ORNL/TM-2017/039

Acceptance Test Data for BWXT Coated Particle Batches 93172B and 93173B

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August 2017

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

ORNL/TM-2017/039

Fusion and Materials for Nuclear Systems Division

ACCEPTANCE TEST DATA FOR BWXT COATED PARTICLE BATCHES 93172B AND 93173B

Defective IPyC Fraction and Pyrocarbon Anisotropy

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Date Published: August 2017

Work sponsored by
US DEPARTMENT OF ENERGY
Office of Nuclear Energy
under the
Advanced Gas Reactor Fuel Development and Qualification Program

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

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ACRONYMS

2-MGEM	Two-Modulator Generalized Ellipsometry Microscope
AGR	Advanced Gas Reactor (Fuel Development and Qualification Program)
AGR-5/6/7	Fifth/sixth/seventh AGR program irradiation experiments
ATR	Advanced Test Reactor
BWXT	BWX Technologies
CVD	Chemical vapor deposition
DAM	Data Acquisition Method
DRF	Data Report Form
INL	Idaho National Laboratory
IPyC	Inner pyrolytic carbon (TRISO layer)
IRF	Inspection Report Form
LBL	Leach-burn-leach
MTS	Methyltrichlorosilane
N	Diattenuation
OPTAF	Optical anisotropy factor [OPTAF=(1+N)/(1-N)]
OPyC	Outer pyrolytic carbon (TRISO layer)
ORNL	Oak Ridge National Laboratory
PIP	Product Inspection Plan
PyC	Pyrolytic carbon or pyrocarbon
QC	Quality control
SiC	Silicon carbide (TRISO layer)
TRISO	Tristructural-isotropic (coated particles)
UCO	Uranium carbide/uranium oxide mixture (fuel kernels)

ACKNOWLEDGMENTS

This work was sponsored by the U.S. Department of Energy, Office of Nuclear Energy, through the Idaho National Laboratory Advanced Reactor Technologies Technology Development Office as part of the Advanced Gas Reactor Fuel Development and Qualification Program. Special thanks to Jeff Pryor and Eric Vidal for performing x-ray radiography on particles as part of the analysis to determine the fraction of particles with defective inner pyrocarbon.

1. INTRODUCTION AND SUMMARY*

Coated particle batches J52O-16-93172B and J52O-16-93173B were produced by Babcock and Wilcox Technologies (BWXT) as part of the production campaign for the Advanced Gas Reactor Fuel Development and Qualification (AGR) Program's AGR-5/6/7 irradiation test in the Idaho National Laboratory (INL) Advanced Test Reactor (ATR), but were not used in the final fuel composite. However, these batches may be used as demonstration production-scale coated particle fuel for other experiments. Each batch was coated in a 150-mm-diameter production-scale fluidized-bed chemical vapor deposition (CVD) furnace. Tristructural isotropic (TRISO) coatings were deposited on 425-µm-nominal-diameter spherical kernels from BWXT lot J52R-16-69317 containing a mixture of 15.5%-enriched uranium carbide and uranium oxide (UCO). The TRISO coatings consisted of four consecutive CVD layers: a ~50% dense carbon buffer layer with 100-µm-nominal thickness, a dense inner pyrolytic carbon (IPyC) layer with 40-µm-nominal thickness, a silicon carbide (SiC) layer with 35-µm-nominal thickness, and a dense outer pyrolytic carbon (OPyC) layer with 40-µm-nominal thickness. The TRISO-coated particle batches were sieved to upgrade the particles by removing over-sized and under-sized material, and the upgraded batches were designated by appending the letter A to the end of the batch number (e.g., 93172A). Secondary upgrading by sieving was performed on the A-designated batches to remove particles with missing or very-thin buffer layers that were identified during previous analysis of the individual batches for defective IPyC, as reported in the acceptance test data report for the AGR-5/6/7 production batches [Hunn et al. 2017b]. The additionally-upgraded batches were designated by appending the letter B to the end of the batch number (e.g., 93172B).

Two samples each were riffled from upgraded TRISO batches 93172B and 93173B and shipped to the Oak Ridge National Laboratory (ORNL) for quality control (QC) acceptance testing and analysis. The AGR-5/6/7 Fuel Specification, SPC-1352 [Marshall 2016], provides the requirements necessary for acceptance of the fuel manufactured for the AGR-5/6/7 irradiation test. The kernel QC acceptance testing and most of the coated particles QC acceptance testing was performed at BWXT and is not contained in this report. Two specified TRISO particle properties were measured at ORNL: pyrolytic carbon (PyC) anisotropy and defective IPyC fraction. The procedures for the ORNL characterization and QC acceptance testing of the particles are outlined in the ORNL Product Inspection Plan for AGR-5/6/7 Coated Particles, AGR-CHAR-PIP-28 [Hunn 2016], which is consistent with the INL Statistical Sampling Plan for AGR-5/6/7 Fuel Materials, PLN-4352 [Lybeck 2016].

Particles with excessive IPyC permeability can allow the infiltration of HCl into the buffer region of a TRISO particle during the initial stages of SiC deposition. HCl is a byproduct of the SiC CVD process when using hydrogen (H_2) and methyltrichlorosilane (MTS) precursors. This HCl can react with the kernel and disperse uranium into the surrounding buffer and IPyC layers, especially when particles are heated to 1800°C during the compact manufacturing process. Excessive uranium dispersion can be detected by x-ray radiography of the TRISO-coated particles because the higher relative x-ray absorption of uranium versus carbon makes it easy to detect small concentrations of uranium in the buffer and IPyC layers. Visual standards for what constitutes excessive uranium dispersion are included in the AGR-5/6/7 Fuel Specification, and particles that exhibit excessive uranium dispersion are counted as having a defective IPyC coating. Data Acquisition Method AGR-CHAR-DAM-47, Counting of TRISO Particles with Excessive Uranium Dispersion Inside SiC [Hunn 2013], provides the detailed procedures and requirements for the analysis that was performed to determine the defect IPyC fraction.

Prior to x-ray imaging for determination of defective IPyC based on the presence of excessive uranium dispersion, data acquisition method AGR-CHAR-DAM-41 [Kercher 2010] was performed to simulate

* Background content in this introduction section also appeared in a report on similar analysis of other AGR-5/6/7 campaign batches [Hunn et al. 2017b].

compact heat treatment and induce uranium dispersion in particles with defective IPyC. The AGR-5/6/7 Fuel Specification specifies heat treatment of the compacts for at least 1 h between 1650°C and 1800°C. Particles were heated with the furnace schedule used for the compacts produced for the previous three AGR irradiation experiments (~20°C/min ramping and a one-hour hold at 1800°C). The loose particles were heated in a bed of graphite powder to minimize stress from temperature gradients.

