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Acceptance Test Data for Candidate AGR-5/6/7 TRISO Particle Batches

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February 2017

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

ORNL/TM-2017/036

Fusion and Materials for Nuclear Systems Division

ACCEPTANCE TEST DATA FOR CANDIDATE AGR-5/6/7 TRISO PARTICLE BATCHES

BWXT Coater Batches 93165 – 93172
Defective IPyC Fraction and Pyrocarbon Anisotropy

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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)

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1. INTRODUCTION AND SUMMARY

Coated particle fuel batches J52O-16-93165, 93166, 93168, 93169, 93170, and 93172 were produced by Babcock and Wilcox Technologies (BWXT) for possible selection as fuel 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). Some of these batches may alternately be used as demonstration 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., 93165A).

Samples riffled from each upgraded TRISO batch were 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 particle 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 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 each sample batch in Sections 2–7. Table 1-1 is a summary of the results. All analyzed batches satisfied the AGR-5/6/7 Fuel Specification for pyrocarbon anisotropy, with average diattenuation values well below the specified upper limits of ≤0.0170 for the IPyC layer and ≤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 from Batch 93172A to 1800°C to simulate compacting as described above, the average anisotropy of the IPyC layer increased from 0.0129 to 0.0159 and the average anisotropy of the OPyC layer increased from 0.0086 to 0.0116.

Table 1-1. Summary of measured properties

TRISO Batch	Sample	Average IPyC Anisotropy	Average OPyC Anisotropy	Defective IPyC [a] (red=out-of-spec)	Missing Buffer [b]
93165A[c]	NP-C1323 NP-C1350	N=0.0138 OPTAF=1.0279	N=0.0097 OPTAF=1.0195	2/121032=1.65×10^{-5} (5.21×10^{-5} at 95%)	1/121032=8.26×10^{-6} (3.92×10^{-5} at 95%)
93166RA	NP-C1358	N=0.0130 OPTAF=1.0264	N=0.0096 OPTAF=1.0193	18/120474=1.49×10^{-4} (2.22×10^{-4} at 95%)	1/120474=8.30×10^{-6} (3.94×10^{-5} at 95%)
93168A	NP-C1369	N=0.0127 OPTAF=1.0258	N=0.0089 OPTAF=1.0179	18/120819=1.49×10^{-4} (2.21×10^{-4} at 95%)	3/120819=2.48×10^{-5} (6.42×10^{-5} at 95%)
93169A	NP-C1391	N=0.0138 OPTAF=1.0279	N=0.0097 OPTAF=1.0195	5/122226=4.09×10^{-5} (8.61×10^{-5} at 95%)	20/122226=1.64×10^{-4} (2.38×10^{-4} at 95%)
93170A	NP-C1402	N=0.0135 OPTAF=1.0273	N=0.0089 OPTAF=1.0180	8/119831=6.68×10^{-5} (1.21×10^{-4} at 95%)	25/119831=2.09×10^{-4} (2.92×10^{-4} at 95%)
93172A	NP-C1421	N=0.0129 OPTAF=1.0262	N=0.0086 OPTAF=1.0173	10/121383=8.24×10^{-5} (1.40×10^{-4} at 95%)	4/121383=3.30×10^{-5} (7.55×10^{-5} at 95%)

[a] Values in parentheses are the 95%-confidence binomial distribution prediction of the maximum fraction in the TRISO particle batch and red shading indicates the batch did not meet the specified requirement.

[b] Missing buffer fraction was not a specified parameter for AGR-5/6/7 fuel but is included here as important information acquired during the defective IPyC analysis.

[c] Sample NP-1323 was used for pyrocarbon anisotropy determination and Sample NP-C1350 was used for analysis of defective IPyC; both were random samples riffled from Batch 93165A.

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 batch 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 1-1, Batches 93165A and 93169A passed the Stage 2 criteria. Batch 93165A had the lowest observed defective IPyC fraction and also passed the Stage 1 criteria with 2 defects out of 63045 particles. The subsamples taken from Batches 93170A and 93172A had too many defects to meet either acceptance criteria, but because the measured defect fraction in the analyzed sample (shown in Table 1-1) was below 10^{-4} there is a reasonable probability (but not assurance) that these batches could be shown to meet the specified requirement for defective IPyC if a large enough sample size was analyzed to reduce the statistical penalty in the determination of the maximum defect fraction in the batch at 95% confidence. Based on the current sampling, Batches 93170 and 93172 fail to meet the specified requirement for defective IPyC because they can only be predicted to have less than a 10^{-4} fraction of defective IPyC at 85% confidence (Batch 93170) and 67% confidence (Batch 93172). Batches 93166RA and 93168A both failed to meet the specified requirement for defective IPyC and, based on the relatively high number of observed defects, there is negligible probability that additional sampling would change this result.

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 2–7 for each sample analyzed for defective IPyC. One anomaly of particular interest was the presence of particles with a missing or very-thin buffer layer; this anomaly was most prevalent in batches 93169 and 93170 (Table 1-1). 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 the batches selected for the AGR-5/6/7 irradiation test by performing additional sieving to reduce the population of particles with this anomaly. Other anomalies that were specifically noted 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 is presented and the observed anomalies are discussed in greater detail in a separate report [Helmreich et al. 2017].

Similar analyses of pyrocarbon anisotropy, defective IPyC, and microstructural anomalies on a sample riffled from a BWXT pre-production batch of coated particles, 93164A, have been previously reported [Hunn et al. 2017a]. Batch 93164A was fabricated from an earlier kernel lot and was not considered for inclusion in the AGR-5/6/7 composite. Samples riffled from the final AGR-5/6/7 irradiation test fuel composite will be analyzed and documented in a future report [Hunn et al. 2017b]. The composite will include material from Batches 93165A, 93168A, 93169A, and 93170A discussed in this report. However, additional sieving will be performed to remove under-sized material and the designation for these batches will be changed by appending the letter B to the end of the batch number (e.g., 93165B). Analysis of the composite will identify any improvements achieved through the additional upgrading.

