32	0
34	0
36	0
38	0
40	0
42	13
44	39
46	65
48	59
50	17
52	1
54	0
56	0
>56	0



Andrew K. Kercher
Operator

June 27, 2008
Date

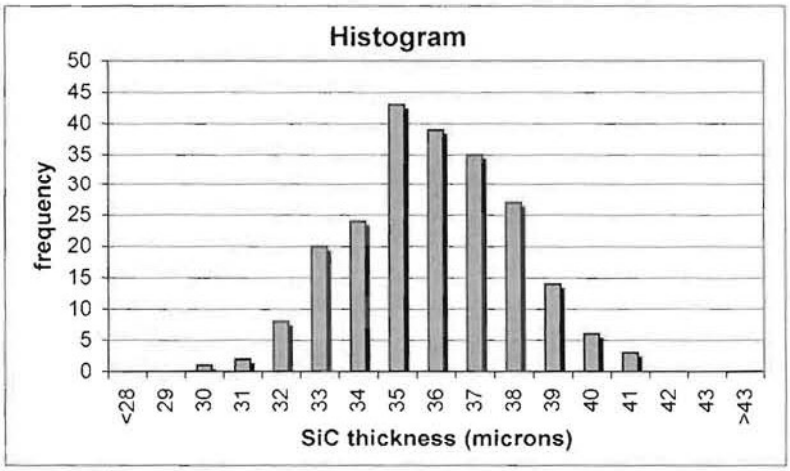
Data Report Form DRF-11C: Measurement of Silicon Carbide Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501\
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501_output\

Number of silicon carbide layers analyzed:	222
Mean of the average SiC thickness of each particle (μm):	35.4
Standard deviation in the average SiC thickness of each particle (μm):	2.1

Distribution of the average SiC layer thickness (top binned)

SiC Thickness (μm)	*Frequency*
<28	0
29	0
30	1
31	2
32	8
33	20
34	24
35	43
36	39
37	35
38	27
39	14
40	6
41	3
42	0
43	0
>43	0



Andrew K. Kercher — June 27, 2008

Operator — Date

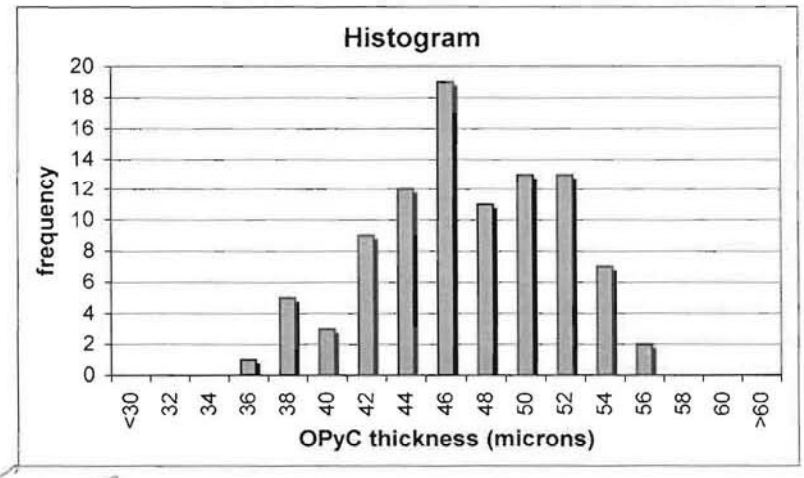
Data Report Form DRF-11D: Measurement of Outer Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501\
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08062501_output\

Number of outer pyrocarbon layers analyzed:	95
Mean of the average OPyC thickness of each particle (μm):	46.0
Standard deviation in the average OPyC thickness of each particle (μm):	4.5

Distribution of the average OPyC layer thickness (top binned)

OPyC Thickness (μm)	*Frequency*
<30	0
32	0
34	0
36	1
38	5
40	3
42	9
44	12
46	19
48	11
50	13
52	13
54	7
56	2
58	0
60	0
>60	0



Operator

June 27, 2008
Date

High porosity in the outer portion of the OPyC layer resulted in an increased uncertainty in determining the thickness of this layer due to the undefined nature of the outer boundary. Only 95 out of the 240 particles analyzed produced error free image analysis results for the OPyC outer boundary. In addition, because the identified outer OPyC boundary included areas where large pores were cross-sectioned, the measured thickness was less than would be obtained if these regions of apparent thinner OPyC were not included.

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08032802
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08060501\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	3,3	0.0022	0.0011	0.0005	1.0044	0.0022	0.0010
2	3,4	0.0021	0.0010	0.0005	1.0042	0.0020	0.0010
3	3,5	0.0022	0.0010	0.0005	1.0044	0.0020	0.0010
4	4,3	0.0020	0.0010	0.0005	1.0040	0.0020	0.0010
5	4,4	0.0021	0.0010	0.0005	1.0042	0.0020	0.0010
6	4,5	0.0020	0.0009	0.0009	1.0040	0.0018	0.0018
7	5,3	0.0022	0.0010	0.0005	1.0044	0.0020	0.0010
8	5,4	0.0021	0.0010	0.0005	1.0042	0.0020	0.0010
9	5,5	0.0022	0.0010	0.0006	1.0044	0.0020	0.0012
10	4,6	0.0021	0.0011	0.0005	1.0042	0.0022	0.0010
Average		0.0021	0.0010	0.0006	1.0042	0.0020	0.0011

Mean of average BAFo per particle:	1.0042
Standard deviation of average BAFo per particle:	0.0002

Comments

A. E. Jellison
Operator

6/05/08
Date

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08032802
Sample ID:	TR-64
Sample Description:	KAERI CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08060501\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	3,3	0.0023	0.0012	0.0005	1.0046	0.0024	0.0010
2	3,4	0.0022	0.0012	0.0005	1.0044	0.0024	0.0010
3	3,5	0.0021	0.0011	0.0005	1.0042	0.0022	0.0010
4	4,3	0.0023	0.0011	0.0005	1.0046	0.0022	0.0010
5	4,4	0.0023	0.0012	0.0006	1.0046	0.0024	0.0012
6	4,5	0.0021	0.0011	0.0006	1.0042	0.0022	0.0012
7	5,3	0.0024	0.0012	0.0006	1.0048	0.0024	0.0012
8	5,4	0.0024	0.0012	0.0006	1.0048	0.0024	0.0012
9	5,5	0.0023	0.0012	0.0006	1.0046	0.0024	0.0012
10	4,6	0.0022	0.0011	0.0006	1.0044	0.0022	0.0012
Average		0.0023	0.0012	0.0006	1.0045	0.0023	0.0011

Mean of average BAFo per particle:	1.0045
Standard deviation of average BAFo per particle:	0.0002

Comments

G. E. Jellison — Operator

6/05/08 — Date

A scanning electron microscope (SEM) was used to image particles polished close to midplane. The following 4 secondary electron (SE) images show the TRISO coatings and interfaces.

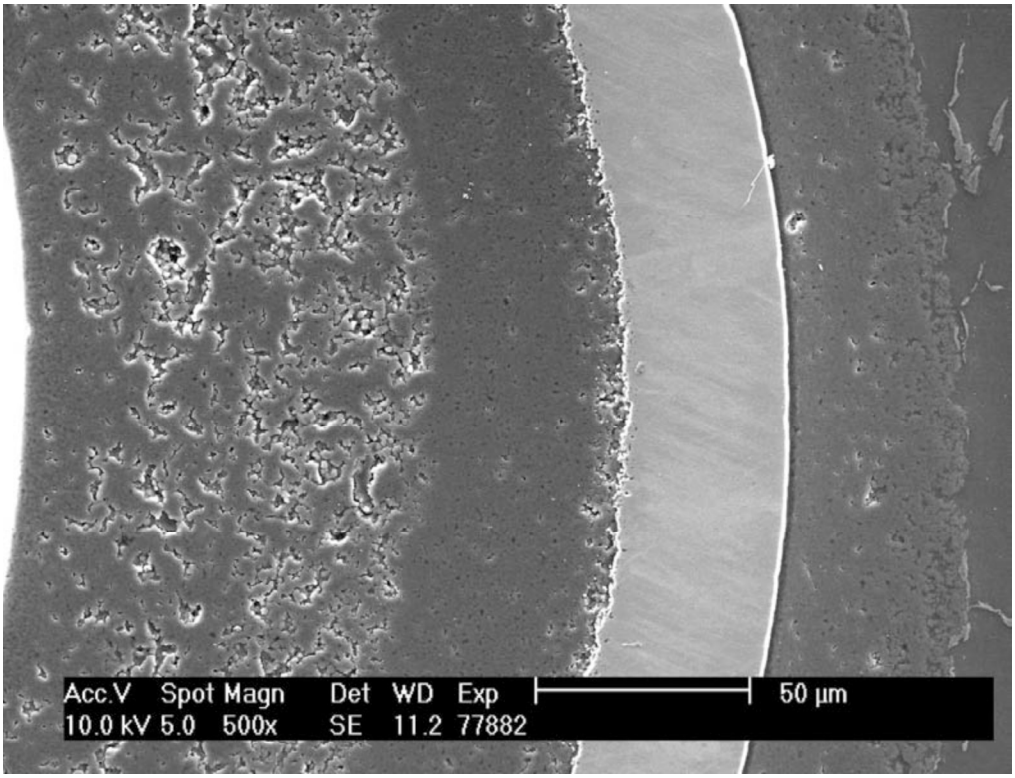


Figure 31. SE image of TRISO coatings on a typical particle.

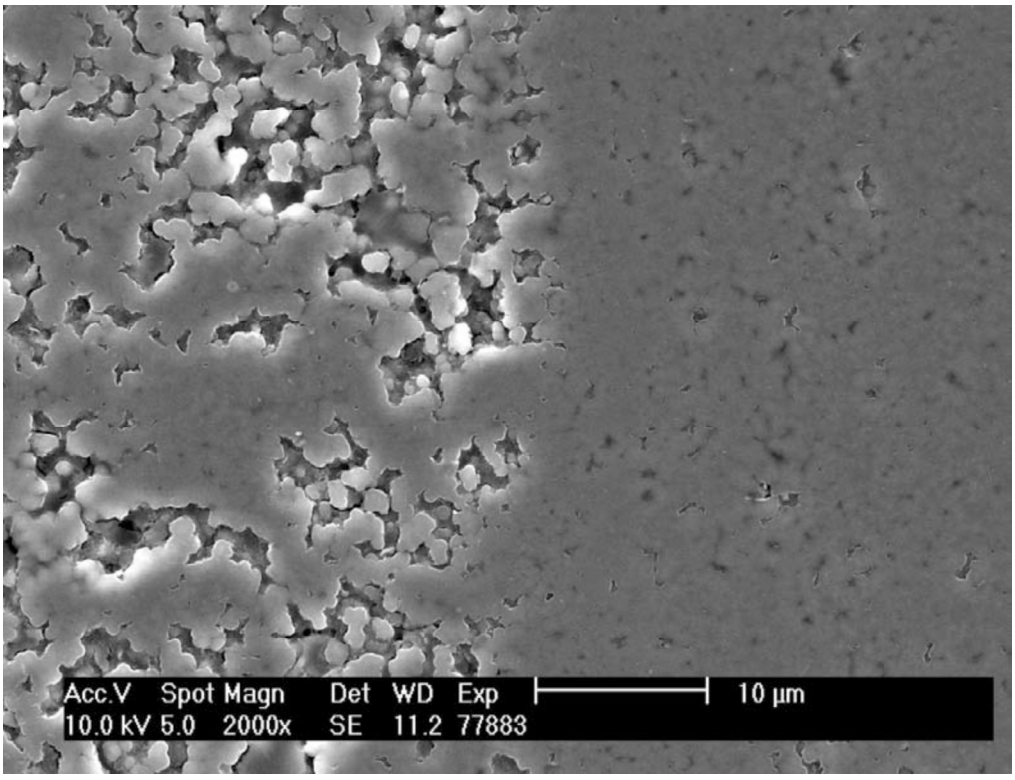


Figure 32. SE image of Buffer/IPyC interface.