Pyrocarbon anisotropy is a key parameter that can influence the radiation behavior of the IPyC and OPyC layers in TRISO-coated particle fuel. Excessive preferred orientation of the graphene planes within the pyrocarbon layers can lead to overall asymmetric shrinkage and fracture under irradiation. Because of the very large anisotropy for the reflection of light polarized parallel to the graphene planes versus light polarized perpendicular to the graphene planes, determination of the PyC optical anisotropy (OPTAF), defined as the ratio of the maximum to minimum reflectivity of polarized light, can be used as a relative measure of the preferred orientation of the graphene planes within the layer. The ORNL Two-Modulator Generalized Ellipsometry Microscope (2-MGEM) uses advanced ellipsometry techniques to measure the diattenuation (N) of a material, which is related the optical anisotropy by OPTAF = (1+N)/(1-N) [Jellison and Hunn 2008]. Data Acquisition Method AGR-CHAR-DAM-18, Measurement of Pyrocarbon Anisotropy Using the Second Generation Two-Modulator Generalized Ellipsometry Microscope [Hunn and Jellison 2016], provides the detailed procedures and requirements for the analysis that was performed to determine the optical anisotropy of the IPyC and OPyC layers.

Results of the determination of defective IPyC fraction and average IPyC and OPyC anisotropy are reported for Batches 93172B and 93173B in Sections 2 and 3, respectively. Table 1-1 is a summary of the results. Both analyzed batches satisfied the AGR-5/6/7 Fuel Specification for pyrocarbon anisotropy, with average diattenuation values below the specified upper limits of N≤0.0170 for the IPyC layer and N≤0.0122 for the OPyC layer. The higher allowable IPyC diattenuation is related to the fact that pyrocarbon anisotropy is measured after all TRISO coatings are deposited. During SiC deposition, the IPyC layer is heated to around 1550°C for over 2 h; this heat treatment after pyrocarbon deposition at lower temperatures increases the average anisotropy of the layer [Hunn et al. 2007]. Further increase in the average pyrocarbon anisotropy can be expected when compacts are heat treated to even higher temperatures. For example, after heating particles to 1800°C to simulate compacting as described above, the average anisotropy of the both the IPyC and OPyC layers increased, as shown in Table 1-1.

Table 1-1. Summary of pyrocarbon anisotropy for coated particle batches 93172B and 93173B

Coating Batch	Average anisotropy of as-deposited TRISO		Average anisotropy after heating to 1800°C	
	IPyC	OPyC	IPyC	OPyC
93172B	N=0.0154 OPTAF=1.0313	N=0.0099 OPTAF=1.0199	N=0.0177 OPTAF=1.0361	N=0.0138 OPTAF=1.0279
93173B	N=0.0139 OPTAF=1.0282	N=0.0098 OPTAF=1.0198	N=0.0174 OPTAF=1.0353	N=0.0136 OPTAF=1.0276

The upper limit on the defective IPyC fraction is specified as $\leq 10^{-4}$ with a requirement that statistical sampling demonstrate with at least 95% confidence that the batch has a defect fraction less than this limit. Acceptance testing was performed by riffling, per PIP-28, two random subsamples from each batch with the appropriate number of particles to apply two predetermined acceptance criteria derived using binomial distribution statistics. The Stage 1 acceptance criteria was ≤2 defects in a random group of at least 62956 particles. The Stage 2 acceptance criteria was ≤6 defects in a random group of at least 118422 particles. The analysis results from the first riffled subsample were used for Stage 1, and the combined results from both subsamples were used for Stage 2. Target weights for the riffled subsamples were determined based on the average particle weight, with a sufficient margin based on the uncertainty in the average particle

weight to ensure the subsamples provided at least the required minimum number of particles, while minimizing overshoot. Minimizing overshoot is important because the probability that a sublot with an acceptable defect population will satisfy the acceptance criteria decreases as a function of increasing difference between the actual number of particles analyzed and the minimum required. The exact number of particles in each subsample was determined by counting the particles in the x-ray radiographs acquired for the defective IPyC analysis. As shown in Table 1-2, both coating batches passed the Stage 2 criteria.

Table 1-2. Summary of Defective IPyC for coated particle batches 93172B and 93173B

Coating Batch	Number of particles in analyzed sample	Number of particles with Defective IPyC	Measured defect fraction in sample	Max defect fraction at 95% confidence
93172B	120048	3 (pass)	2.50×10^{-5}	6.46×10^{-5}
93173B	120897	1 (pass)	8.27×10^{-6}	3.93×10^{-5}

The careful examination of the x-ray radiographs required for determination of defective IPyC fraction provided an opportunity to also inspect the 120,000-particle samples for other microstructural anomalies. A supplemental data report form (DRF-47 Supplemental) is included in Sections 2 and 3 for each sample analyzed for defective IPyC where the presence of these anomalies is recorded. One anomaly of particular interest was the presence of particles with a missing or very-thin buffer layer; this anomaly was observed in some of the batches selected for the AGR-5/6/7 irradiation test composite [Hunn et al. 2017b]. Due to the concern that particles with missing or very-thin buffer may be more likely to fail during irradiation, a decision was made to upgrade these batches by performing additional sieving to reduce the population of particles with this anomaly. A sample of particles from Batch 93172A (prior to secondary sieving) was found to have 4 particles with very-thin or missing buffer out of 121383 surveyed [Hunn et al. 2017b]. No particles with very-thin or missing buffer were observed in the 120,048-particle sample from Batch 93172B, indicating the effectiveness of the secondary sieving. Similarly, Batch 93173B had no particles with very-thin or missing buffer. A few particles with moderately-thin (~50-µm-thick) buffer were noted during the inspection, like that shown in Figure 1-1. These particles were slightly undersized but apparently not enough to be removed by the sieving process. The presence of these particle is less of a concern with regard to irradiation performance compared to those with very-thin or missing buffer.

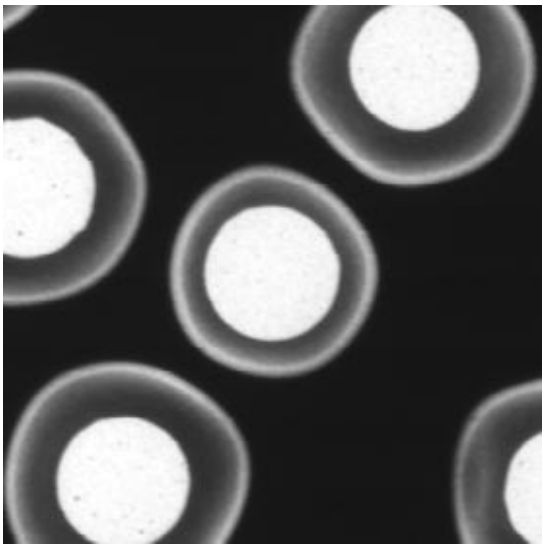


Figure 1-1. X-ray radiograph of particles from Batch 93173B subsample NP-C1533-D01 showing a particle with a moderately-thin buffer layer.