2. BATCH 93165A

Two samples riffled from upgraded TRISO batch J52O-16-93165A were shipped to ORNL for QC acceptance testing and analysis. Sample NP-C1323 was a 0.24-gram sample riffled by BWXT and shipped to ORNL for just pyrocarbon anisotropy measurement and was shipped in advance of Sample NP-C1350. Sample NP-C1350 was a 130-gram sample riffled by BWXT for defective IPyC analysis.

2.1 BATCH 93165A: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93165A Sample NP-C1350. 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 93165A 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-C1350
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93165A

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)	121032	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	2	pass	DRF-47

Comments

See NP-C1350-C01_DRF47R0 and NP-C1350-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 particle with excessive U dispersion out of 63045.

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 2-1. Inspection report for Batch 93165A 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 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. 2017].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Grant Helmreich
Particle sample ID:	NP-C1350-B00
Particle sample description:	Particles from BWXT coating batch J52O-16-93165A
Filename:	\\mc-agr\AGR\ParticleWeight\W16101402_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1599	0.1871	0.1579	0.1352	0.1386
Number of particles:	159	187	157	135	138
Average weight/particle (g):	1.01E-03	1.00E-03	1.01E-03	1.00E-03	1.00E-03

Mean average weight/particle (g):	1.004E-03
Standard error in mean average weight/particle (g):	1.08E-06

Operator

10/14/16

Date

Figure 2-2. Data report for Batch 93165A 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\H16101901_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	122.0298	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93165A				

Furnace calibration due date	10/13/17

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-C1350-C01 (63.5529 g)	Crucible Marking	X
Sample ID	NP-C1350-D01 (58.4769 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

Optical pyrometer calibration due 5/31/17.

John Hunn — Operator

10-19-16 — Date

Figure 2-3. Data report for Batch 93165A particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1350-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93165A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1350-C01_DRF47R0.xlsm

Weight of particles in sample (g):	63.5529
Number of particles in sample:	63045
Average weight/particle (g):	1.0081E-03

Number of particles with excessive U dispersion:	1

Comments

Tape mounts T16102101 thru T16102118.

Operator

11/18/16

Date

Figure 2-4. Data report for defective IPyC analysis of Batch 93165A subsample NP-C1350-C01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1350-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93165A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1350-C01_DRF47R0.xlsm

Number of particles in sample:	63045
Number of radiographs analyzed:	18

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	1	1.59E-05	7.6E-05
White spots	13	2.06E-04	3.3E-04
Thin or low density SiC	4	6.34E-05	1.5E-04
Extra layers	0	0.00E+00	4.8E-05
Missing kernel	2	3.17E-05	1.0E-04
Kernel migration	0	0.00E+00	4.8E-05
Missing buffer	0	0.00E+00	4.8E-05
Particles with kernel anomalies	732	1.16E-02	1.3E-02
Dimple or facet	*637*	*1.01E-02*	*1.1E-02*
Severe dimple or facet	*NA*	*NA*	*4.8E-05*
Notched kernel	*46*	*7.30E-04*	*9.4E-04*
Irregular kernel	*49*	*7.77E-04*	*9.9E-04*
Multi-kernel	*0*	*0.00E+00*	*4.8E-05*

Comments

Severe dimple or facet not categorized for this analysis.

Operator

11/18/16

Date

Figure 2-5. Summary of anomalies observed during defective IPyC analysis of Batch 93165A subsample NP-C1350-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1350-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93165A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1350-D01_DRF47R0.xlsm

Weight of particles in sample (g):	58.4769
Number of particles in sample:	57987
Average weight/particle (g):	1.0084E-03

Number of particles with excessive U dispersion:	1

Comments

Tape mounts T16102119 thru T16102135.

Operator

Date

Figure 2-6. Data report for defective IPyC analysis of Batch 93165A subsample NP-C1350-D01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1350-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93165A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1350-D01_DRF47R0.xlsm

Number of particles in sample:	57987
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	1	1.72E-05	8.2E-05
White spots	13	2.24E-04	3.6E-04
Thin or low density SiC	2	3.45E-05	1.1E-04
Extra layers	0	0.00E+00	5.2E-05
Missing kernel	0	0.00E+00	5.2E-05
Kernel migration	0	0.00E+00	5.2E-05
Missing buffer	1	1.72E-05	8.2E-05
Particles with kernel anomalies	902	1.56E-02	1.7E-02
Dimple or facet	*837*	*1.44E-02*	*1.6E-02*
Severe dimple or facet	*NA*	*NA*	*NA*
Notched kernel	*32*	*5.52E-04*	*7.5E-04*
Irregular kernel	*33*	*5.69E-04*	*7.7E-04*
Multi-kernel	*0*	*0.00E+00*	*5.2E-05*

Comments

Severe dimple or facet not categorized for this analysis.

Operator

11/18/16

Date

Figure 2-7. Summary of anomalies observed during defective IPyC analysis of Batch 93165A subsample NP-C1350-D01.

2.2 BATCH 93165A: PYROCARBON ANISOTROPY

Average optical anisotropies of the IPyC and OPyC layers were measured on polished cross sections of 10 particles from TRISO Batch 93165A Sample NP-C1323. Anisotropy measurements were not repeated on a riffled subsample from TRISO Batch 93165A Sample NP-C1350 per Product Inspection Plan PIP-28 because the measurement had been satisfactorily completed on the earlier sample.

The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 2-8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93165A 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-C1323
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93165A

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	0.0138	0.0009	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	0.017	pass	
OPyC diattenuation	0.0097	0.0005	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 R16080401_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0279 (IPyC) and 1.0195 (OPyC).

QC Supervisor — 1-27-17 — Date

QA Reviewer — 1/30/17 — Date

Figure 2-8. Inspection report for Batch 93165A pyrocarbon anisotropy.