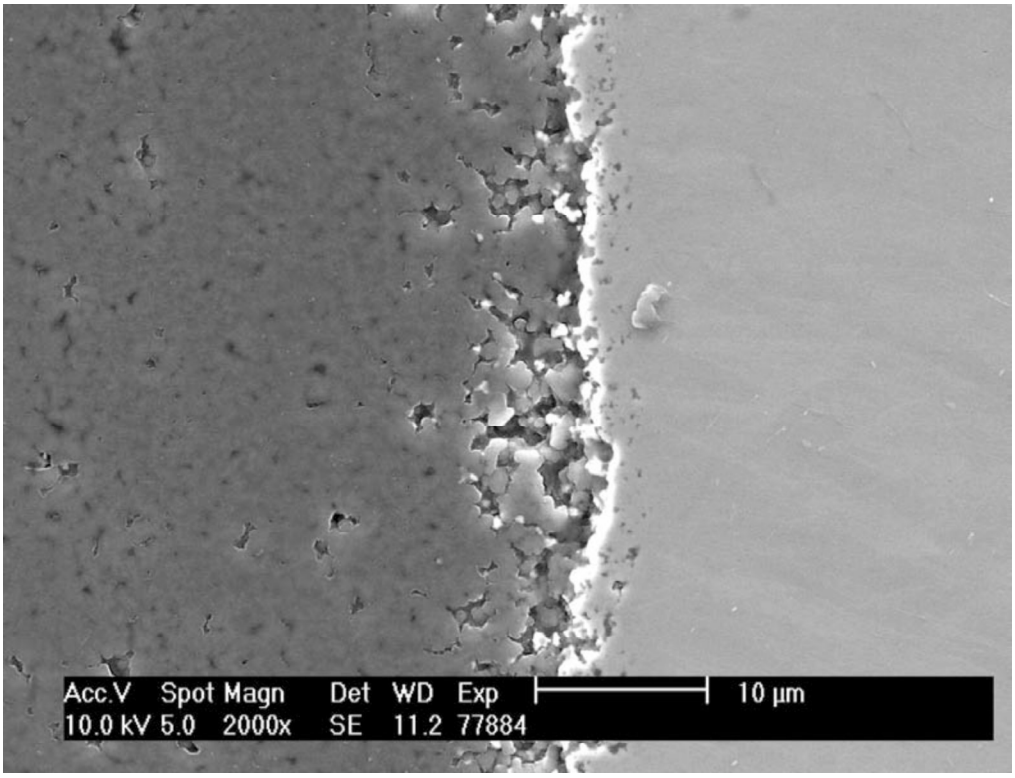


Figure 33. SE image of IPyC/SiC interface.

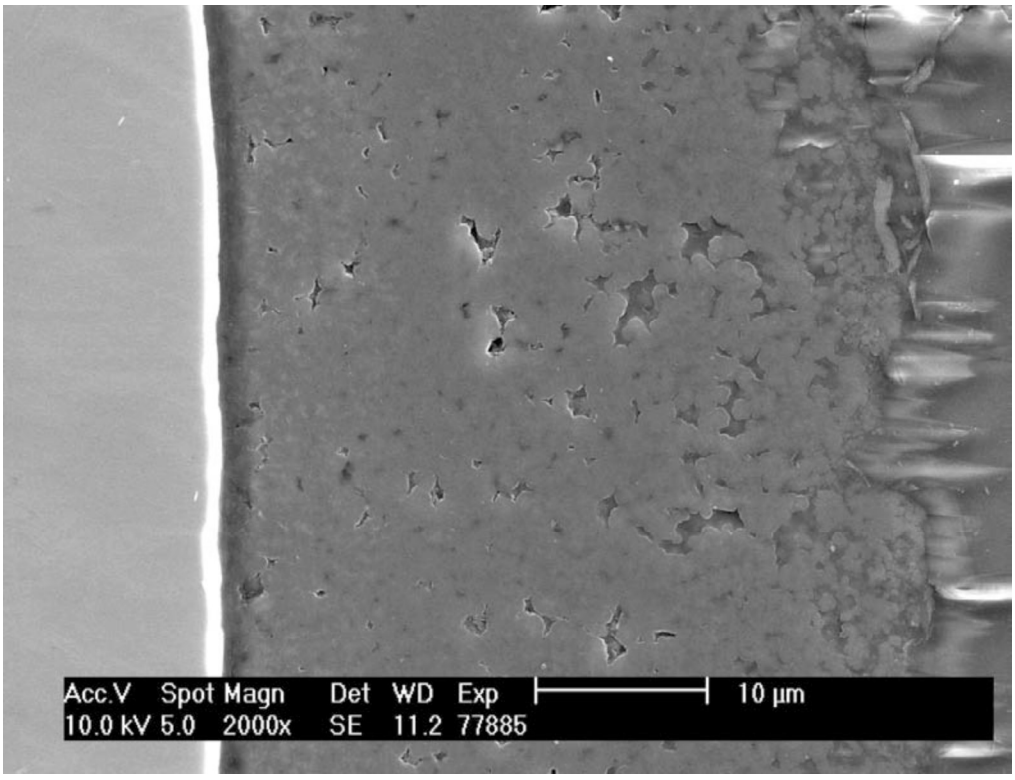


Figure 34. SE image of SiC/OPyC interface.

Backscattered electron imaging (BSE) was used to view the SiC grain structure. The following images show 2 different particles, each at two magnifications.

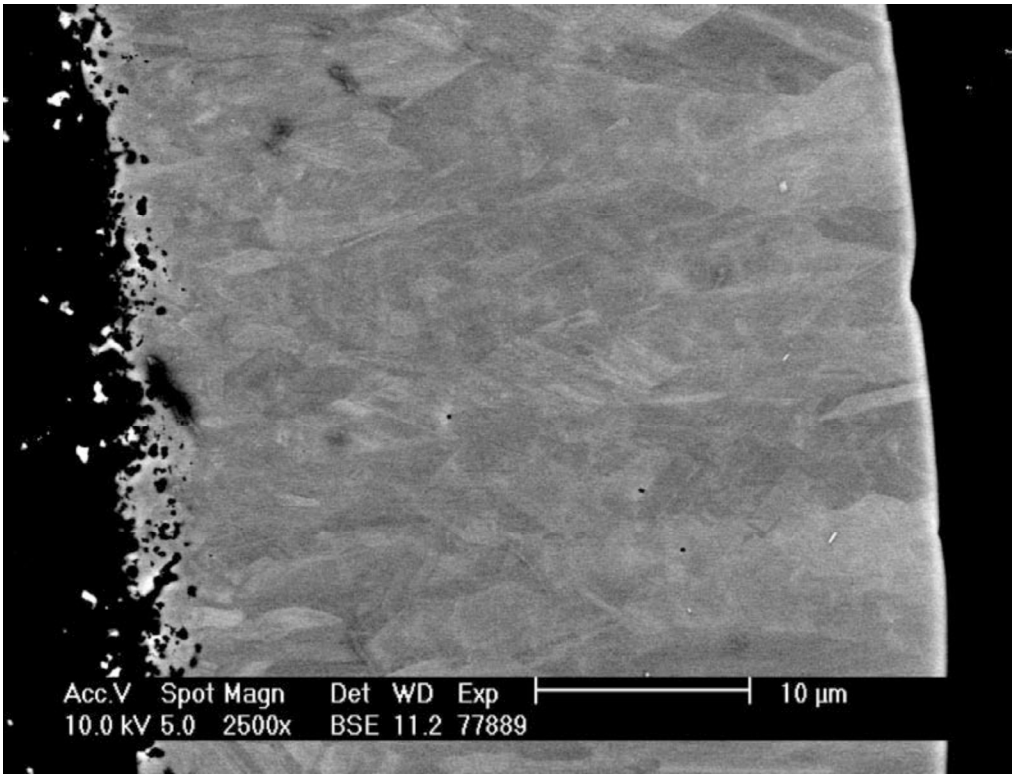


Figure 35. Particle 1 SiC grain structure shown by BSE.

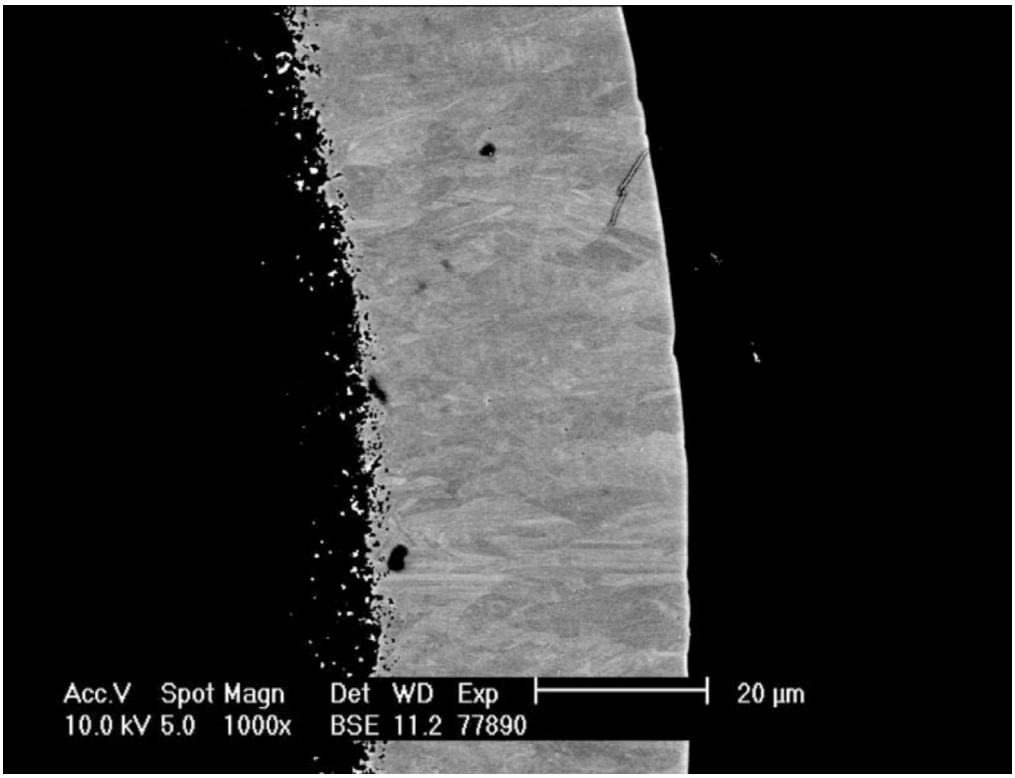


Figure 36. Particle 1 SiC grain structure shown by BSE.