Other anomalies that were specifically looked for and quantified were missing kernels, thin-appearing SiC, extra coating layers, various non-spherical kernel shapes, white spots in the radiographs not obviously related to uranium dispersion, and kernel migration from excessive kernel-buffer interaction at 1800°C due to CO_2 release through cracked TRISO coatings. White spots were typically related to debris on the particles surface. Particles with cracked TRISO coatings are identified as defective fuel particles in the AGR-5/6/7 Fuel Specification but defect fraction determination and acceptance criteria are based on the leach-burn-leach (LBL) analysis method, so data obtained by x-ray radiography is for information-only. Nevertheless, the x-ray imaging provided valuable additional information on these defects and the fraction of particles observed compared well with the official defect fraction values acquired with LBL. Particles with defective IPyC and example particles exhibiting the other anomalies were extracted from the Kapton tape holders used for radiography, and these particles were subjected to additional analysis by higher-resolution x-ray tomography. Results of the x-ray analysis of similar defects and anomalies found in previously observed samples are presented and discussed in greater detail in a separate report [Helmreich et al. 2017a].

Similar analyses of pyrocarbon anisotropy, defective IPyC, and microstructural anomalies have been previously reported on a sample riffled from a BWXT pre-production batch of coated particles [Hunn et al. 2017a], samples riffled from BWXT production batches of coated particles [Hunn et al. 2017b], and the BWXT irradiation test fuel composite particles [Helmreich et al. 2017b].

2. BATCH 93172B SAMPLE 1517

Sample NP-C1517 was a 130-gram sample riffled by BWXT from upgraded TRISO batch J52R-16-93172B and shipped to ORNL for QC acceptance testing and analysis.

2.1 BATCH 93172B SAMPLE 1517: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93172B Sample NP-C1517. Subsamples were riffled at ORNL according to the sampling instructions in Product Inspection Plan PIP-28. The combined number of particles with defective IPyC in these two subsamples is reported on Inspection Report Form IRF-28A (Figure 2-1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93172B Sample NP-C1517 meets the AGR-5/6/7 Fuel Specification requirements for the maximum defective IPyC fraction.

Inspection Report Form IRF-28A: AGR-5/6/7 Coated Particles

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1517
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B

Property	Measured Data # of particles	Specification INL SPC-1352	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
Defective IPyC coating fraction (fraction of total particles)	120048	≤ 1.0×10^{-4}	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	3	pass	DRF-47

Comments

See NP-C1517-C01_DRF47R0 and NP-C1517-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography.
Sample also passed Stage 1 testing with 2 particles with excessive U dispersion out of 62991.

QC Supervisor — Date: 8-2-2017

QA Reviewer — Date: 8/8/17

Figure 2-1. Inspection report for Batch 93172B Sample NP-C1517 defective IPyC.

Figure 2-2 through Figure 2-7 are copies of the data report forms generated as part of the completion of Product Inspection Plan PIP-28. Figure 2-2 is the particle weight determination used to ensure that each defect IPyC subsample had sufficient particles to meet the two acceptance test stages called out in the Statistical Sampling Plan for AGR-5/6/7 Fuel Materials. The minimum particle number requirements for this two-stage sampling appear in the acceptance criteria column in IRF-28A (Figure 2-1). Figure 2-3 is a record of the conditions of the particle heat treatment procedure. Figure 2-4 and Figure 2-6 are the individual results of the defective IPyC analysis for the two subsamples; these Data Report Forms (DRF-47) document the number of particles tested in each subsample and the number of particles counted as having defective IPyC based on their exhibition of excessive uranium dispersion. Figure 2-5 and Figure 2-7 are the associated DRF-47 supplemental data forms for the two analyzed subsamples and document the number of particles that had other anomalies of interest visible in the single x-ray radiograph image acquired of each particle. The supplemental data forms also report the fraction of particles in each subsample that exhibited each anomaly and a 95%-confidence prediction of the

maximum fraction in the TRISO particle batch, based on the observed number, the subsample size, and using binomial distribution statistics. Details about these anomalies and additional images of similar particles acquired by high-resolution x-ray tomography to further characterize them, as well as the uranium dispersion in the particles with defective IPyC, are available in a separate summary report [Helmreich et al. 2017a].

A unique anomaly was observed in Batch 93172B Sample NP-C1517-D01. Nineteen particles were identified to have a small amount of high-Z material dispersed around the outside of the particle. High-resolution x-ray tomography of three of these particles confirmed that the material was present only on the exterior of the OPyC layer, as shown in Figure 2-8. The location of the high-Z material on the exterior of the particles along with the fact that this anomaly was observed only in this subsample strongly suggests that these particles were the result of contamination during the sample preparation process. This contamination must have occurred after riffling, as there were no corresponding anomalies in the other subsample from this material batch, and is most likely to have occurred during heat treatment, possibly due to broken particles resulting in exposed kernels within the material batch. Uranium from exposed kernels could migrate and react with the OPyC layer during the 1800°C heat treatment.

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Grant Helmreich
Particle sample ID:	NP-C1517
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B
Filename:	\\mc-agr\AGR\ParticleWeight\W17051701_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1298	0.1142	0.1415	0.1704	0.1627
Number of particles:	124	109	135	162	156
Average weight/particle (g):	1.047E-03	1.048E-03	1.048E-03	1.052E-03	1.043E-03

Mean average weight/particle (g):	1.047E-03
Standard error in mean average weight/particle (g):	1.43E-06

Operator

7/28/17

Date

Figure 2-2. Data report for Batch 93172B Sample NP-C1517 average particle weight measured for subsample riffling.

Data Report Form DRF-41: Heat-treatment of Loose Particles Using a Graphite Furance

Procedure:	AGR-CHAR-DAM-41 Rev. 0
Operator:	John Hunn/Darren Skitt
DRF filename:	\\mc-agr\AGR\Furnaces\H17052201_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28R1				
Particle weight (g)	125.9521 g	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93172B				

Furnace calibration due date	10/13/2017

Thermal schedule				
Ramp 1	20	°C/min to	1800	°C
Dwell 1	1		hr	
Ramp 2	-20	°C/min to	700	°C
Dwell 2	0		hr	
Ramp 3	N/A	°C/min to	50	°C
Dwell 3	0		hr	
Ramp 4		°C/min to		°C
Dwell 4			hr	

Sample loading (top to bottom)			
Sample ID	NP-C1517-C01 (66.0683 g)	Crucible Marking	X
Sample ID	NP-C1517-D01 (59.8838 g)	Crucible Marking	Y
Sample ID		Crucible Marking	
Sample ID		Crucible Marking	
Sample ID		Crucible Marking	

3 vacuum / gas purges	1	2	3
Heat-treatment atmosphere	Vacuum		
Flow rate	N/A		

Comments

Ran furnace in Yokogawa auto mode. Controlled setpoint between 1770-1775°C to hold 1800°C on optical pyrometer.
Optical pyrometer calibration due 5/12/2018.