The data report forms in Figure 2-9 and Figure 2-10 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.

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:	M16080202
Sample ID:	NP-C1323
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93165A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R16080401\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0142	0.0033	0.0012	1.0288	0.0068	0.0025
2	0.0154	0.0033	0.0013	1.0313	0.0068	0.0027
3	0.0139	0.0031	0.0013	1.0282	0.0064	0.0027
4	0.0141	0.0031	0.0014	1.0286	0.0064	0.0029
5	0.0133	0.0037	0.0014	1.0270	0.0076	0.0029
6	0.0128	0.0032	0.0014	1.0259	0.0066	0.0029
7	0.0137	0.0039	0.0014	1.0278	0.0080	0.0029
8	0.0122	0.0033	0.0014	1.0247	0.0068	0.0029
9	0.0146	0.0045	0.0014	1.0296	0.0093	0.0029
10	0.0133	0.0037	0.0014	1.0270	0.0076	0.0029
Average	0.0138	0.0035	0.0014	1.0279	0.0072	0.0028
St. Dev.	0.0009	0.0004	0.0001	0.0019	0.0009	0.0001

Comments
Sample NP-C1323 was a 0.24-gram sample riffled by BWXT and shipped to ORNL in July 2016. Because the sample was already an appropriately-small size, no riffling was done at ORNL and particles were selected for analysis directly from the received sample.

Operator

8/5/16

Date

Figure 2-9. Data report for Batch 93165A 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:	M16080202
Sample ID:	NP-C1323
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93165A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R16080401\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0099	0.0029	0.0012	1.0200	0.0059	0.0024
2	0.0102	0.0028	0.0013	1.0206	0.0057	0.0027
3	0.0103	0.0029	0.0014	1.0208	0.0059	0.0029
4	0.0093	0.0032	0.0013	1.0188	0.0065	0.0026
5	0.0098	0.0034	0.0013	1.0198	0.0069	0.0027
6	0.0100	0.0036	0.0014	1.0202	0.0073	0.0029
7	0.0092	0.0033	0.0015	1.0186	0.0067	0.0031
8	0.0090	0.0035	0.0015	1.0182	0.0071	0.0031
9	0.0095	0.0035	0.0014	1.0192	0.0071	0.0029
10	0.0093	0.0036	0.0015	1.0188	0.0073	0.0031
Average	0.0097	0.0033	0.0014	1.0195	0.0067	0.0028
St. Dev.	0.0005	0.0003	0.0001	0.0009	0.0006	0.0002

Comments
Sample NP-C1323 was a 0.24-gram sample riffled by BWXT and shipped to ORNL in July 2016. Because the sample was already an appropriately-small size, no riffling was done at ORNL and particles were selected for analysis directly from the received sample.

Operator

Date

Figure 2-10. Data report for Batch 93165A OPyC anisotropy.

3. BATCH 93166RA

Coated particle fuel batch J52O-16-93166 was not coated using the standard uninterrupted process due to a coater problem, and this fact was designated by appending an "R" to the run number (i.e., 93166R) to indicate the run was restarted after an interruption. After coating, the TRISO-coated particle batch was sieved similar to other batches to upgrade the particles by removing over-sized and under-sized material, and the upgraded batch was designated by appending the letter A to the end of the batch number (i.e., 93166RA). Sample NP-C1358 was a 130-gram sample riffled by BWXT from upgraded TRISO batch 93166RA and shipped to ORNL for QC acceptance testing and analysis.

3.1 BATCH 93166RA: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93166RA Sample NP-C1358. 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 93166RA does not meet 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-C1358
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93166RA

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)	120474	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	18	fail	DRF-47

Comments

See NP-C1358-C01_DRF47R0 and NP-C1358-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample also failed Stage 1 testing with 11 particles with excessive U dispersion out of 63685.

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 3-1. Inspection report for Batch 93166RA 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 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. 2017].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Grant Helmreich
Particle sample ID:	NP-C1358-B00
Particle sample description:	Particles from BWXT coating batch J52O-16-93166R
Filename:	\\mc-agr\AGR\ParticleWeight\W16102802_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1295	0.1182	0.1086	0.1406	0.1465
Number of particles:	125	114	105	136	141
Average weight/particle (g):	1.04E-03	1.04E-03	1.03E-03	1.03E-03	1.04E-03

Mean average weight/particle (g):	1.036E-03
Standard error in mean average weight/particle (g):	9.33E-07

Operator

10/28/16

Date

Figure 3-2. Data report for Batch 93166RA 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:	Darren Skitt/John Hunn
DRF filename:	\\mc-agr\AGR\Furnaces\H16110101_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	123.7491	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93166RA				

Furnace calibration due date	10/13/17

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-C1358-C01 (65.4097 g)	Crucible Marking	X
Sample ID	NP-C1358-D01 (58.3394 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

Optical pyrometer calibration due 5/31/17.

John Hunn — Operator

11-01-16 — Date

Figure 3-3. Data report for Batch 93166RA particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1358-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93166RA
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1358-C01_DRF47R0.xlsm

Weight of particles in sample (g):	65.4097
Number of particles in sample:	63685
Average weight/particle (g):	1.0271E-03

Number of particles with excessive U dispersion:	11

Comments

Tape mounts T16110201 thru T16110219.

Operator

11/18/16

Date

Figure 3-4. Data report for defective IPyC analysis of Batch 93166RA subsample NP-1358-C01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1358-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93166RA
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1358-C01_DRF47R0.xlsm

Number of particles in sample:	63685
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	11	1.73E-04	2.9E-04
White spots	4	6.28E-05	1.5E-04
Thin or low density SiC	4	6.28E-05	1.5E-04
Extra layers	1	1.57E-05	7.5E-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	712	1.12E-02	1.2E-02
Dimple or facet	*640*	*1.00E-02*	*1.1E-02*
Severe dimple or facet	*NA*	*NA*	*NA*
Notched kernel	*38*	*5.97E-04*	*7.9E-04*
Irregular kernel	*54*	*8.48E-04*	*1.1E-03*
Multi-kernel	*1*	*1.57E-05*	*7.5E-05*

Comments

Severe dimple or facet not categorized for this analysis.