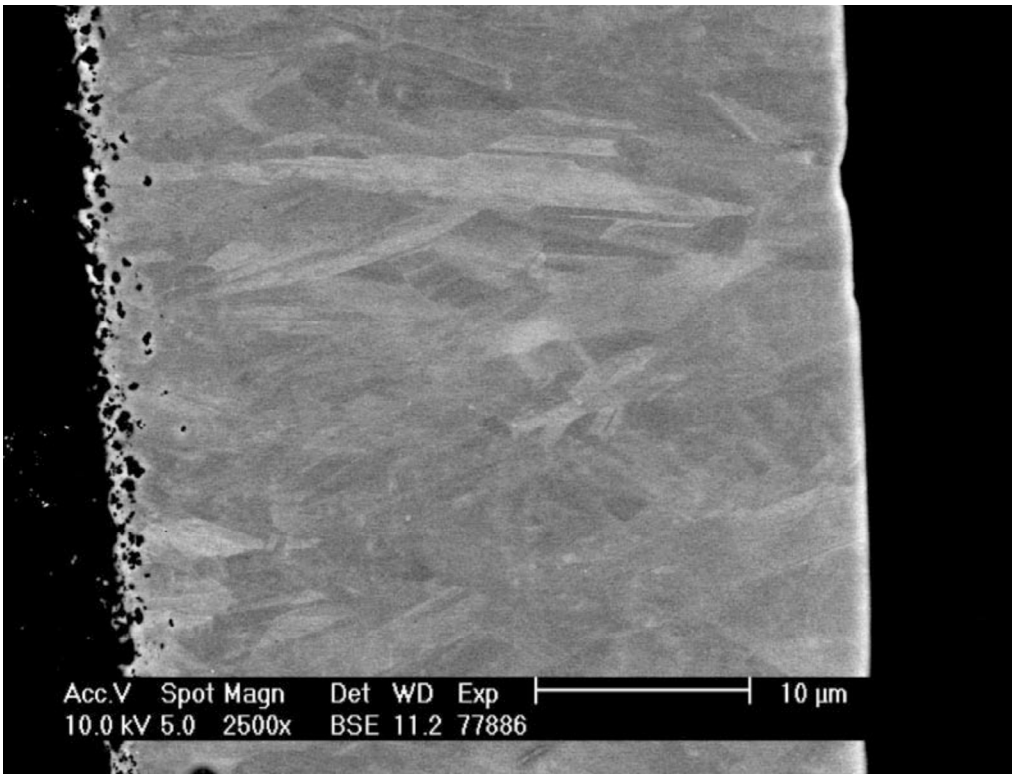


Figure 37. Particle 2 SiC grain structure shown by BSE.

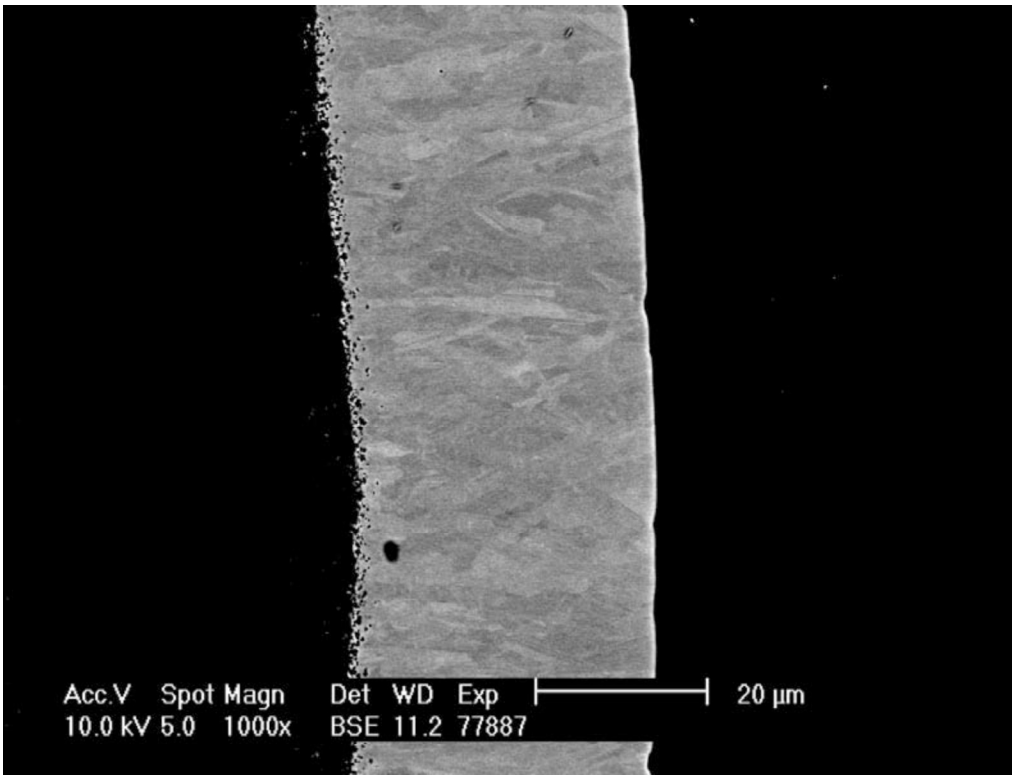


Figure 38. Particle 2 SiC grain structure shown by BSE.

One particle was selected at random and imaged using a high resolution x-ray tomograph.

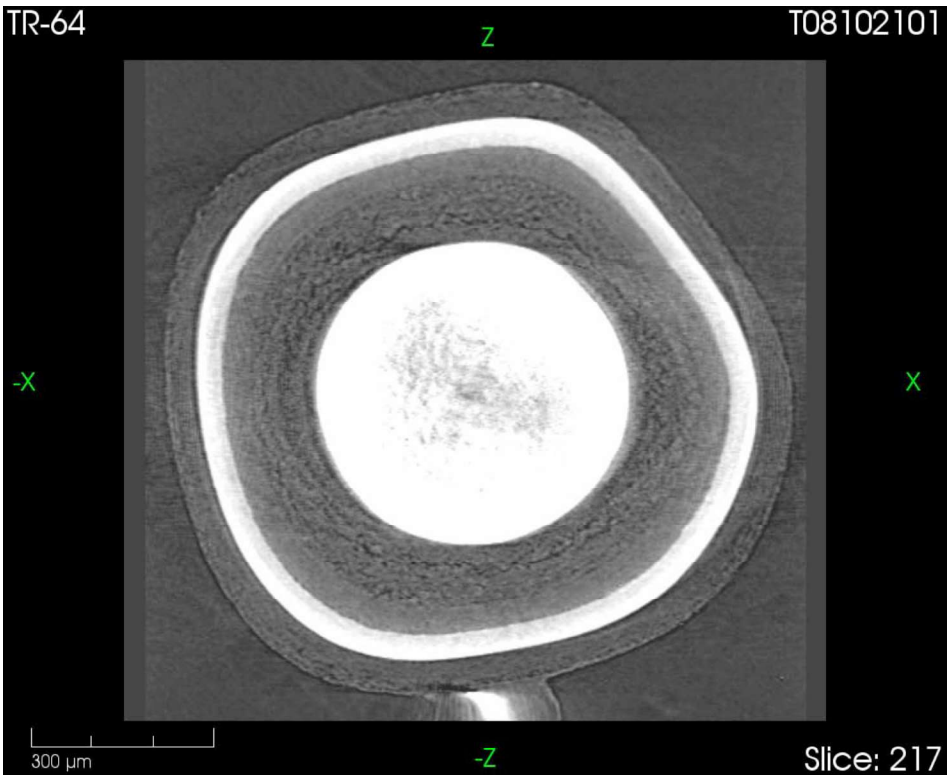


Figure 39. Tomographic cross-section of particle with rotation axis (z) in plane.

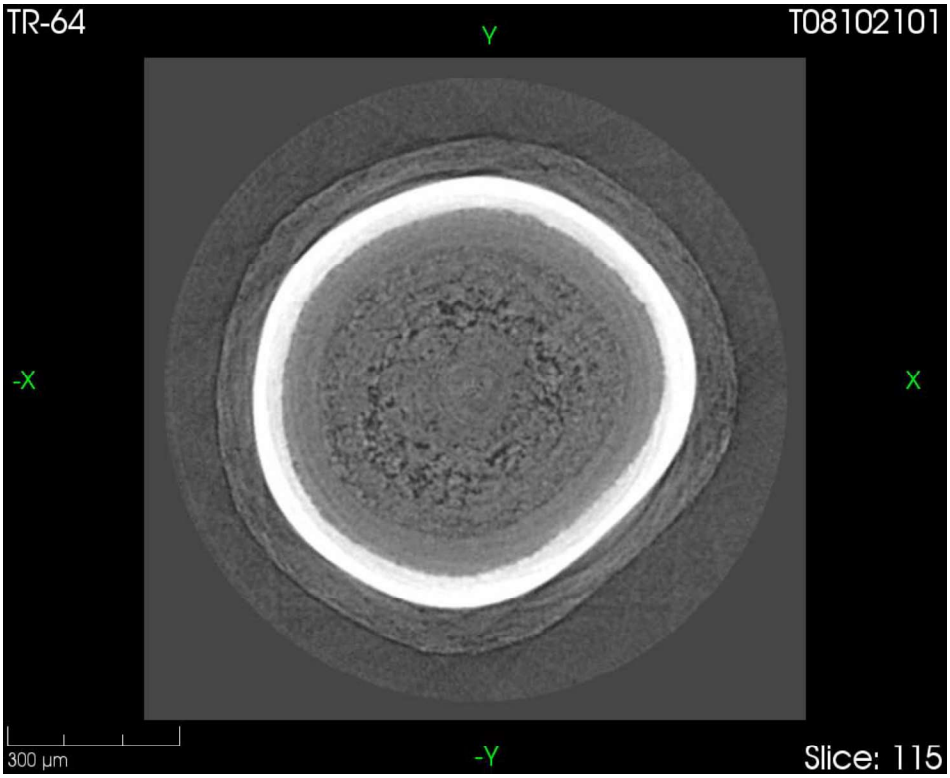


Figure 40. Tomographic cross-section perpendicular to rotation axis (z) showing a plane above kernel, where kernel effects on tomographic reconstruction are not present.

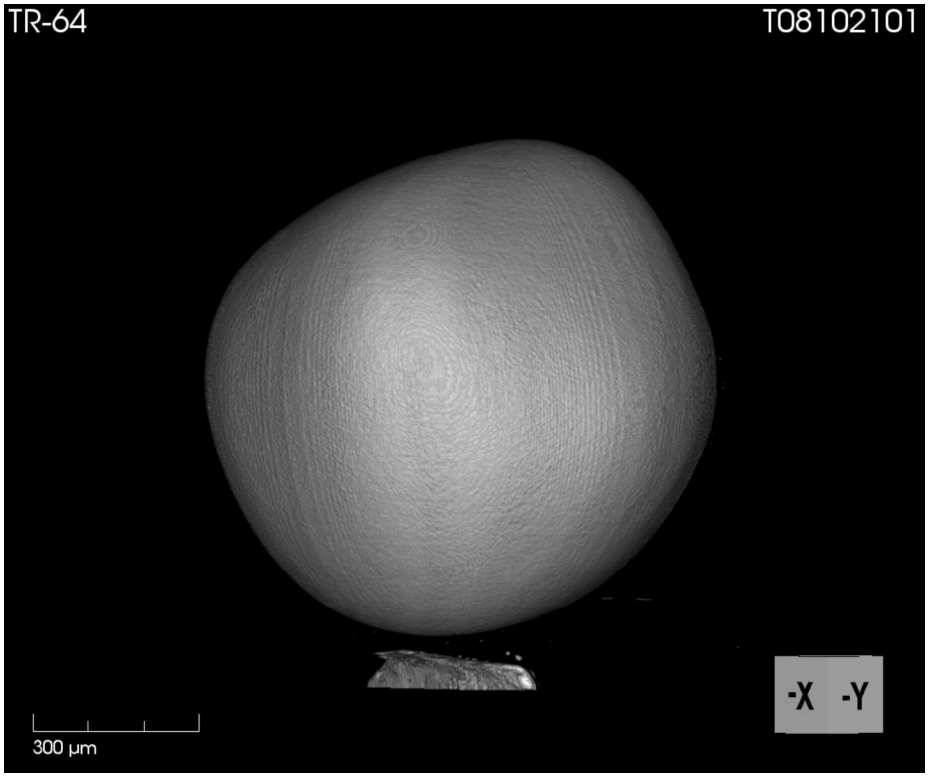


Figure 41. Tomographic 3-D visualization of SiC surface, oriented to show maximum faceting.