Operator

5-22-17

Date

Figure 2-3. Data report for Batch 93172B Sample NP-C1517 particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Indicating Defective IPyC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Austin Schumacher / Darren Skitt / Grant Helmreich
Particle sample ID:	NP-C1517-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052301\NP-C1517-C01_DRF47R0.xlsm

Weight of particles in sample (g):	66.0683
Number of particles in sample:	62991
Average weight/particle (g):	1.0489E-03

Number of particles identified as having Defective IPyC:	2

Comments

Tape mounts T17052301 thru T17052319.

Operator

8/14/17

Date

Figure 2-4. Data report for defective IPyC analysis of Batch 93172B subsample NP-C1517-C01.

Data Report Form DRF-47 Supplemental: Counting of Defects and Anomalies by Radiography

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Austin Schumacher / Darren Skitt / Grant Helmreich
Particle sample ID:	NP-C1517-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052301\NP-C1517-C01_DRF47R0.xlsm

Number of particles in sample:	62991
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	2	3.18E-05	1.0E-04
Defective-IPyC related	*2*	*3.18E-05*	*1.0E-04*
Fractured-kernel related	*0*	*0.00E+00*	*4.8E-05*
White spots	4	6.35E-05	1.5E-04
Thin or low density SiC	2	3.18E-05	1.0E-04
Extra layers	0	0.00E+00	4.8E-05
Missing kernel	0	0.00E+00	4.8E-05
Kernel migration	0	0.00E+00	4.8E-05
Missing buffer	0	0.00E+00	4.8E-05
Particles with kernel anomalies	775	1.23E-02	1.4E-02
Dimple or facet	*668*	*1.06E-02*	*1.2E-02*
Severe dimple or facet	*39*	*6.19E-04*	*8.1E-04*
Notched kernel	*13*	*2.06E-04*	*3.3E-04*
Irregular kernel	*62*	*9.84E-04*	*1.3E-03*
Multi-kernel	*0*	*0.00E+00*	*4.8E-05*

Comments

Tape mounts T17052301 thru T17052319.

Operator

8/14/17

Date

Figure 2-5. Summary of anomalies observed during defective IPyC analysis of Batch 93172B subsample NP-C1517-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Indicating Defective IPyC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich / Austin Schumacher
Particle sample ID:	NP-C1517-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052325\NP-C12517-D01_DRF47R0.xlsm

Weight of particles in sample (g):	59.8838
Number of particles in sample:	57057
Average weight/particle (g):	1.0495E-03

Number of particles identified as having Defective IPyC:	1

Comments

Tape mounts T17052320 thru T17052336.

Two additional particles were identified with uranium dispersion that was not caused by defective IPyC. Both were determined to be associated with kernel debris trapped in the buffer layer near the kernel surface.

One additional particle was identified to have a large kernel fragment embedded in the OPyC layer.

Nineteen particles were identified to have a dusting of high-Z material around the surface of the OPyC. This was observed only in this one particle sample, and it was concluded to be caused by contamination of the crucible used in heat treatment.

Operator

8/14/18

Date

Figure 2-6. Data report for defective IPyC analysis of Batch 93172B subsample NP-C1517-D01.

Data Report Form DRF-47 Supplemental: Counting of Defects and Anomalies by Radiography

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich / Austin Schumacher
Particle sample ID:	NP-C1517-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052325\NP-C12517-D01_DRF47R0.xlsm

Number of particles in sample:	57057
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	3	5.26E-05	1.4E-04
Defective-IPyC related	*1*	*1.75E-05*	*8.4E-05*
Fractured-kernel related	*2*	*3.51E-05*	*1.2E-04*
White spots	9	1.58E-04	2.8E-04
Thin or low density SiC	3	5.26E-05	1.4E-04
Extra layers	0	0.00E+00	5.3E-05
Missing kernel	0	0.00E+00	5.3E-05
Kernel migration	0	0.00E+00	5.3E-05
Missing buffer	0	0.00E+00	5.3E-05
Particles with kernel anomalies	1373	2.41E-02	2.6E-02
Dimple or facet	*1247*	*2.19E-02*	*2.3E-02*
Severe dimple or facet	*29*	*5.08E-04*	*7.0E-04*
Notched kernel	*32*	*5.61E-04*	*7.6E-04*
Irregular kernel	*81*	*1.42E-03*	*1.8E-03*
Multi-kernel	*1*	*1.75E-05*	*8.4E-05*

Comments

Tape mounts T17052320 thru T17052336.

Two particles were identified with uranium dispersion that was not caused by defective IPyC. Both were determined to be assoiciated with kernel debris trapped in the buffer layer near the kernel surface.

One additional particle was identified to have a large kernel fragment embedded in the OPyC layer.

Nineteen particles were identified to have a dusting of high-Z material around the surface of the OPyC. This was observed only in this one particle sample, and it was concluded to be caused by contamination of the crucible used in heat treatment.

Operator

8/14/17

Date

Figure 2-7. Summary of anomalies observed during defective IPyC analysis of Batch 93172B subsample NP-C1517-D01.