Operator

Date

Figure 3-5. Summary of anomalies observed during defective IPyC analysis of Batch 93166RA subsample NP-1358-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1358-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93166RA
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1358-D01_DRF47R0.xlsm

Weight of particles in sample (g):	58.3394
Number of particles in sample:	56789
Average weight/particle (g):	1.0273E-03

Number of particles with excessive U dispersion:	7

Comments

Tape mounts T16110220 thru T16110236.

Operator

Date

Figure 3-6. Data report for defective IPyC analysis of Batch 93166RA subsample NP-1358-D01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	John Hunn/Grant Helmreich
Particle sample ID:	NP-C1358-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93166RA
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161102\NP-C1358-D01_DRF47R0.xlsm

Number of particles in sample:	56789
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	7	1.23E-04	2.4E-04
White spots	5	8.80E-05	1.9E-04
Thin or low density SiC	6	1.06E-04	2.1E-04
Extra layers	1	1.76E-05	8.4E-05
Missing kernel	0	0.00E+00	5.3E-05
Kernel migration	1	1.76E-05	8.4E-05
Missing buffer	1	1.76E-05	8.4E-05
Particles with kernel anomalies	939	1.65E-02	1.8E-02
Dimple or facet	*886*	*1.56E-02*	*1.7E-02*
Severe dimple or facet	*NA*	*NA*	*NA*
Notched kernel	*57*	*1.00E-03*	*1.3E-03*
Irregular kernel	*81*	*1.43E-03*	*1.8E-03*
Multi-kernel	*0*	*0.00E+00*	*5.3E-05*

Comments

Severe dimple or facet not categorized for this analysis.

Operator

11/18/16

Date

Figure 3-7. Summary of anomalies observed during defective IPyC analysis of Batch 93166RA subsample NP-1358-D01.

3.2 BATCH 93166RA: 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 93166RA Sample NP-C1358, 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 93166RA 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-C1358
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93166RA

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	0.0130	0.0005	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.013	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.015	pass	
OPyC diattenuation	0.0096	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 R16120501_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0264 (IPyC) and 1.0193 (OPyC).

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 3-8. Inspection report for Batch 93166RA 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.

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:	M16110202
Sample ID:	NP-C1358
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93166RA
Folder containing data:	\\mc-agr\AGR\2-MGEM\R16120501\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0121	0.0024	0.0010	1.0245	0.0049	0.0020
2	0.0129	0.0020	0.0010	1.0261	0.0041	0.0021
3	0.0127	0.0020	0.0010	1.0257	0.0041	0.0021
4	0.0130	0.0020	0.0010	1.0263	0.0041	0.0021
5	0.0131	0.0017	0.0010	1.0265	0.0035	0.0021
6	0.0136	0.0021	0.0011	1.0276	0.0043	0.0023
7	0.0138	0.0020	0.0010	1.0280	0.0041	0.0021
8	0.0129	0.0021	0.0011	1.0261	0.0043	0.0023
9	0.0135	0.0022	0.0011	1.0274	0.0045	0.0023
10	0.0125	0.0021	0.0011	1.0253	0.0043	0.0023
Average	0.0130	0.0021	0.0010	1.0264	0.0042	0.0021
St. Dev.	0.0005	0.0002	0.0001	0.0011	0.0004	0.0001

Comments

Operator

12/6/16

Date

Figure 3-9. Data report for Batch 93166RA 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:	M16110202
Sample ID:	NP-C1358
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93166RA
Folder containing data:	\\mc-agr\AGR\2-MGEM\R16120501\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0091	0.0024	0.0011	1.0184	0.0049	0.0022
2	0.0100	0.0220	0.0010	1.0202	0.0449	0.0020
3	0.0095	0.0018	0.0011	1.0192	0.0037	0.0022
4	0.0105	0.0023	0.0011	1.0212	0.0047	0.0022
5	0.0091	0.0017	0.0010	1.0184	0.0035	0.0020
6	0.0099	0.0015	0.0010	1.0200	0.0031	0.0020
7	0.0091	0.0026	0.0010	1.0184	0.0053	0.0020
8	0.0103	0.0017	0.0012	1.0208	0.0035	0.0025
9	0.0091	0.0025	0.0011	1.0184	0.0051	0.0022
10	0.0090	0.0024	0.0011	1.0182	0.0049	0.0022
Average	0.0096	0.0041	0.0011	1.0193	0.0083	0.0022
St. Dev.	0.0006	0.0063	0.0001	0.0012	0.0129	0.0001

Comments

Operator

12/6/16

Date

Figure 3-10. Data report for Batch 93166RA OPyC anisotropy.

4. BATCH 93168A

Sample NP-C1369 was a 130-gram sample riffled by BWXT from upgraded TRISO batch 93168A and shipped to ORNL for QC acceptance testing and analysis.

4.1 BATCH 93168A: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93168A Sample NP-C1369. 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 4-1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93168A does not meet 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-C1369
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93168A

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)	120819	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	18	fail	DRF-47

Comments

See NP-C1369-C01_DRF47R0 and NP-C1369-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample also failed Stage 1 testing with 9 particles with excessive U dispersion out of 64035.

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 4-1. Inspection report for Batch 93168A defective IPyC.