QC of PBMR TRISO Sample

This section contains the detailed data for the characterization measurements performed on PBMR TRISO sample CPT-T-G148.

On the following page is an inspection report form that summarizes the results of each measurement. The mean and standard deviation for each measurement is listed in the table, where appropriate. Also provided on this form are the statistically calculated 95% confidence limits for the mean and dispersion of the parent batch of material.

A and B are the minimum and maximum predicted values for the mean of the parent batch up to the 95% confidence level.

$$A = \bar{x} - \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$$B = \bar{x} + \frac{t_{n-1}\sigma_x}{\sqrt{n}}$$

$\bar{x}$ and σ_x are the mean and the standard deviation obtained from the measurement performed on a sample of n items. t_{n-1} is the one-tailed Student's-t value for n-1 degrees of freedom.

C is the minimum value for which all but 1% of the population is above (at 95% confidence). D is the maximum value for which all but 1% of the population is below (at 95% confidence).

$$C = \bar{x} - k_n\sigma_x$$

$$D = \bar{x} + k_n\sigma_x$$

k_n is the one-tailed tolerance factor for a sample of n items, given a 1% tolerance limit and a 95% confidence level.

Following the inspection report form are the individual data report forms for each measurement. These forms provide more detail of the measurement performed and include either data for each measured item or histograms of this data.

At the end of this section are images of a few particles obtained using scanning electron microscopy and x-ray tomography. These images show the typical microstructure of the coating layers and interfaces.

Inspection Report Form IRF-CRP6: QC Benchmark Measurements on Coated Particles

Coated particle composite ID:	CPT-T-G148
Coated particle composite description:	PBMR CRP-6 QC-Benchmark TRISO

Property	Measured Data: Mean (x)	Std. Dev. (s)	# measured (n)	k or t value	95% Confidence Limits for Particle Lot			Data Records
SiC sink/float density (Mg/m³)	3.2073	0.0007	32	1.696	mean ≥ A	A = x - ts/√n	3.207	DRF-02
					mean ≤ B	B = x + ts/√n	3.208	
				3.021	99% of population > C	C = x - ks	3.205	
					99% of population < D	D = x + ks	3.209	
OPyC sink/float density (Mg/m³)	1.6336	0.0067	41	1.684	mean ≥ A	A = x - ts/√n	1.632	DRF-03
					mean ≤ B	B = x + ts/√n	1.635	
				2.922	99% of population > C	C = x - ks	1.614	
					99% of population < D	D = x + ks	1.653	
Particle Diameter (µm)	1076.2	28.0	341	1.649	mean ≥ A	A = x - ts/√n	1073.7	DRF-07 DRF-10
					mean ≤ B	B = x + ts/√n	1078.7	
				2.508	99% of population > C	C = x - ks	1006.0	
					99% of population < D	D = x + ks	1146.4	
Particle aspect ratio	1.041		341		number of particles > 1.14		0	DRF-07 DRF-10
Average buffer thickness for each particle (µm)	115.4	10.7	216	1.652	mean ≥ A	A = x - ts/√n	114.2	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	116.6	
				2.558	99% of population > C	C = x - ks	88.0	
					99% of population < D	D = x + ks	142.8	
Average IPyC thickness for each particle (µm)	78.3	3.9	216	1.652	mean ≥ A	A = x - ts/√n	77.9	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	78.7	
				2.558	99% of population > C	C = x - ks	68.3	
					99% of population < D	D = x + ks	88.3	
Average SiC thickness for each particle (µm)	29.6	0.9	220	1.652	mean ≥ A	A = x - ts/√n	29.5	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	29.7	
				2.556	99% of population > C	C = x - ks	27.3	
					99% of population < D	D = x + ks	31.9	
Average OPyC thickness for each particle (µm)	53.1	2.6	228	1.652	mean ≥ A	A = x - ts/√n	52.8	DRF-08 DRF-11
					mean ≤ B	B = x + ts/√n	53.4	
				2.551	99% of population > C	C = x - ks	46.5	
					99% of population < D	D = x + ks	59.7	
IPyC anisotropy (BAFo)	1.0058	0.0002	10	1.833	mean ≥ A	A = x - ts/√n	1.0057	DRF-18
					mean ≤ B	B = x + ts/√n	1.0059	
				3.981	99% of population > C	C = x - ks	1.0050	
					99% of population < D	D = x + ks	1.0066	
OPyC anisotropy (BAFo)	1.0056	0.0002	10	1.833	mean ≥ A	A = x - ts/√n	1.0055	DRF-18
					mean ≤ B	B = x + ts/√n	1.0057	
				3.981	99% of population > C	C = x - ks	1.0048	
					99% of population < D	D = x + ks	1.0064	
Average particle weight (mg)					mean ≥ A	A = x - ts/√n		DRF-22
					mean ≤ B	B = x + ts/√n		
					99% of population > C	C = x - ks		
					99% of population < D	D = x + ks		
OPyC open porosity (ml/m²)								DRF-31

Comments

Sample was analyzed by SEM for SiC grain structure and general microstructure of layers and interfaces.
A particle was imaged using high resolution x-ray tomography.
Particle weight and open porosity were not measured because the available sample size was insufficient for these tasks.

QC Supervisor

11-17-08

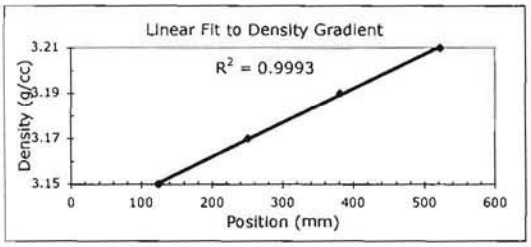
Date

Data Report Form DRF-02: Measurement of SiC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-02 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08111701_DRF02R3.xls
Sample ID:	CPT-T-G148
Sample description:	PBMR CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	10/2009
Bath temperature:	23.5 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
3.150	110.71	131.42	124.52
3.170	234.54	258.91	250.79
3.190	366.53	388.85	381.41
3.210	507.22	530.45	522.71

Linear Fit			
slope	StDev	Intercept	StDev
1.51E-04	2.66E-06	3.13E+00	8.27E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	492.25	3.2060	26	504.36	3.2078	51		
2	494.88	3.2064	27	502.87	3.2076	52		
3	496.81	3.2067	28	500.94	3.2073	53		
4	496.81	3.2067	29	499.67	3.2071	54		
5	497.04	3.2067	30	498.04	3.2069	55		
6	498.02	3.2069	31	498.14	3.2069	56		
7	498.02	3.2069	32	497.24	3.2068	57		
8	498.42	3.2069	33			58		
9	498.96	3.2070	34			59		
10	499.37	3.2071	35			60		
11	498.36	3.2069	36			61		
12	498.36	3.2069	37			62		
13	498.80	3.2070	38			63		
14	499.43	3.2071	39			64		
15	499.58	3.2071	40			65		
16	501.12	3.2073	41			66		
17	501.34	3.2074	42			67		
18	501.74	3.2074	43			68		
19	501.74	3.2074	44			69		
20	502.97	3.2076	45			70		
21	504.69	3.2079	46			71		
22	506.89	3.2082	47			72		
23	508.12	3.2084	48			73		
24	514.26	3.2093	49			74		
25	508.42	3.2084	50			75		

Average density of SiC fragments:	3.2073
Standard deviation in density of SiC fragments:	0.0007
Uncertainty in calculated density of SiC fragments:	0.0016

Dixie Barker
Operator

11-17-08
Date

Data Report Form DRF-03: Measurement of PyC Density using a Density Gradient Column

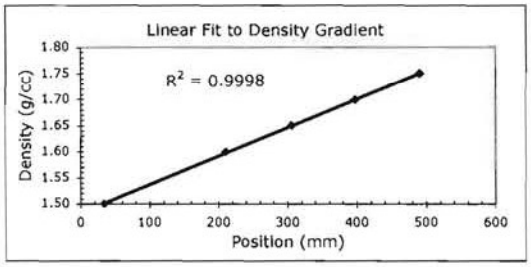
Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dizxie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08110301_DRF03R3_very low range.xls
Sample ID:	CPT-T-G148
Sample description:	PBMR CRP-6 QC Benchmark TRISO
Float expiration date:	07/2012
Gauge expiration date:	10/2009
Bath temperature:	23.6 °C

Calibrated Floats

Density	Top of Float	Bottom of Float	Center of Mass
1.500	30.93	37.22	34.08
1.600	206.81	212.96	209.89
1.650	302.55	308.88	305.72
1.700	392.89	401.87	397.38
1.750	487.93	493.80	490.87

Linear Fit

slope	StDev	intercept	StDev
5.46E-04	3.15E-06	1.48E+00	9.15E-04



Sample Density

Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	256.99	1.6233	26	279.11	1.6354	51		
2	257.20	1.6234	27	281.18	1.6365	52		
3	258.86	1.6243	28	281.75	1.6368	53		
4	261.50	1.6258	29	284.12	1.6381	54		
5	259.18	1.6245	30	284.12	1.6381	55		
6	259.93	1.6249	31	284.12	1.6381	56		
7	261.27	1.6256	32	285.87	1.6391	57		
8	261.69	1.6259	33	286.41	1.6394	58		
9	264.06	1.6272	34	287.98	1.6402	59		
10	265.38	1.6279	35	290.81	1.6418	60		
11	265.38	1.6279	36	290.81	1.6418	61		
12	267.87	1.6292	37	292.66	1.6428	62		
13	268.93	1.6298	38	293.54	1.6433	63		
14	268.93	1.6298	39	294.80	1.6439	64		
15	270.81	1.6308	40	296.02	1.6446	65		
16	270.81	1.6308	41	304.31	1.6491	66		
17	271.89	1.6314	42			67		
18	272.13	1.6316	43			68		
19	273.47	1.6323	44			69		
20	274.54	1.6329	45			70		
21	274.54	1.6329	46			71		
22	276.77	1.6341	47			72		
23	277.92	1.6347	48			73		
24	277.92	1.6347	49			74		
25	278.65	1.6351	50			75		

Average density of PyC fragments:	1.6336
Standard deviation in density of PyC fragments:	0.0067
Uncertainty in calculated density of PyC fragments:	0.0013

Dizzie Barker
Operator

11-3-08
Date

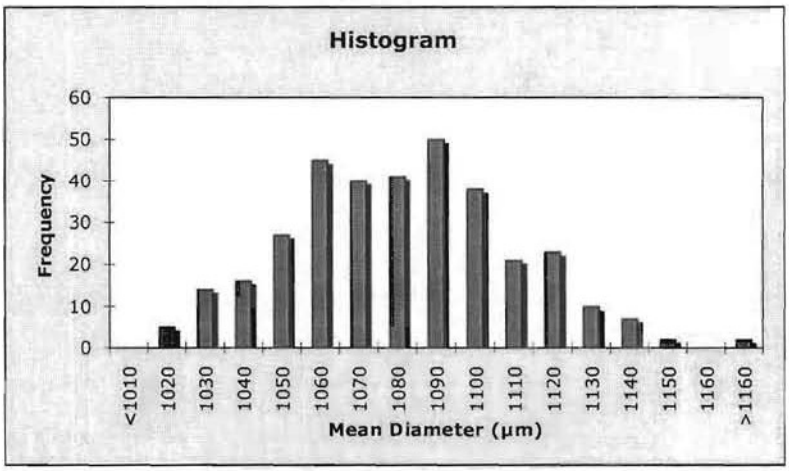
Data Report Form DRF-10A: Measurement of Particle Diameter