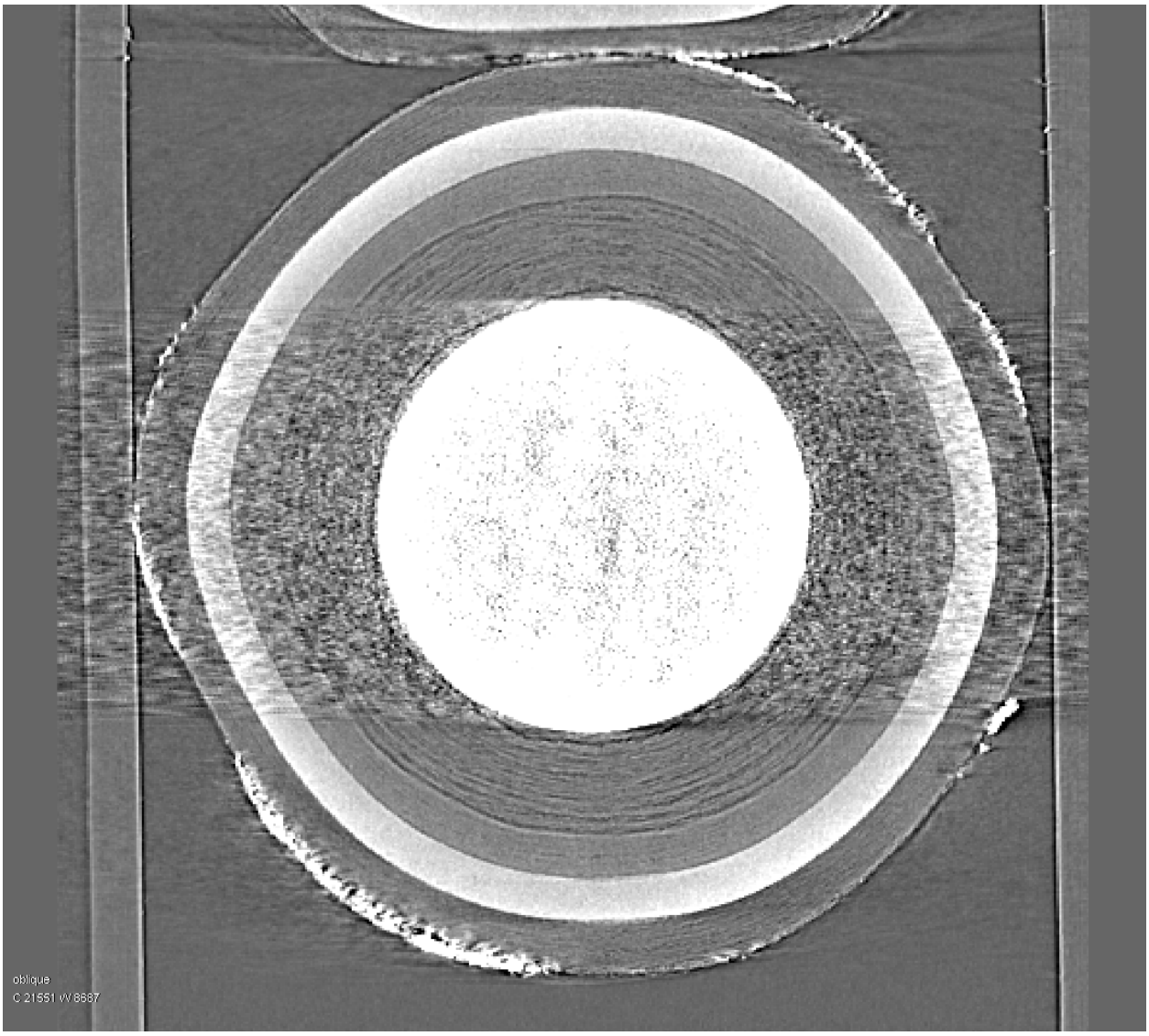


Figure 2-8. High-resolution x-ray tomographic cross section of a particle from Batch 93172B subsample NP-C1517-D01 with high-Z contamination on the outer surface of the OPyC layer, presumed to have occurred during heat treatment.

2.2 BATCH 93172B SAMPLE 1517: PYROCARBON ANISOTROPY

Average optical anisotropies of the IPyC and OPyC layers were measured on polished cross sections of 10 particles from a nominally 0.15-g subsample riffled at ORNL from TRISO Batch 93172B Sample NP-C1517 according to the sampling instructions in Product Inspection Plan PIP-28. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 2-9) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93172B meets the AGR-5/6/7 Fuel Specification requirements for the IPyC and OPyC diattenuation.

Inspection Report Form IRF-28B: AGR-5/6/7 Coated Particles

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1517
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172B

Property	Measured Data			k or t value	Specification INL SPC-923	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)						
IPyC diattenuation	0.0154	0.0006	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.016	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.018	pass	
OPyC diattenuation	0.0099	0.0006	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.010	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.012	pass	

Comments

See R17073101_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0313 (IPyC) and 1.0199 (OPyC).

QC Supervisor — 8-2-2017 — Date

QA Reviewer — 8/8/17 — Date

Figure 2-9. Inspection report for Batch 93172B Sample NP-C1517 pyrocarbon anisotropy.

The data report forms in Figure 2-10 and Figure 2-11 show the average anisotropy data for each particle cross section in terms of both the diattenuation and the OPTAF. Note that the standard deviation in the measured anisotropy within each layer was greater than the standard deviation in the distribution of measured values for the ten-particle sample. This illustrates that even though there is significant localized variation in the PyC microstructure within each layer, the average PyC anisotropy is relatively consistent from particle to particle. The data report forms in Figure 2-12 and Figure 2-13 show the average anisotropy data for each particle cross section in terms of both the diattenuation and the OPTAF for particles after heat treatment for one hour at 1800°C per DAM-41. These show that for both the OPyC and the IPyC there is a significant increase in anisotropy following heat treatment which simulates compacting. This data is not relevant to the acceptance criteria for these particles, and is provided for information only.

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17072801
Sample ID:	NP-C1517-A01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93172B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17073101\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0166	0.0031	0.0018	1.0338	0.0064	0.0037
2	0.0153	0.0030	0.0019	1.0311	0.0062	0.0039
3	0.0155	0.0038	0.0020	1.0315	0.0078	0.0041
4	0.0158	0.0031	0.0020	1.0321	0.0064	0.0041
5	0.0150	0.0030	0.0020	1.0305	0.0062	0.0041
6	0.0151	0.0037	0.0020	1.0307	0.0076	0.0041
7	0.0152	0.0040	0.0020	1.0309	0.0082	0.0041
8	0.0144	0.0034	0.0019	1.0292	0.0070	0.0039
9	0.0162	0.0033	0.0020	1.0329	0.0068	0.0041
10	0.0150	0.0032	0.0019	1.0305	0.0066	0.0039
Average	0.0154	0.0034	0.0020	1.0313	0.0069	0.0040
St. Dev.	0.0006	0.0004	0.0001	0.0013	0.0007	0.0001

Comments

Operator

8/2/17

Date

Figure 2-10. Data report for Batch 93172B Sample NP-C1517 IPyC anisotropy.

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17072801
Sample ID:	NP-C1517-A01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93172B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17073101\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0090	0.0025	0.0017	1.0182	0.0051	0.0035
2	0.0096	0.0024	0.0017	1.0194	0.0049	0.0035
3	0.0103	0.0031	0.0018	1.0208	0.0063	0.0037
4	0.0101	0.0031	0.0018	1.0204	0.0063	0.0037
5	0.0092	0.0027	0.0018	1.0186	0.0055	0.0037
6	0.0096	0.0030	0.0017	1.0194	0.0061	0.0035
7	0.0110	0.0028	0.0018	1.0222	0.0057	0.0037
8	0.0098	0.0026	0.0018	1.0198	0.0053	0.0037
9	0.0102	0.0031	0.0018	1.0206	0.0063	0.0037
10	0.0099	0.0028	0.0018	1.0200	0.0057	0.0037
Average	0.0099	0.0028	0.0018	1.0199	0.0057	0.0036
St. Dev.	0.0006	0.0003	0.0000	0.0012	0.0005	0.0001

Comments

Operator

8/2/17

Date

Figure 2-11. Data report for Batch 93172B Sample NP-C1517 OPyC anisotropy.