Figure 4-2 through Figure 4-7 are copies of the data report forms generated as part of the completion of Product Inspection Form PIP-28. Figure 4-2 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 4-1). Figure 4-3 is a record of the conditions of the particle heat treatment procedure. Figure 4-4 and Figure 4-6 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 4-5 and Figure 4-7 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 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. 2017].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Darren Skitt
Particle sample ID:	NP-C1369-B00
Particle sample description:	Particles from BWXT coating batch J52O-16-93168A
Filename:	\\mc-agr\AGR\ParticleWeight\W16121501_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.0985	0.1013	0.1112	0.1405	0.1858
Number of particles:	98	99	110	137	183
Average weight/particle (g):	1.01E-03	1.02E-03	1.01E-03	1.03E-03	1.02E-03

Mean average weight/particle (g):	1.016E-03
Standard error in mean average weight/particle (g):	3.80E-06

Operator

12/15/16

Date

Figure 4-2. Data report for Batch 93168A 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:	Darren Skitt/John Hunn
DRF filename:	\\mc-agr\AGR\Furnaces\H16122701_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	122.1559	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93168A				

Furnace calibration due date	10/13/17

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-C1369-C01 (64.7520 g)	Crucible Marking	X
Sample ID	NP-C1369-D01 (57.4039 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

Furnace issues made hold 20°C/min ramp difficult, maximum ramp rate was 25-27°C/min.
Temperature slowly increased during 1 hour hold from 1780-1820.
Optical pyrometer calibration due 5/31/17.

John Hunn
Operator

12-27-16
Date

Figure 4-3. Data report for Batch 93168A particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich
Particle sample ID:	NP-C1369-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93168A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161228\NP-C1369-C01_DRF47R0.xlsm

Weight of particles in sample (g):	64.7520
Number of particles in sample:	64035
Average weight/particle (g):	1.0112E-03

Number of particles with excessive U dispersion:	9

Comments

Tape mounts T16122818 thru T16122836.

Operator | Date

Figure 4-4. Data report for defective IPyC analysis of Batch 93168A subsample NP-1369-C01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich
Particle sample ID:	NP-C1369-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93168A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161228\NP-C1369-C01_DRF47R0.xlsm

Number of particles in sample:	64035
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	9	1.41E-04	2.5E-04
White spots	18	2.81E-04	4.2E-04
Thin or low density SiC	3	4.68E-05	1.3E-04
Extra layers	0	0.00E+00	4.7E-05
Missing kernel	0	0.00E+00	4.7E-05
Kernel migration	0	0.00E+00	4.7E-05
Missing buffer	1	1.56E-05	7.5E-05
Particles with kernel anomalies	1558	2.43E-02	2.6E-02
Dimple or facet	*1356*	*2.12E-02*	*2.3E-02*
Severe dimple or facet	*106*	*1.66E-03*	*2.0E-03*
Notched kernel	*46*	*7.18E-04*	*9.2E-04*
Irregular kernel	*72*	*1.12E-03*	*1.4E-03*
Multi-kernel	*7*	*1.09E-04*	*2.1E-04*

Comments

Two unique particles (T16122818_x6234_y6837 and T16122828_x1298_y248) were found which had a kernel fragment trapped between the SiC and OPyC layers.

Operator

1/27/17

Date

Figure 4-5. Summary of anomalies observed during defective IPyC analysis of Batch 93168A subsample NP-1369-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Austin Schumacher
Particle sample ID:	NP-C1369-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93168A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161228\NP-C1369-D01_DRF47R0.xlsm

Weight of particles in sample (g):	57.4039
Number of particles in sample:	56784
Average weight/particle (g):	1.0109E-03

Number of particles with excessive U dispersion:	9

Comments

Tape mounts T16122801 thru T16122817.

Operator

1/27/17

Date

Figure 4-6. Data report for defective IPyC analysis of Batch 93168A subsample NP-1369-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-C1369-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93168A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161228\NP-C1369-D01_DRF47R0.xlsm

Number of particles in sample:	56784
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	9	1.58E-04	2.8E-04
White spots	9	1.58E-04	2.8E-04
Thin or low density SiC	6	1.06E-04	2.1E-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	2	3.52E-05	1.2E-04
Particles with kernel anomalies	1823	3.21E-02	3.4E-02
Dimple or facet	*1539*	*2.71E-02*	*2.9E-02*
Severe dimple or facet	*62*	*1.09E-03*	*1.4E-03*
Notched kernel	*76*	*1.34E-03*	*1.7E-03*
Irregular kernel	*173*	*3.05E-03*	*3.5E-03*
Multi-kernel	*16*	*2.82E-04*	*4.3E-04*

Comments

Operator

1/27/17

Date

Figure 4-7. Summary of anomalies observed during defective IPyC analysis of Batch 93168A subsample NP-1369-D01.

4.2 BATCH 93168A: 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 Batch 93168A Sample NP-C1369. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 4-8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93168A 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-C1369
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93168A

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	0.0127	0.0005	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.013	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.015	pass	
OPyC diattenuation	0.0089	0.0004	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.009	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.010	pass	

Comments

See R17010901_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0258 (IPyC) and 1.0179 (OPyC).

QC Supervisor — 1-27-17 — Date

QA Reviewer — 1/30/17 — Date

Figure 4-8. Inspection report for Batch 93168A pyrocarbon anisotropy.

The data report forms in Figure 4-9 and Figure 4-10 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.