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08091701\
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08091701_output\

Number of particles analyzed:	341
Mean of the average diameter of each particle (μm)	1076.2
Standard deviation in the average diameter of each particle (μm):	28

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<1010	0
1020	5
1030	14
1040	16
1050	27
1060	45
1070	40
1080	41
1090	50
1100	38
1110	21
1120	23
1130	10
1140	7
1150	2
1160	0
>1160	2



Operator

September 18, 2008

Date

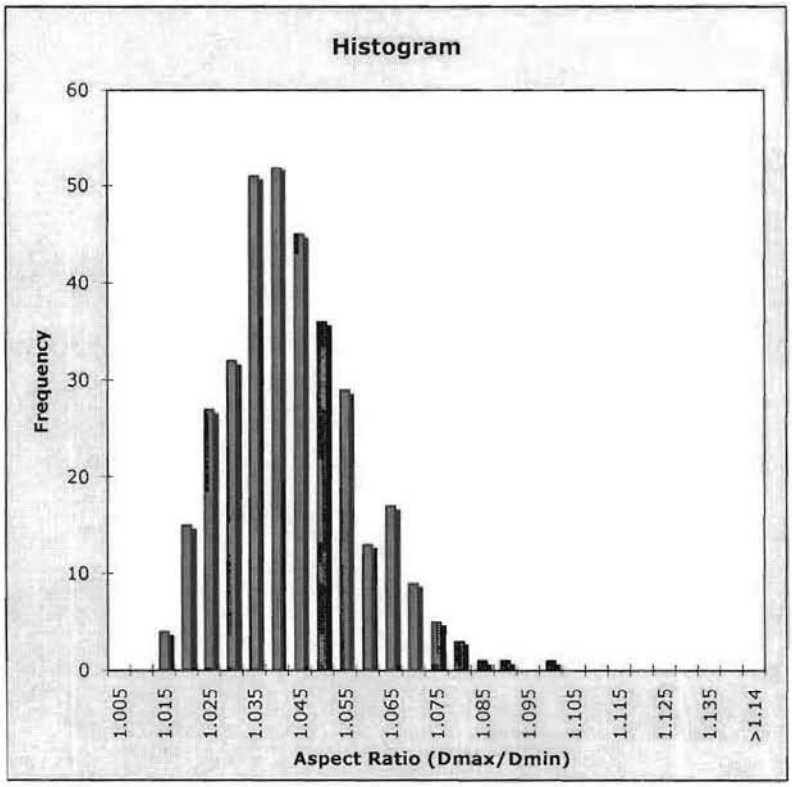
Data Report Form DRF-10B: Measurement of Particle Aspect Ratio (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08091701\
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P08091701_output\

Number of particles analyzed:	341
Number of particles with aspect ratio ≥ 1.14	0
Average particle aspect ratio:	1.041

Distribution of the aspect ratio (top binned)

Aspect Ratio (D)	*Frequency*
1.005	0
1.010	0
1.015	4
1.020	15
1.025	27
1.030	32
1.035	51
1.040	52
1.045	45
1.050	36
1.055	29
1.060	13
1.065	17
1.070	9
1.075	5
1.080	3
1.085	1
1.090	1
1.095	0
1.100	1
1.105	0
1.110	0
1.115	0
1.120	0
1.125	0
1.130	0
1.135	0
1.140	0
>1.14	0



Andrew K. Kercher
Operator

September 18, 2008
Date

Data Report Form DRF-08-10x: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	CPT-T-G148
Sample description:	PBMR CRP-6 QC Benchmark TRISO
Mount ID number:	M08091801
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08100101\P0810010101\

DMR calibration expiration date:	9/15/09
Calibrated pixels/micron:	2.2633
Stage micrometer calibration expiration date:	2/13/09
Measured value for 600 μm in stage micrometer image (μm):	599.6

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
588	535	485	445

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
260	115	78	33	53

Comments
The procedure was modified for this sample. 10x magnification was used rather than 12.5x to accommodate larger particle diameter.

Operator

October 1, 2008

Date

Data Report Form DRF-08-10x: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

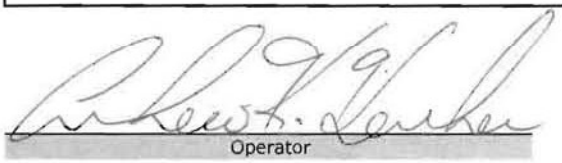
Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	CPT-T-G148
Sample description:	PBMR CRP-6 QC Benchmark TRISO
Mount ID number:	M08091802
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08100101\P0810010102\

DMR calibration expiration date:	9/15/09
Calibrated pixels/micron:	2.2633
Stage micrometer calibration expiration date:	2/13/09
Measured value for 600 μm in stage micrometer image (μm):	599.6

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
523	560	487	526

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
263	118	75	32	55

Comments
The procedure was modified for this sample. 10x magnification was used rather than 12.5x to accommodate larger particle diameter.



Operator

October 1, 2008

Date

Data Report Form DRF-08-10x: Imaging of Coated Particle Cross-sections Using an Optical Microscope System

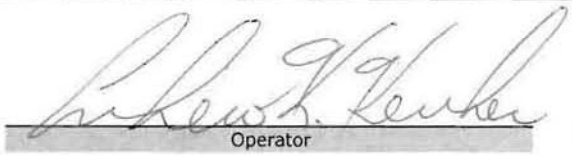
Procedure:	AGR-CHAR-DAM-08 Rev. 2
Operator:	Andrew K. Kercher
Sample ID:	CPT-T-G148
Sample description:	PBMR CRP-6 QC Benchmark TRISO
Mount ID number:	M08091803
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08100101\P0810010103\

DMR calibration expiration date:	9/15/09
Calibrated pixels/micron:	2.2633
Stage micrometer calibration expiration date:	2/13/09
Measured value for 600 μm in stage micrometer image (μm):	599.6

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
499	548	517	566

Approximate layer width in polish plane (μm)				
Kernel radius	Buffer	IPyC	SiC	OPyC
256	110	81	34	57

Comments
The procedure was modified for this sample. 10x magnification was used rather than 12.5x to accommodate larger particle diameter.



Operator

October 1, 2008

Date

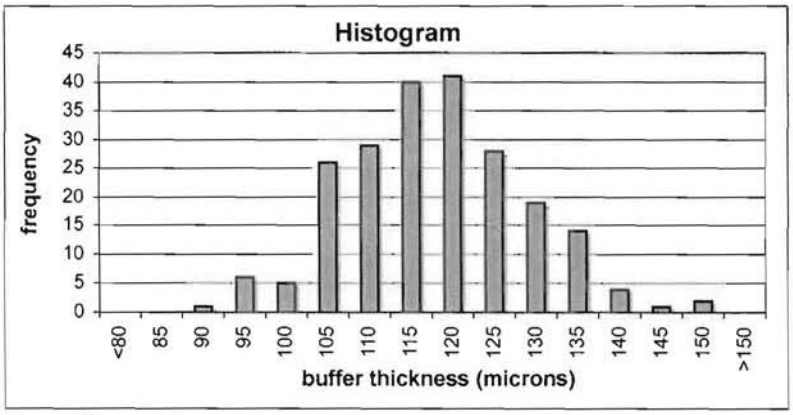
Data Report Form DRF-11A: Measurement of Buffer Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101\
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101_output\

Number of buffer layers analyzed:	216
Mean of the average buffer thickness of each particle (μm):	115.4
Standard deviation in the average buffer thickness of each particle (μm):	10.7

Distribution of the average buffer layer thickness (top binned)

Buffer Thickness (μm)	*Frequency*
<80	0
85	0
90	1
95	6
100	5
105	26
110	29
115	40
120	41
125	28
130	19
135	14
140	4
145	1
150	2
>150	0



Andrew K. Kercher
Operator

October 3, 2008
Date

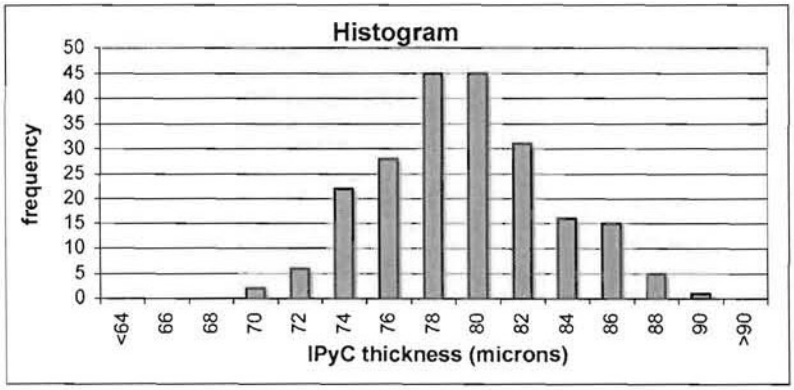
Data Report Form DRF-11B: Measurement of Inner Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101\
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101_output\

Number of inner pyrocarbon layers analyzed:	216
Mean of the average IPyC thickness of each particle (μm):	78.3
Standard deviation in the average IPyC thickness of each particle (μm):	3.9

Distribution of the average IPyC layer thickness (top binned)

IPyC Thickness (μm)	*Frequency*
<64	0
66	0
68	0
70	2
72	6
74	22
76	28
78	45
80	45
82	31
84	16
86	15
88	5
90	1
>90	0



Operator

October 3, 2008

Date

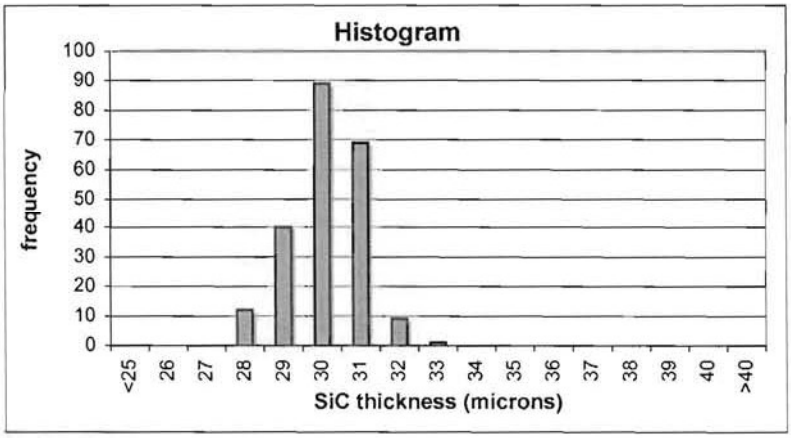
Data Report Form DRF-11C: Measurement of Silicon Carbide Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101\
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101_output\

Number of silicon carbide layers analyzed:	220
Mean of the average SiC thickness of each particle (μm):	29.6
Standard deviation in the average SiC thickness of each particle (μm):	0.9

Distribution of the average SiC layer thickness (top binned)

SiC Thickness (μm)	*Frequency*
<25	0
26	0
27	0
28	12
29	40
30	89
31	69
32	9
33	1
34	0
35	0
36	0
37	0
38	0
39	0
40	0
>40	0