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17052401
Sample ID:	NP-C1517-D01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93172B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17062801\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0175	0.0036	0.0013	1.0356	0.0075	0.0027
2	0.0174	0.0029	0.0013	1.0354	0.0060	0.0027
3	0.0189	0.0035	0.0013	1.0385	0.0073	0.0027
4	0.0172	0.0029	0.0013	1.0350	0.0060	0.0027
5	0.0168	0.0029	0.0013	1.0342	0.0060	0.0027
6	0.0175	0.0029	0.0013	1.0356	0.0060	0.0027
7	0.0189	0.0031	0.0013	1.0385	0.0064	0.0027
8	0.0180	0.0031	0.0013	1.0367	0.0064	0.0027
9	0.0174	0.0031	0.0013	1.0354	0.0064	0.0027
10	0.0177	0.0036	0.0013	1.0360	0.0075	0.0027
Average	0.0177	0.0032	0.0013	1.0361	0.0066	0.0027
St. Dev.	0.0007	0.0003	0.0000	0.0014	0.0006	0.0000

Comments
Particles were heat treated for one hour at 1800C per DAM-41.


Operator


Date

Figure 2-12. Data report for Batch 93172B Sample NP-C1517 IPyC anisotropy after heating to 1800°C for one hour per DAM-41.

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17052401
Sample ID:	NP-C1517-D01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93172B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17062801\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0133	0.0031	0.0012	1.0270	0.0064	0.0025
2	0.0123	0.0029	0.0013	1.0249	0.0059	0.0027
3	0.0148	0.0029	0.0013	1.0300	0.0060	0.0027
4	0.0140	0.0030	0.0013	1.0284	0.0062	0.0027
5	0.0129	0.0027	0.0013	1.0261	0.0055	0.0027
6	0.0146	0.0029	0.0013	1.0296	0.0060	0.0027
7	0.0142	0.0040	0.0013	1.0288	0.0082	0.0027
8	0.0149	0.0027	0.0012	1.0303	0.0056	0.0025
9	0.0137	0.0027	0.0013	1.0278	0.0056	0.0027
10	0.0128	0.0039	0.0014	1.0259	0.0080	0.0029
Average	0.0138	0.0031	0.0013	1.0279	0.0063	0.0027
St. Dev.	0.0009	0.0005	0.0001	0.0019	0.0010	0.0001

Comments
Particles were heat treated for one hour at 1800C per DAM-41.

Operator

8/2/17
Date

Figure 2-13. Data report for Batch 93172B Sample NP-C1517 OPyC anisotropy after heating to 1800°C for one hour per DAM-41.

3. BATCH 93173B SAMPLE 1533

Sample NP-C1533 was a 130-gram sample riffled by BWXT from upgraded TRISO batch J52R-16-93173B and shipped to ORNL for QC acceptance testing and analysis.

3.1 BATCH 93173B SAMPLE 1533: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93173B Sample NP-C1533. Subsamples were riffled at ORNL according to the sampling instructions in Product Inspection Plan PIP-28. The combined number of particles with defective IPyC in these two subsamples is reported on Inspection Report Form IRF-28A (Figure 3-1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93173B Sample NP-C1533 meets the AGR-5/6/7 Fuel Specification requirements for the maximum defective IPyC fraction.

Inspection Report Form IRF-28A: AGR-5/6/7 Coated Particles

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1533
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B

Property	Measured Data # of particles	Specification INL SPC-1352	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
Defective IPyC coating fraction (fraction of total particles)	120897	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	1	pass	DRF-47

Comments

See NP-C1533-C01_DRF47R0 and NP-C1533-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography.
Sample also passed Stage 1 testing with 1 particles with excessive U dispersion out of 63585.

QC Supervisor — Date: 8-2-2017

QA Reviewer — Date: 8/8/17

Figure 3-1. Inspection report for Batch 93173B Sample NP-C1533 defective IPyC.

Figure 3-2 through Figure 3-7 are copies of the data report forms generated as part of the completion of Product Inspection Form PIP-28. Figure 3-2 is the particle weight determination used to ensure that each defect IPyC subsample had sufficient particles to meet the two acceptance test stages called out in the Statistical Sampling Plan for AGR-5/6/7 Fuel Materials. The minimum particle number requirements for this two-stage sampling appear in the acceptance criteria column in IRF-28A (Figure 3-1). Figure 3-3 is a record of the conditions of the particle heat treatment procedure. Figure 3-4 and Figure 3-6 are the individual results of the defective IPyC analysis for the two subsamples; these Data Report Forms (DRF-47) document the number of particles tested in each subsample and the number of particles counted as having defective IPyC based on their exhibition of excessive uranium dispersion. Figure 3-5 and Figure 3-7 are the associated DRF-47 supplemental data forms for the two analyzed subsamples and document the number of particles that had other anomalies of interest visible in the single x-ray radiograph image acquired of each particle. The supplemental data forms also report the fraction of particles in each subsample that exhibited each anomaly and a 95%-confidence prediction of the

maximum fraction in the TRISO particle batch, based on the observed number, the subsample size, and using binomial distribution statistics. Details about these anomalies and additional images of similar particles acquired by high-resolution x-ray tomography to further characterize them, as well as the uranium dispersion in the particles with defective IPyC, are available in a separate summary report [Helmreich et al. 2017a].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Grant Helmreich
Particle sample ID:	NP-C1533
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B
Filename:	\\mc-agr\AGR\ParticleWeight\W17051702_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1357	0.1067	0.1306	0.1119	0.1529
Number of particles:	131	103	125	107	146
Average weight/particle (g):	1.036E-03	1.036E-03	1.045E-03	1.046E-03	1.047E-03

Mean average weight/particle (g):	1.042E-03
Standard error in mean average weight/particle (g):	2.49E-06

Operator

7/28/17

Date

Figure 3-2. Data report for Batch 93173B Sample NP-C1533 average particle weight measured for subsample riffling.

Data Report Form DRF-41: Heat-treatment of Loose Particles Using a Graphite Furance

Procedure:	AGR-CHAR-DAM-41 Rev. 0
Operator:	John Hunn/Darren Skitt
DRF filename:	\\mc-agr\AGR\Furnaces\H17052301_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28R1				
Particle weight (g)	126.1192 g	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93173B				

Furnace calibration due date	10/13/2017

Thermal schedule				
Ramp 1	20	°C/min to	1800	°C
Dwell 1	1		hr	
Ramp 2	-20	°C/min to	700	°C
Dwell 2	0		hr	
Ramp 3	N/A	°C/min to	50	°C
Dwell 3	0		hr	
Ramp 4		°C/min to		°C
Dwell 4			hr	

Sample loading (top to bottom)			
Sample ID	NP-C1533-C01 (66.3325 g)	Crucible Marking	X
Sample ID	NP-C1533-D01 (59.7867 g)	Crucible Marking	Y
Sample ID		Crucible Marking	
Sample ID		Crucible Marking	
Sample ID		Crucible Marking	

3 vacuum / gas purges	1	2	3
Heat-treatment atmosphere	Vacuum		
Flow rate	N/A		

Comments

Ran furnace in Yokogawa auto mode. Controlled setpoint between 1770-1775°C to hold 1800°C on optical pyrometer.
Optical pyrometer calibration due 5/12/2018.