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:	M16121501
Sample ID:	NP-C1369
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93168A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17010901\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0129	0.0024	0.0011	1.0261	0.0049	0.0023
2	0.0119	0.0027	0.0012	1.0241	0.0055	0.0025
3	0.0127	0.0019	0.0011	1.0257	0.0039	0.0023
4	0.0129	0.0020	0.0012	1.0261	0.0041	0.0025
5	0.0123	0.0017	0.0012	1.0249	0.0035	0.0025
6	0.0135	0.0017	0.0012	1.0274	0.0035	0.0025
7	0.0123	0.0021	0.0012	1.0249	0.0043	0.0025
8	0.0132	0.0017	0.0012	1.0268	0.0035	0.0025
9	0.0129	0.0020	0.0013	1.0261	0.0041	0.0027
10	0.0128	0.0017	0.0013	1.0259	0.0035	0.0027
Average	0.0127	0.0020	0.0012	1.0258	0.0041	0.0025
St. Dev.	0.0005	0.0003	0.0001	0.0010	0.0007	0.0001

Comments

Operator

1/10/17

Date

Figure 4-9. Data report for Batch 93168A 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:	M16121501
Sample ID:	NP-C1369
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93168A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17010901\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0090	0.0022	0.0010	1.0182	0.0045	0.0020
2	0.0083	0.0020	0.0012	1.0167	0.0041	0.0024
3	0.0086	0.0019	0.0011	1.0173	0.0039	0.0022
4	0.0091	0.0018	0.0011	1.0184	0.0037	0.0022
5	0.0087	0.0019	0.0013	1.0176	0.0039	0.0026
6	0.0084	0.0017	0.0013	1.0169	0.0035	0.0026
7	0.0087	0.0019	0.0013	1.0176	0.0039	0.0026
8	0.0097	0.0021	0.0013	1.0196	0.0043	0.0027
9	0.0093	0.0022	0.0013	1.0188	0.0045	0.0026
10	0.0089	0.0019	0.0013	1.0180	0.0039	0.0026
Average	0.0089	0.0020	0.0012	1.0179	0.0040	0.0025
St. Dev.	0.0004	0.0002	0.0001	0.0009	0.0003	0.0002

Comments

Operator

1/10/17

Date

Figure 4-10. Data report for Batch 93168A OPyC anisotropy.

5. BATCH 93169A

Sample NP-C1391 was a 130-gram sample riffled by BWXT from upgraded TRISO batch 93169A and shipped to ORNL for QC acceptance testing and analysis.

5.1 BATCH 93169A: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93169A Sample NP-C1391. 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 5-1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93169A 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-C1391
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93169A

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)	122226	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	5	pass	DRF-47

Comments

See NP-C1391-C01_DRF47R0 and NP-C1391-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample failed Stage 1 testing with 3 particles with excessive U dispersion out of 64652.

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 5-1. Inspection report for Batch 93169A defective IPyC.

Figure 5-2 through Figure 5-7 are copies of the data report forms generated as part of the completion of Product Inspection Form PIP-28. Figure 5-2 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 5-1). Figure 5-3 is a record of the conditions of the particle heat treatment procedure. Figure 5-4 and Figure 5-6 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 5-5 and Figure 5-7 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 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. 2017].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	John Dyer/Felipe Mora
Particle sample ID:	NP-C1391-B00
Particle sample description:	Particles from BWXT coating batch J52O-16-93169A
Filename:	\\mc-agr\AGR\ParticleWeight\W16121502_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1154	0.1250	0.1299	0.1644	0.1347
Number of particles:	115	124	130	165	133
Average weight/particle (g):	1.00E-03	1.01E-03	9.99E-04	9.96E-04	1.01E-03

Mean average weight/particle (g):	1.004E-03
Standard error in mean average weight/particle (g):	2.96E-06

Operator

12/15/2016
Date

Figure 5-2. Data report for Batch 93169A 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:	Darren Skitt/John Hunn
DRF filename:	\\mc-agr\AGR\Furnaces\H16122801_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	123.2550	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93169A				

Furnace calibration due date	10/13/17

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-C1391-C01 (65.1854 g)	Crucible Marking	W
Sample ID	NP-C1391-D01 (58.0696 g)	Crucible Marking	Z
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 manual power control mode. Held ramp rate between 15-20°C/min, then 1 hour between 1800-1814°C.
Cooled furnace at 20°C/min down to 960°C, at which point power was 0 and remaining cool was <20°C/min.
Optical pyrometer calibration due 5/31/17.

John Hunn — Operator

12-28-16 — Date

Figure 5-3. Data report for Batch 93169A particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1391-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93169A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161229\NP-C1391-C01_DRF47R0.xlsm

Weight of particles in sample (g):	65.1854
Number of particles in sample:	64652
Average weight/particle (g):	1.0083E-03

Number of particles with excessive U dispersion:	3

Comments

Tape mounts T16122901 thru T16122919.

Operator

1/27/17

Date

Figure 5-4. Data report for defective IPyC analysis of Batch 93169A subsample NP-1391-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-C1391-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93169A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161229\NP-C1391-C01_DRF47R0.xlsm

Number of particles in sample:	64652
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	3	4.64E-05	1.2E-04
White spots	0	0.00E+00	4.7E-05
Thin or low density SiC	5	7.73E-05	1.7E-04
Extra layers	0	0.00E+00	4.7E-05
Missing kernel	0	0.00E+00	4.7E-05
Kernel migration	0	0.00E+00	4.7E-05
Missing buffer	10	1.55E-04	2.7E-04
Particles with kernel anomalies	1163	1.80E-02	1.9E-02
Dimple or facet	*1035*	*1.60E-02*	*1.7E-02*
Severe dimple or facet	*38*	*5.88E-04*	*7.8E-04*
Notched kernel	*32*	*4.95E-04*	*6.7E-04*
Irregular kernel	*82*	*1.27E-03*	*1.6E-03*
Multi-kernel	*1*	*1.55E-05*	*7.4E-05*

Comments

One unique particle (T16122904_x3870_y4447) was found with a very small kernel fragment apparently encased in soot then coated with buffer, IPyC, buffer, IPyC, SiC, and OPyC.

Operator

1/27/17

Date

Figure 5-5. Summary of anomalies observed during defective IPyC analysis of Batch 93169A subsample NP-1391-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt/Austin Schumacher
Particle sample ID:	NP-C1391-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93169A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161229\NP-C1391-D01_DRF47R0.xlsm

Weight of particles in sample (g):	58.0696
Number of particles in sample:	57574
Average weight/particle (g):	1.0086E-03

Number of particles with excessive U dispersion:	2

Comments

Tape mounts T16122920 thru T16122936.