Andrew K. Kercher
Operator

October 3, 2008
Date

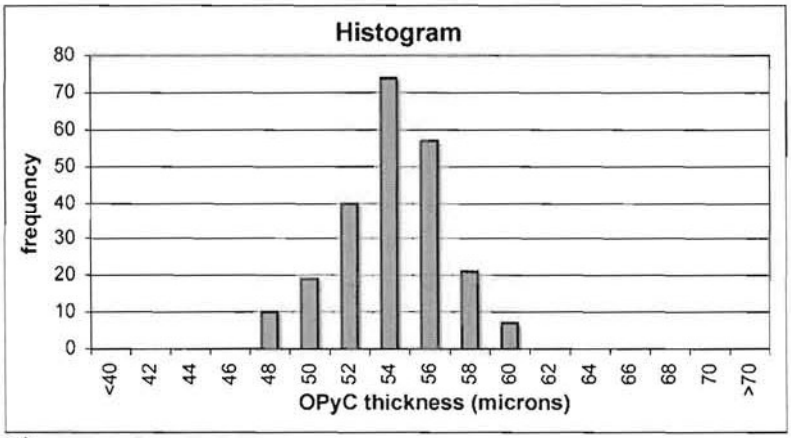
Data Report Form DRF-11D: Measurement of Outer Pyrocarbon Layer Thickness

Procedure:	AGR-CHAR-DAM-11 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101\
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100101_output\

Number of outer pyrocarbon layers analyzed:	228
Mean of the average OPyC thickness of each particle (μm):	53.1
Standard deviation in the average OPyC thickness of each particle (μm):	2.6

Distribution of the average OPyC layer thickness (top binned)

OPyC Thickness (μm)	*Frequency*
<40	0
42	0
44	0
46	0
48	10
50	19
52	40
54	74
56	57
58	21
60	7
62	0
64	0
66	0
68	0
70	0
>70	0



Andrew K. Kercher — Operator

October 3, 2008 — Date

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08100301
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08101101\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0030	0.0014	0.0005	1.0060	0.0028	0.0010
2	4,5	0.0029	0.0013	0.0005	1.0058	0.0026	0.0010
3	4,6	0.0030	0.0013	0.0006	1.0060	0.0026	0.0012
4	5,4	0.0027	0.0013	0.0005	1.0054	0.0026	0.0010
5	5,5	0.0028	0.0014	0.0005	1.0056	0.0028	0.0010
6	5,6	0.0030	0.0013	0.0005	1.0060	0.0026	0.0010
7	6,4	0.0028	0.0013	0.0006	1.0056	0.0026	0.0012
8	6,5	0.0030	0.0015	0.0005	1.0060	0.0030	0.0010
9	6,6	0.0029	0.0013	0.0005	1.0058	0.0026	0.0010
10	7,4	0.0030	0.0013	0.0005	1.0060	0.0026	0.0010
Average		0.0029	0.0013	0.0005	1.0058	0.0027	0.0010

Mean of average BAFo per particle:	1.0058
Standard deviation of average BAFo per particle:	0.0002

Comments

G. E. Jellison — Operator

10/11/08 — Date

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - OPvC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08100301
Sample ID:	CPT-T-G148
Sample Description:	PBMR CRP-6 QC Benchmark TRISO
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08101101\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0028	0.0013	0.0007	1.0056	0.0026	0.0014
2	4,5	0.0027	0.0013	0.0007	1.0054	0.0026	0.0014
3	4,6	0.0028	0.0013	0.0007	1.0056	0.0026	0.0014
4	5,4	0.0029	0.0014	0.0006	1.0058	0.0028	0.0012
5	5,5	0.0026	0.0014	0.0006	1.0052	0.0028	0.0012
6	5,6	0.0027	0.0013	0.0006	1.0054	0.0026	0.0012
7	6,4	0.0027	0.0013	0.0007	1.0054	0.0026	0.0014
8	6,5	0.0029	0.0018	0.0007	1.0058	0.0036	0.0014
9	6,6	0.0030	0.0015	0.0007	1.0060	0.0030	0.0014
10	7,4	0.0027	0.0014	0.0006	1.0054	0.0028	0.0012
Average		0.0028	0.0014	0.0007	1.0056	0.0028	0.0013

Mean of average BAFo per particle:	1.0056
Standard deviation of average BAFo per particle:	0.0002

Comments

G. E. Jellison
Operator

10/11/08
Date

A scanning electron microscope (SEM) was used to image particles polished close to midplane. The following 3 secondary electron (SE) images show the TRISO coatings and interfaces.

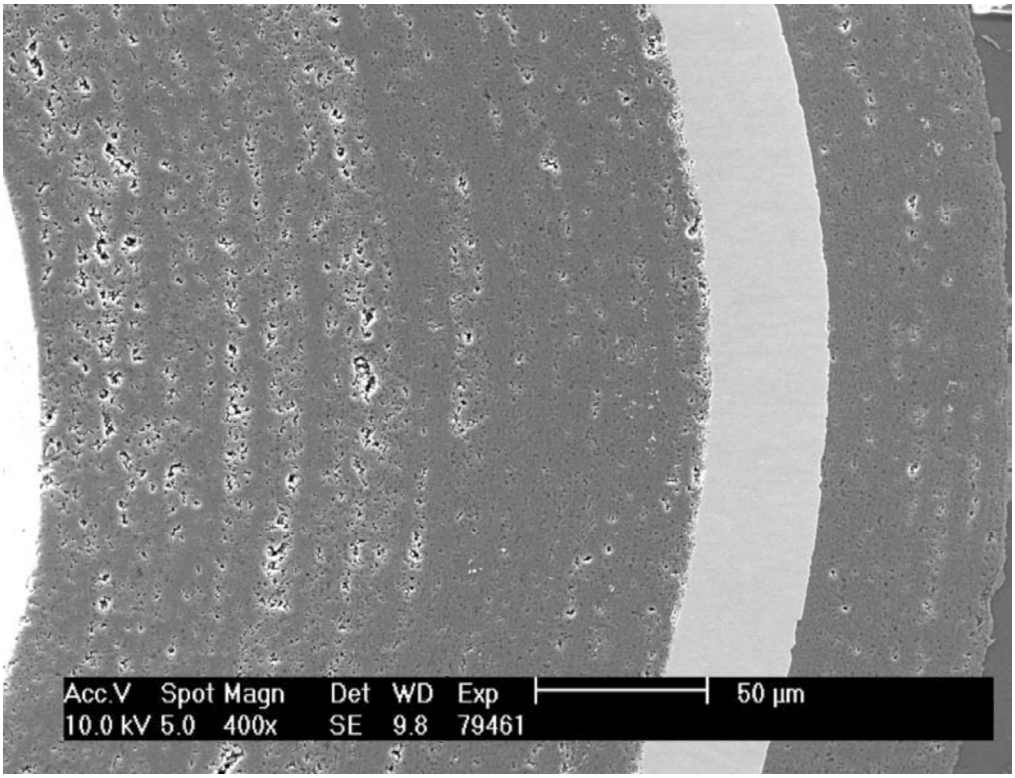


Figure 42. SE image of TRISO coatings on a typical particle.

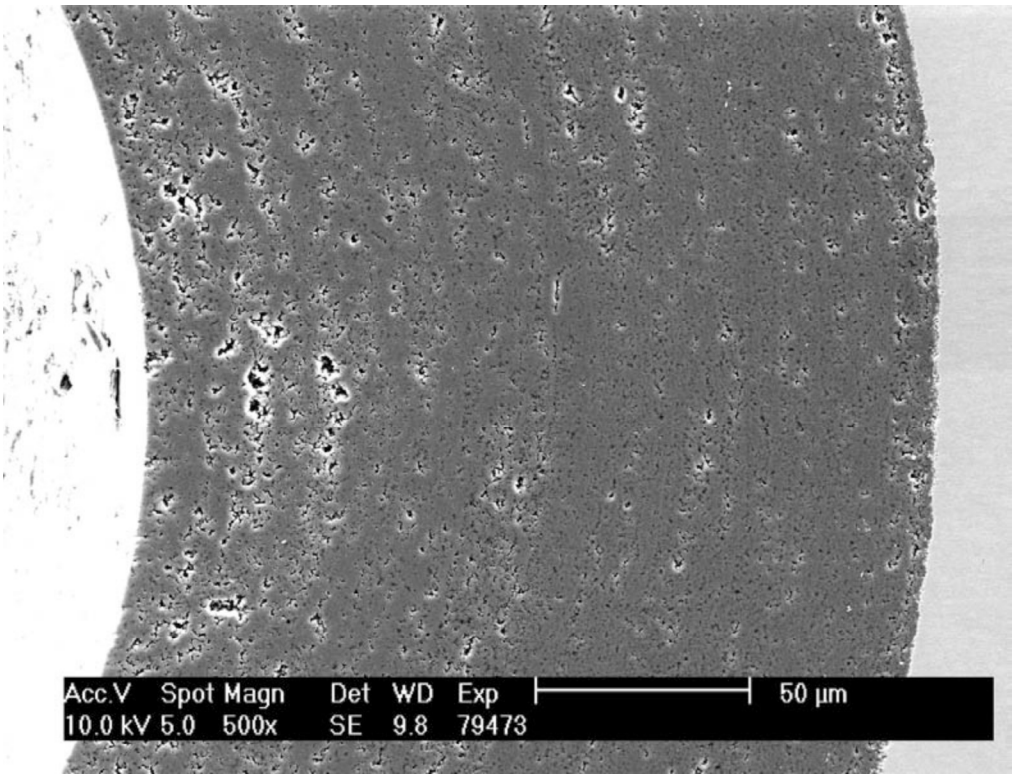


Figure 43. SE image of Buffer/IPyC interface.

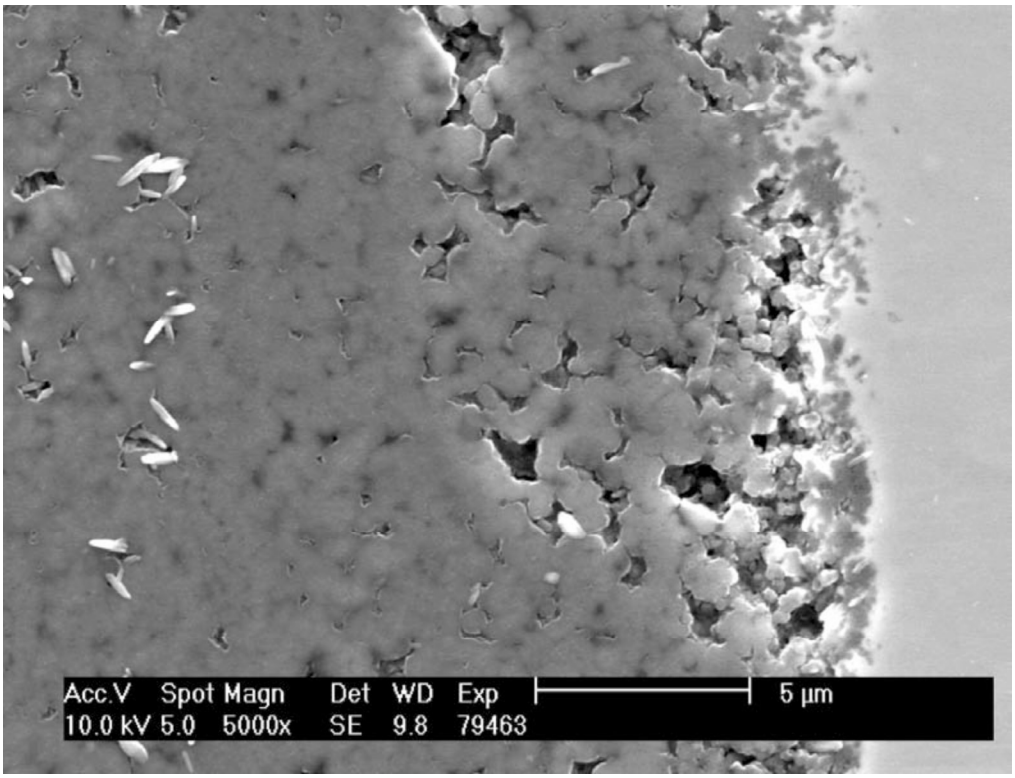


Figure 44. SE image of IPyC/SiC interface.