Operator

5-23-17

Date

Figure 3-3. Data report for Batch 93173B Sample NP-C1533 particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Indicating Defective IPyC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich / Darren Skitt
Particle sample ID:	NP-C1533-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052413\NP-C1533-C01_DRF47R0.xlsm

Weight of particles in sample (g):	66.3325
Number of particles in sample:	63585
Average weight/particle (g):	1.0432E-03

Number of particles identified as having Defective IPyC:	1

Comments

Tape mounts T17052401 thru T17052419.

Two additional particles were identified with uranium dispersion that was not caused by defective IPyC. One was associated with kernel debris trapped between the kernel and the buffer layer. The other was associated with a particle with two full kernels featuring localized dispersion.

Operator

8/14/17

Date

Figure 3-4. Data report for defective IPyC analysis of Batch 93173B subsample NP-C1533-C01.

Data Report Form DRF-47 Supplemental: Counting of Defects and Anomalies by Radiography

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich / Darren Skitt
Particle sample ID:	NP-C1533-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052413\NP-C1533-C01_DRF47R0.xlsm

Number of particles in sample:	63585
Number of radiographs analyzed:	7

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	3	4.72E-05	1.3E-04
Defective-IPyC related	*1*	*1.57E-05*	*7.5E-05*
Fractured-kernel related	*2*	*3.15E-05*	*1.0E-04*
White spots	3	4.72E-05	1.3E-04
Thin or low density SiC	5	7.86E-05	1.7E-04
Extra layers	0	0.00E+00	4.8E-05
Missing kernel	0	0.00E+00	4.8E-05
Kernel migration	0	0.00E+00	4.8E-05
Missing buffer	0	0.00E+00	4.8E-05
Particles with kernel anomalies	868	1.37E-02	1.5E-02
Dimple or facet	*810*	*1.27E-02*	*1.4E-02*
Severe dimple or facet	*17*	*2.67E-04*	*4.1E-04*
Notched kernel	*27*	*4.25E-04*	*5.9E-04*
Irregular kernel	*31*	*4.88E-04*	*6.6E-04*
Multi-kernel	*1*	*1.57E-05*	*7.5E-05*

Comments

Tape mounts T17052401 thru T17052419.

Two particles were identified with uranium dispersion that was not caused by defective IPyC. One was associated with kernel debris trapped between the kernel and the buffer layer. The other was associated with a particle with two full kernels featuring localized dispersion.

Operator

Date

Figure 3-5. Summary of anomalies observed during defective IPyC analysis of Batch 93173B subsample NP-C1533-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Indicating Defective IPyC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich / Austin Schumacher
Particle sample ID:	NP-C1533-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052513\NP-C1533-D01_DRF47R0.xlsm

Weight of particles in sample (g):	59.7867
Number of particles in sample:	57312
Average weight/particle (g):	1.0432E-03

Number of particles identified as having Defective IPyC:	0

Comments

Tape mounts T17052501 thru T17052517.

Five additional particles were identified with uranium dispersion that was not caused by defective IPyC. All five were determined to be associated with kernel debris trapped in the buffer layer near the kernel surface. Several fragments of broken kernels were identified in the sample, and one particle was identified in which an external kernel had reacted with the OPyC, SiC, and IPyC layers and infiltrated the buffer layer.

Operator

8/14/17

Date

Figure 3-6. Data report for defective IPyC analysis of Batch 93173B subsample NP-C1533-D01.

Data Report Form DRF-47 Supplemental: Counting of Defects and Anomalies by Radiography

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich / Austin Schumacher
Particle sample ID:	NP-C1533-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T17052513\NP-C1533-D01_DRF47R0.xlsm

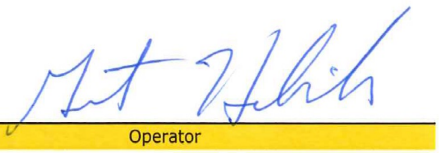
Number of particles in sample:	57312
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	5	8.72E-05	1.9E-04
Defective-IPyC related	*0*	*0.00E+00*	*5.3E-05*
Fractured-kernel related	*5*	*8.72E-05*	*1.9E-04*
White spots	8	1.40E-04	2.6E-04
Thin or low density SiC	2	3.49E-05	1.1E-04
Extra layers	0	0.00E+00	5.3E-05
Missing kernel	0	0.00E+00	5.3E-05
Kernel migration	1	1.74E-05	8.3E-05
Missing buffer	0	0.00E+00	5.3E-05
Particles with kernel anomalies	1779	3.10E-02	3.3E-02
Dimple or facet	*1604*	*2.80E-02*	*3.0E-02*
Severe dimple or facet	*48*	*8.38E-04*	*1.1E-03*
Notched kernel	*39*	*6.80E-04*	*8.9E-04*
Irregular kernel	*115*	*2.01E-03*	*2.4E-03*
Multi-kernel	*1*	*1.74E-05*	*8.3E-05*

Comments

Tape mounts T17052501 thru T17052517.

Five additional particles were identified with uranium dispersion that was not caused by defective IPyC. All five were determined to be assoiciated with kernel debris trapped in the buffer layer near the kernel surface. Several fragments of broken kernels were identified in the sample, and one particle was identified in which an external kernel had reacted with the OPyC, SiC, and IPyC layers and infiltrated the buffer layer.



Operator

Date

Figure 3-7. Summary of anomalies observed during defective IPyC analysis of Batch 93173B subsample NP-C1533-D01.

3.2 BATCH 93173B SAMPLE 1533: PYROCARBON ANISOTROPY

Average optical anisotropies of the IPyC and OPyC layers were measured on polished cross sections of 10 particles from a nominally 0.15-g subsample riffled at ORNL from TRISO Batch 93173B Sample NP-C1533, according to the sampling instructions in Product Inspection Plan PIP-28. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 3-8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93173B Sample NP-C1533 meets the AGR-5/6/7 Fuel Specification requirements for the IPyC and OPyC diattenuation.

Inspection Report Form IRF-28B: AGR-5/6/7 Coated Particles

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1533
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93173B

Property	Measured Data				Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value	INL SPC-923				
IPyC diattenuation	0.0139	0.0006	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.014	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.016	pass	
OPyC diattenuation	0.0098	0.0004	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.010	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.011	pass	

Comments

See R17080101_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0282 (IPyC) and 1.0198 (OPyC).

QC Supervisor — 8-2-2017 — Date

QA Reviewer — 8/8/17 — Date

Figure 3-8. Inspection report for Batch 93173B Sample NP-C1533 pyrocarbon anisotropy.