Operator

1/27/17

Date

Figure 5-6. Data report for defective IPyC analysis of Batch 93169A subsample NP-1391-D01.

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/Austin Schumacher
Particle sample ID:	NP-C1391-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93169A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161229\NP-C1391-D01_DRF47R0.xlsm

Number of particles in sample:	57574
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	2	3.47E-05	1.1E-04
White spots	8	1.39E-04	2.6E-04
Thin or low density SiC	8	1.39E-04	2.6E-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	10	1.74E-04	3.0E-04
Particles with kernel anomalies	1447	2.51E-02	2.7E-02
Dimple or facet	*1253*	*2.18E-02*	*2.3E-02*
Severe dimple or facet	*54*	*9.38E-04*	*1.2E-03*
Notched kernel	*49*	*8.51E-04*	*1.1E-03*
Irregular kernel	*129*	*2.24E-03*	*2.6E-03*
Multi-kernel	*4*	*6.95E-05*	*1.6E-04*

Comments

Operator

1/27/17

Date

Figure 5-7. Summary of anomalies observed during defective IPyC analysis of Batch 93169A subsample NP-1391-D01.

5.2 BATCH 93169A: 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 Batch 93169A Sample NP-C1391. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 5-8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93169A 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-C1391
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93169A

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	0.0138	0.0008	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	0.017	pass	
OPyC diattenuation	0.0097	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 R17011001_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0279 (IPyC) and 1.0195 (OPyC).

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 5-8. Inspection report for Batch 93169A pyrocarbon anisotropy.

The data report forms in Figure 5-9 and Figure 5-10 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.

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:	M16121602
Sample ID:	NP-C1391
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93169A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17011001\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0148	0.0021	0.0013	1.0300	0.0043	0.0027
2	0.0140	0.0019	0.0014	1.0284	0.0039	0.0029
3	0.0132	0.0022	0.0014	1.0268	0.0045	0.0029
4	0.0137	0.0027	0.0013	1.0278	0.0056	0.0027
5	0.0121	0.0019	0.0013	1.0245	0.0039	0.0027
6	0.0142	0.0018	0.0013	1.0288	0.0037	0.0027
7	0.0140	0.0019	0.0014	1.0284	0.0039	0.0029
8	0.0143	0.0022	0.0012	1.0290	0.0045	0.0025
9	0.0143	0.0020	0.0012	1.0290	0.0041	0.0025
10	0.0130	0.0018	0.0012	1.0263	0.0037	0.0025
Average	0.0138	0.0021	0.0013	1.0279	0.0042	0.0027
St. Dev.	0.0008	0.0003	0.0001	0.0016	0.0006	0.0002

Comments

Operator

1/13/17

Date

Figure 5-9. Data report for Batch 93169A 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:	M16121602
Sample ID:	NP-C1391
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93169A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17011001\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0096	0.0015	0.0013	1.0194	0.0031	0.0027
2	0.0101	0.0016	0.0014	1.0204	0.0033	0.0029
3	0.0095	0.0021	0.0014	1.0192	0.0043	0.0029
4	0.0104	0.0026	0.0015	1.0210	0.0053	0.0031
5	0.0088	0.0018	0.0015	1.0178	0.0037	0.0031
6	0.0099	0.0019	0.0014	1.0200	0.0039	0.0029
7	0.0095	0.0017	0.0014	1.0192	0.0035	0.0029
8	0.0104	0.0022	0.0013	1.0210	0.0045	0.0027
9	0.0097	0.0022	0.0012	1.0196	0.0045	0.0024
10	0.0087	0.0017	0.0012	1.0176	0.0035	0.0024
Average	0.0097	0.0019	0.0014	1.0195	0.0039	0.0028
St. Dev.	0.0006	0.0003	0.0001	0.0012	0.0007	0.0002

Comments

Operator

1/13/17

Date

Figure 5-10. Data report for Batch 93169A OPyC anisotropy.

6. BATCH 93170A

Sample NP-C1402 was a 130-gram sample riffled by BWXT from upgraded TRISO batch 93170A and shipped to ORNL for QC acceptance testing and analysis.

6.1 BATCH 93170A: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93170A Sample NP-C1402. 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 6-1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93170A does not meet 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-C1402
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93170A

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)	119831	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	8	fail	DRF-47

Comments

See NP-C1402-C01_DRF47R0 and NP-C1402-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample also failed Stage 1 testing with 5 particles with excessive U dispersion out of 63773.

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 6-1. Inspection report for Batch 93170A defective IPyC.

Figure 6-2 through Figure 6-7 are copies of the data report forms generated as part of the completion of Product Inspection Form PIP-28. Figure 6-2 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 6-1). Figure 6-3 is a record of the conditions of the particle heat treatment procedure. Figure 6-4 and Figure 6-6 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 6-5 and Figure 6-7 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 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. 2017].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Darren Skitt
Particle sample ID:	NP-C1402-B00
Particle sample description:	Particles from BWXT coating batch J52O-16-93170A
Filename:	\\mc-agr\AGR\ParticleWeight\W16121601_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1714	0.1724	0.1740	0.2052	0.2166
Number of particles:	168	168	172	202	214
Average weight/particle (g):	1.02E-03	1.03E-03	1.01E-03	1.02E-03	1.01E-03

Mean average weight/particle (g):	1.017E-03
Standard error in mean average weight/particle (g):	2.72E-06

Operator

12/16/16

Date

Figure 6-2. Data report for average particle weight measured for Batch 93170A subsample riffling.

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

Procedure:	AGR-CHAR-DAM-41 Rev. 0
Operator:	Grant Helmreich/John Hunn
DRF filename:	\\mc-agr\AGR\Furnaces\H16122901_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	122.1350	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93170A				

Furnace calibration due date	10/13/17

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-C1402-C01 (65.0014 g)	Crucible Marking	X
Sample ID	NP-C1402-D01 (57.1336 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 manual power control mode. Held ramp rate at 20°C/min, then 1 hour between 1800-1808°C.
Cooled furnace at 20°C/min down to 960°C, at which point power was 0 and remaining cool was <20°C/min.
Optical pyrometer calibration due 5/31/17.