Backscattered electron imaging (BSE) was used to view the SiC grain structure. The following images show 3 different particles, each at two magnifications.

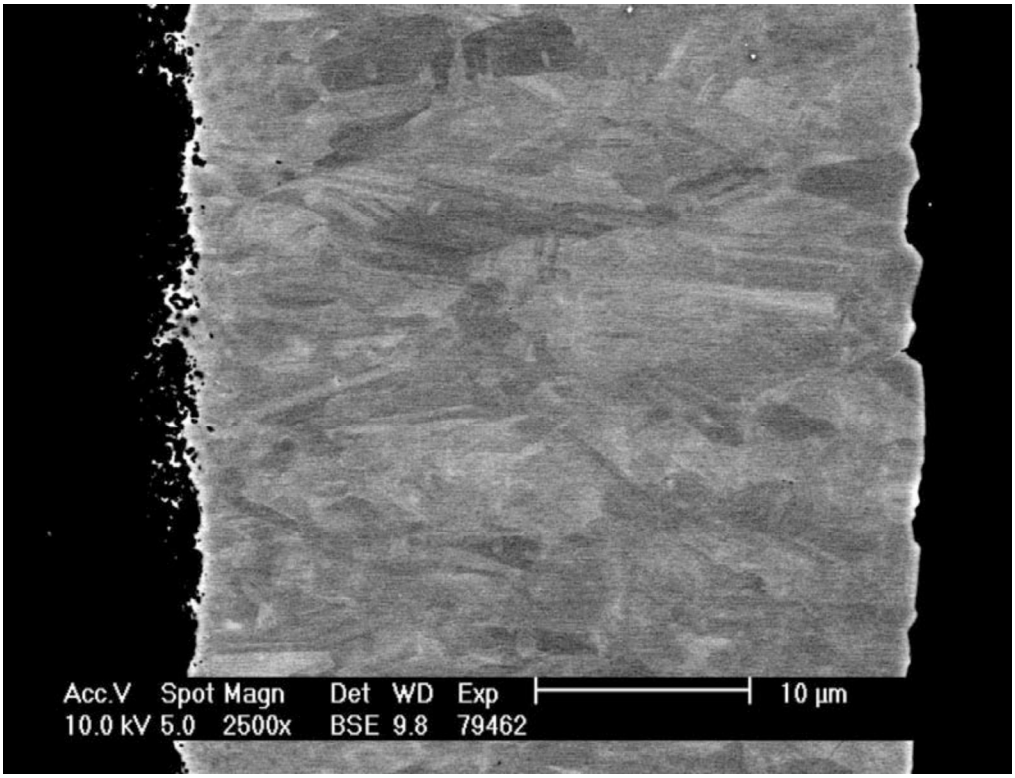


Figure 45. Particle 1 SiC grain structure shown by BSE.

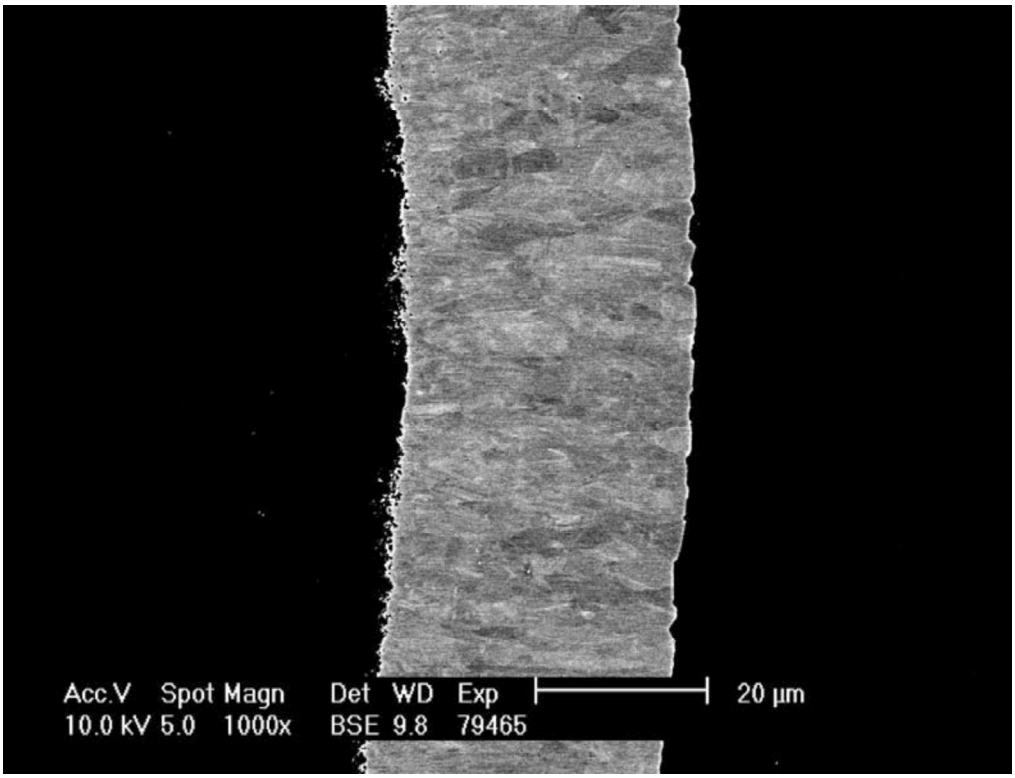


Figure 46. Particle 1 SiC grain structure shown by BSE.

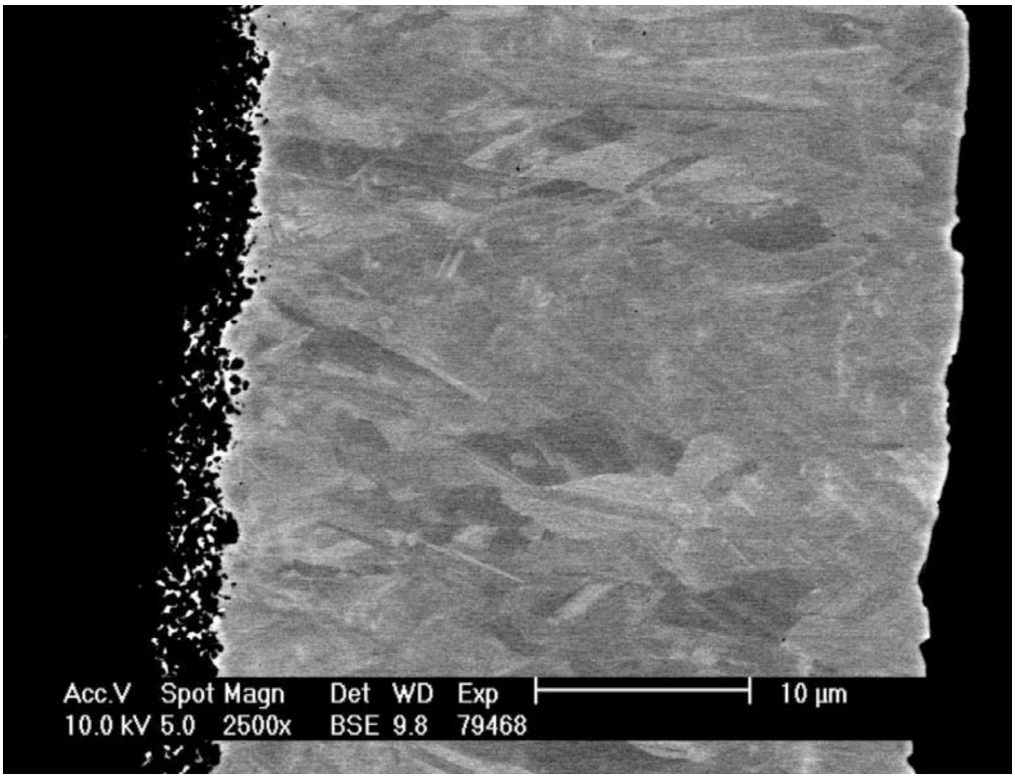


Figure 47. Particle 2 SiC grain structure shown by BSE.

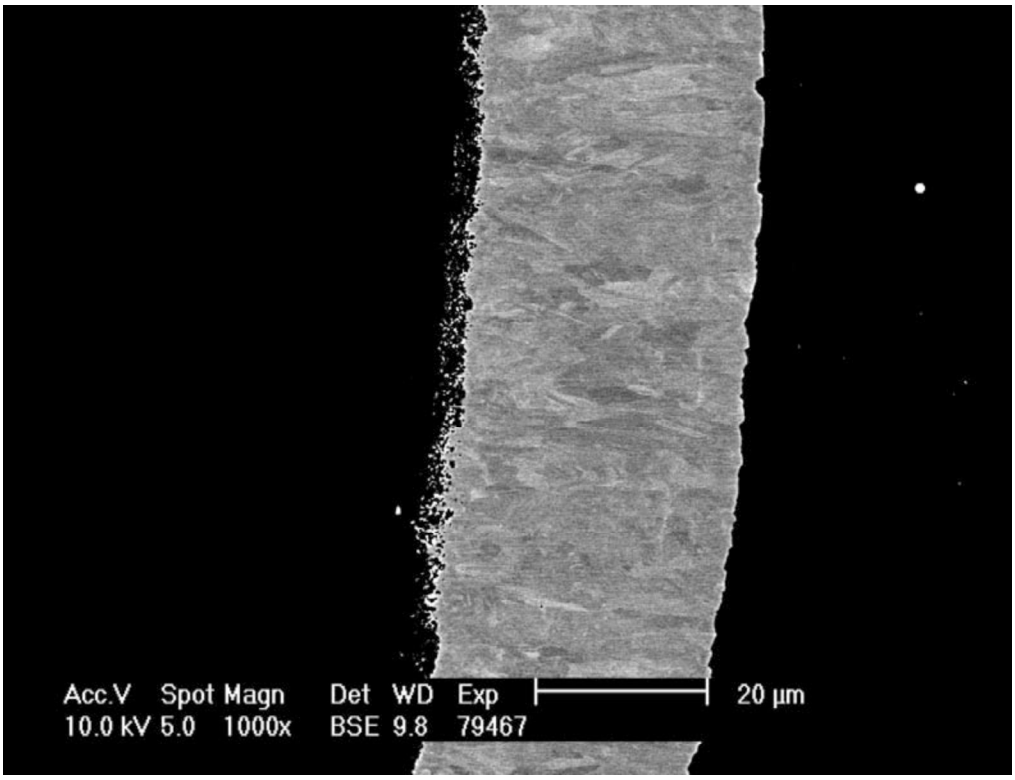


Figure 48. Particle 2 SiC grain structure shown by BSE.