The data report forms in Figure 3-9 and Figure 3-10 show the average anisotropy data for each particle cross section in terms of both the diattenuation and the OPTAF. Note that the standard deviation in the measured anisotropy within each layer was greater than the standard deviation in the distribution of measured values for the ten-particle sample. This illustrates that even though there is significant localized variation in the PyC microstructure within each layer, the average PyC anisotropy is relatively consistent from particle to particle. The data report forms in Figure 3-11and Figure 3-12Figure 2-13 show the average anisotropy data for each particle cross section in terms of both the diattenuation and the OPTAF for particles after heat treatment for one hour at 1800°C per DAM-41. These show that for both the OPyC and the IPyC there is a significant increase in anisotropy following heat treatment which simulates compacting. This data is not relevant to the acceptance criteria for these particles, and is provided for information only.

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17072802
Sample ID:	NP-C1533-A01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93173B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17080101\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0130	0.0029	0.0020	1.0263	0.0060	0.0041
2	0.0132	0.0031	0.0020	1.0268	0.0064	0.0041
3	0.0146	0.0031	0.0020	1.0296	0.0064	0.0041
4	0.0132	0.0026	0.0019	1.0268	0.0053	0.0039
5	0.0139	0.0032	0.0020	1.0282	0.0066	0.0041
6	0.0145	0.0031	0.0021	1.0294	0.0064	0.0043
7	0.0144	0.0033	0.0020	1.0292	0.0068	0.0041
8	0.0134	0.0028	0.0021	1.0272	0.0058	0.0043
9	0.0143	0.0033	0.0021	1.0290	0.0068	0.0043
10	0.0146	0.0031	0.0022	1.0296	0.0064	0.0045
Average	0.0139	0.0031	0.0020	1.0282	0.0063	0.0042
St. Dev.	0.0006	0.0002	0.0001	0.0013	0.0005	0.0002

Comments

Operator

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Date

Figure 3-9. Data report for Batch 93173B Sample NP-C1533 IPyC anisotropy.

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17072802
Sample ID:	NP-C1533-A01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93173B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17080101\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0100	0.0026	0.0017	1.0202	0.0053	0.0035
2	0.0095	0.0032	0.0018	1.0192	0.0065	0.0037
3	0.0104	0.0029	0.0018	1.0210	0.0059	0.0037
4	0.0092	0.0025	0.0018	1.0186	0.0051	0.0037
5	0.0102	0.0033	0.0018	1.0206	0.0067	0.0037
6	0.0099	0.0025	0.0019	1.0200	0.0051	0.0039
7	0.0099	0.0025	0.0018	1.0200	0.0051	0.0037
8	0.0096	0.0028	0.0019	1.0194	0.0057	0.0039
9	0.0099	0.0037	0.0018	1.0200	0.0075	0.0037
10	0.0095	0.0030	0.0019	1.0192	0.0061	0.0039
Average	0.0098	0.0029	0.0018	1.0198	0.0059	0.0037
St. Dev.	0.0004	0.0004	0.0001	0.0007	0.0008	0.0001

Comments

Operator

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Date

Figure 3-10. Data report for Batch 93173B Sample NP-C1533 OPyC anisotropy.

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17052501
Sample ID:	NP-C1533-D01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93173B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17062901\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0173	0.0034	0.0014	1.0352	0.0070	0.0029
2	0.0170	0.0038	0.0014	1.0346	0.0079	0.0029
3	0.0157	0.0032	0.0014	1.0319	0.0066	0.0029
4	0.0182	0.0040	0.0014	1.0371	0.0083	0.0029
5	0.0196	0.0029	0.0014	1.0400	0.0060	0.0029
6	0.0172	0.0026	0.0014	1.0350	0.0054	0.0029
7	0.0172	0.0034	0.0014	1.0350	0.0070	0.0029
8	0.0168	0.0036	0.0014	1.0342	0.0074	0.0029
9	0.0170	0.0029	0.0014	1.0346	0.0060	0.0029
10	0.0176	0.0033	0.0014	1.0358	0.0068	0.0029
Average	0.0174	0.0033	0.0014	1.0353	0.0069	0.0029
St. Dev.	0.0010	0.0004	0.0000	0.0021	0.0009	0.0000

Comments
Particles were heat treated for one hour at 1800C per DAM-41.

Operator

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Date

Figure 3-11. Data report for Batch 93173B Sample NP-C1533 IPyC anisotropy after heating to 1800°C for one hour per DAM-41.

Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM2 - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17052501
Sample ID:	NP-C1533-D01
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93173B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17062901\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0141	0.0034	0.0013	1.0286	0.0070	0.0027
2	0.0128	0.0030	0.0013	1.0259	0.0062	0.0027
3	0.0135	0.0025	0.0013	1.0274	0.0051	0.0027
4	0.0141	0.0040	0.0013	1.0286	0.0082	0.0027
5	0.0151	0.0033	0.0013	1.0307	0.0068	0.0027
6	0.0136	0.0030	0.0013	1.0276	0.0062	0.0027
7	0.0127	0.0032	0.0013	1.0257	0.0066	0.0027
8	0.0127	0.0028	0.0013	1.0257	0.0057	0.0027
9	0.0140	0.0028	0.0013	1.0284	0.0058	0.0027
10	0.0137	0.0027	0.0013	1.0278	0.0056	0.0027
Average	0.0136	0.0031	0.0013	1.0276	0.0063	0.0027
St. Dev.	0.0008	0.0004	0.0000	0.0016	0.0009	0.0000

Comments
Particles were heat treated for one hour at 1800C per DAM-41.

Operator

Date

Figure 3-12. Data report for Batch 93173B Sample NP-C1533 OPyC anisotropy after heating to 1800°C for one hour per DAM-41.

4. CONCLUSION

The analyses called out in the ORNL Product Inspection Plan for AGR-5/6/7 Coated Particles, PIP-28, were completed as part of the acceptance testing of BWXT TRISO-coated particle batches 93172B and 93173B. Subsamples were analyzed by x-ray radiography to look for the uranium dispersion that is a marker for defective IPyC layers and with the ORNL 2-MGEM to measure the optical anisotropy of the pyrocarbon layers. Both batches met the AGR-5/6/7 Fuel Specification requirements for IPyC and OPyC anisotropy, as well as for defective IPyC. See Table 1-1, Table 1-2, and discussion in Section 1 for a summary of the measured values and Sections 2–3 for the associated inspection report forms and data report forms that contain the detailed data.

Additional analysis was performed to examine particles with defective IPyC and other interesting microstructural anomalies. In addition to the information extracted from the examination of the single-orientation radiographs, particles with defective IPyC and some particles with interesting anomalies were extracted from the Kapton tape holders used for radiography and imaged with higher-resolution x-ray tomography. The observed anomalies are briefly discussed in Section 1 and the number identified in each radiography sample is reported in Sections 2–3. Details of this additional analysis is provided in a separate report [Helmreich et al. 2017a].

5. REFERENCES

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