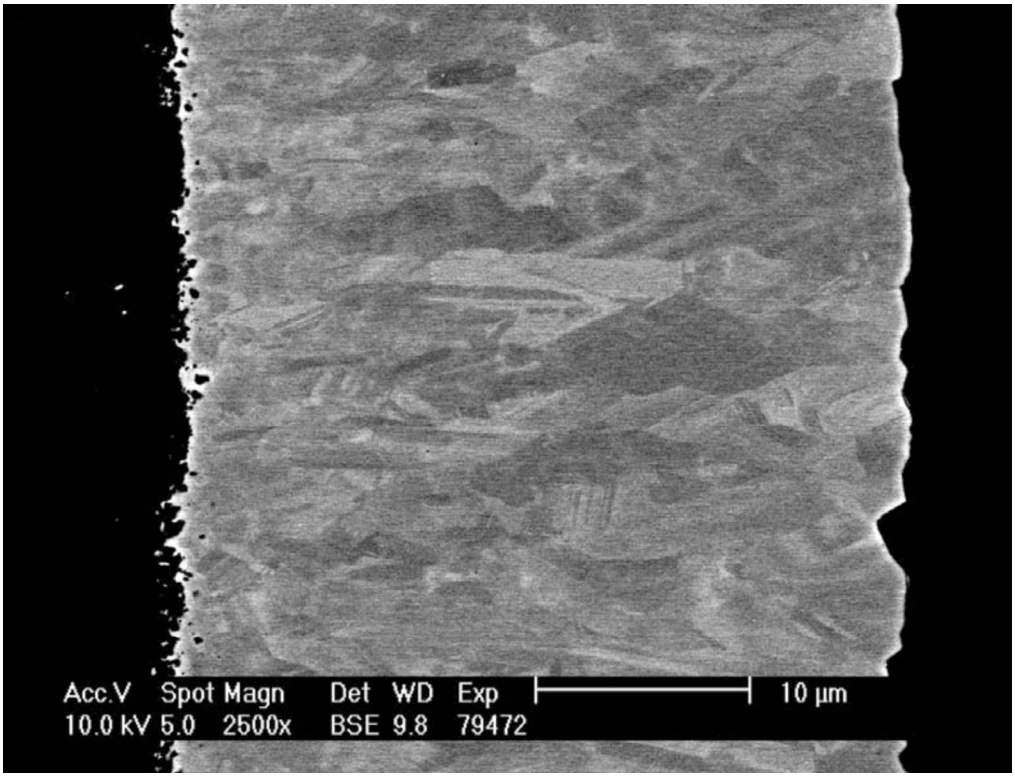


Figure 49. Particle 3 SiC grain structure shown by BSE.

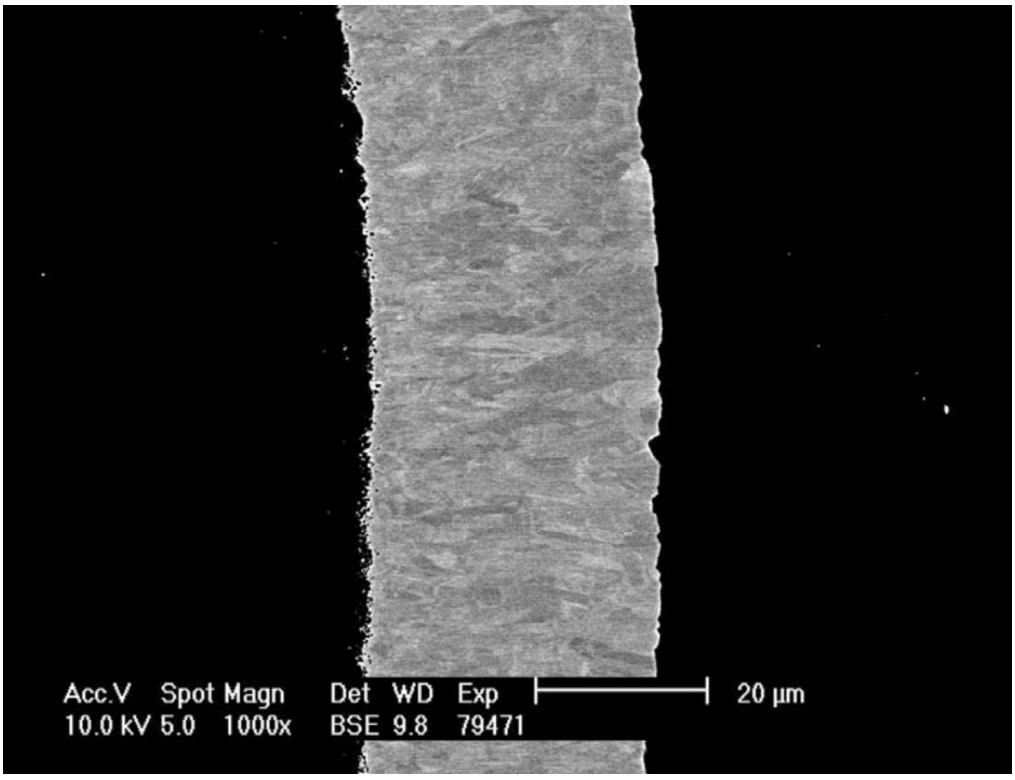


Figure 50. Particle 3 SiC grain structure shown by BSE.

One particle was selected at random and imaged using a high resolution x-ray tomograph.

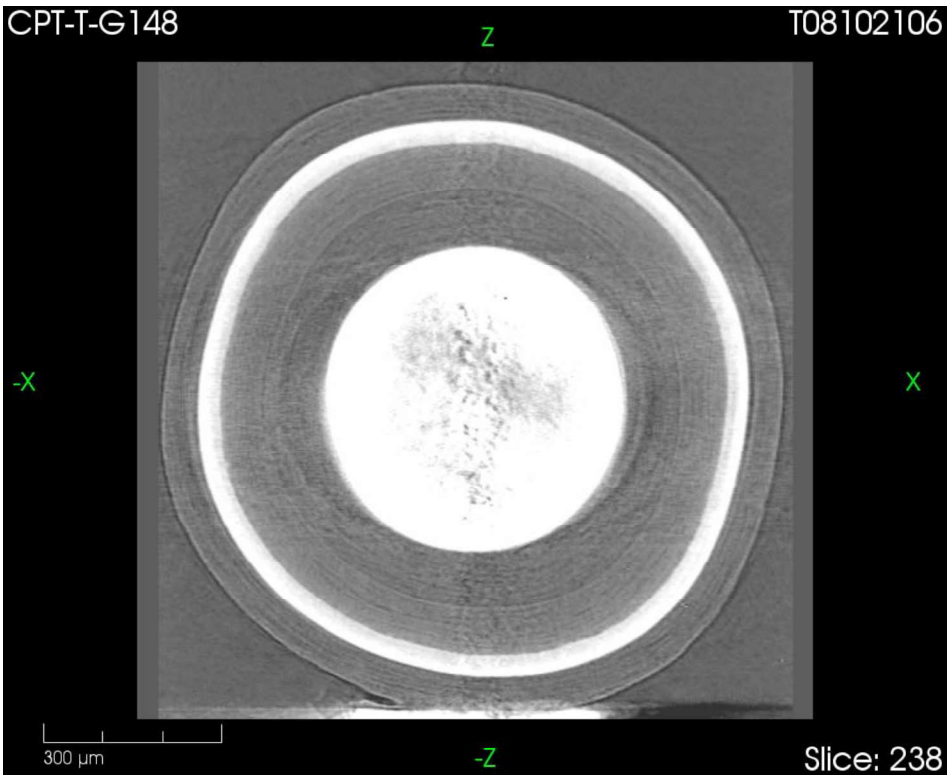


Figure 51. Tomographic cross-section of particle with rotation axis (z) in plane.

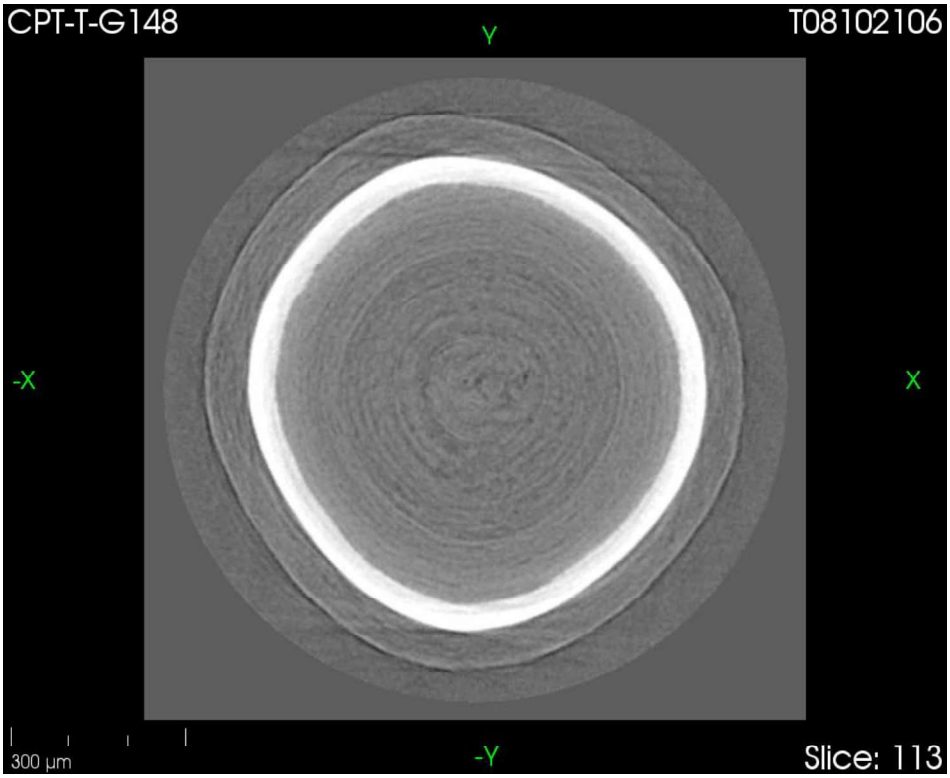


Figure 52. Tomographic cross-section perpendicular to rotation axis (z) showing a plane above kernel, where kernel effects on tomographic reconstruction are not present.

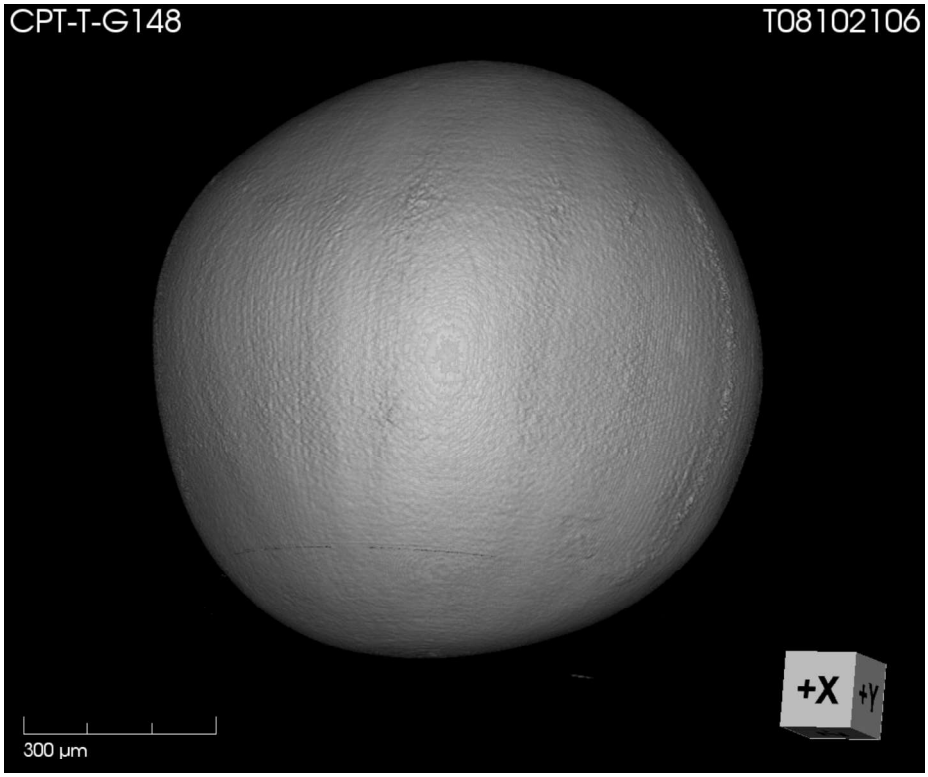


Figure 53. Tomographic 3-D visualization of SiC surface, oriented to show maximum faceting.