Operator

12-29-16

Date

Figure 6-3. Data report for Batch 93170A particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1402-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93170A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161230\NP-C1402-C01_DRF47R0.xlsm

Weight of particles in sample (g):	65.0014
Number of particles in sample:	63773
Average weight/particle (g):	1.0193E-03

Number of particles with excessive U dispersion:	5

Comments

Tape mounts T16123001 thru T16123019.

Operator

1/27/17

Date

Figure 6-4. Data report for defective IPyC analysis of Batch 93170A subsample NP-1402-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-C1402-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93170A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161230\NP-C1402-C01_DRF47R0.xlsm

Number of particles in sample:	63773
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	5	7.84E-05	1.7E-04
White spots	15	2.35E-04	3.7E-04
Thin or low density SiC	15	2.35E-04	3.7E-04
Extra layers	0	0.00E+00	4.7E-05
Missing kernel	0	0.00E+00	4.7E-05
Kernel migration	0	0.00E+00	4.7E-05
Missing buffer	11	1.72E-04	2.9E-04
Particles with kernel anomalies	1551	2.43E-02	2.6E-02
Dimple or facet	*1415*	*2.22E-02*	*2.4E-02*
Severe dimple or facet	*50*	*7.84E-04*	*1.0E-03*
Notched kernel	*41*	*6.43E-04*	*8.4E-04*
Irregular kernel	*75*	*1.18E-03*	*1.5E-03*
Multi-kernel	*1*	*1.57E-05*	*7.5E-05*

Comments

Operator

1/27/17

Date

Figure 6-5. Summary of anomalies observed during defective IPyC analysis of Batch 93170A subsample NP-1402-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

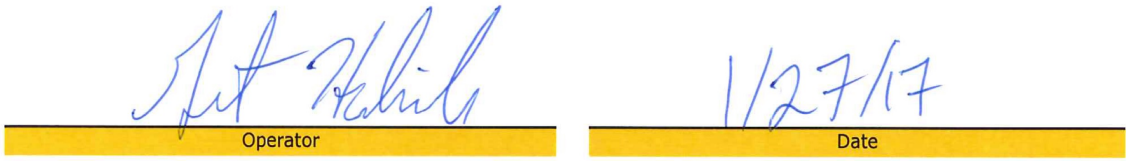
Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1402-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93170A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161230\NP-C1402-D01_DRF47R0.xlsm

Weight of particles in sample (g):	57.1336
Number of particles in sample:	56058
Average weight/particle (g):	1.0192E-03

Number of particles with excessive U dispersion:	3

Comments

Tape mounts T16123020 thru T16123036.



Operator | Date

Figure 6-6. Data report for defective IPyC analysis of Batch 93170A subsample NP-1402-D01.

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-C1402-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93170A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T161230\NP-C1402-D01_DRF47R0.xlsm

Number of particles in sample:	56058
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	3	5.35E-05	1.4E-04
White spots	1	1.78E-05	8.5E-05
Thin or low density SiC	10	1.78E-04	3.1E-04
Extra layers	1	1.78E-05	8.5E-05
Missing kernel	0	0.00E+00	5.4E-05
Kernel migration	0	0.00E+00	5.4E-05
Missing buffer	14	2.50E-04	4.0E-04
Particles with kernel anomalies	781	1.39E-02	1.5E-02
Dimple or facet	*720*	*1.28E-02*	*1.4E-02*
Severe dimple or facet	*6*	*1.07E-04*	*2.2E-04*
Notched kernel	*19*	*3.39E-04*	*5.0E-04*
Irregular kernel	*54*	*9.63E-04*	*1.3E-03*
Multi-kernel	*1*	*1.78E-05*	*8.5E-05*

Comments

Two completely bare kernels were found by radiography and confirmed optically (T16123020_x7142_y10113 and T16123022_x6304_y8586). The particle with extra layers (T16123020_x6803_y321) was found to have extra layers of buffer and IPyC.

Operator

1/27/17

Date

Figure 6-7. Summary of anomalies observed during defective IPyC analysis of Batch 93170A subsample NP-1402-D01.

6.2 BATCH 93170A: 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 Batch 93170A Sample NP-C1402. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 6-8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93170A 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-C1402
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93170A

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	0.0135	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	0.0089	0.0005	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.009	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.011	pass	

Comments

See R17011101_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0273 (IPyC) and 1.0180 (OPyC).

QC Supervisor — Date: 1-27-17

QA Reviewer — Date: 1/30/17

Figure 6-8. Inspection report for Batch 93170A pyrocarbon anisotropy.

The data report forms in Figure 6-9 and Figure 6-10 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.

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:	M16121604
Sample ID:	NP-C1402
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93170A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17011101\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0139	0.0024	0.0012	1.0282	0.0049	0.0025
2	0.0132	0.0017	0.0012	1.0268	0.0035	0.0025
3	0.0127	0.0026	0.0012	1.0257	0.0053	0.0025
4	0.0134	0.0020	0.0012	1.0272	0.0041	0.0025
5	0.0129	0.0025	0.0012	1.0261	0.0051	0.0025
6	0.0137	0.0019	0.0012	1.0278	0.0039	0.0025
7	0.0147	0.0016	0.0012	1.0298	0.0033	0.0025
8	0.0132	0.0020	0.0012	1.0268	0.0041	0.0025
9	0.0131	0.0019	0.0012	1.0265	0.0039	0.0025
10	0.0137	0.0019	0.0012	1.0278	0.0039	0.0025
Average	0.0135	0.0021	0.0012	1.0273	0.0042	0.0025
St. Dev.	0.0006	0.0003	0.0000	0.0012	0.0007	0.0000

Comments

Operator

1/13/17

Date

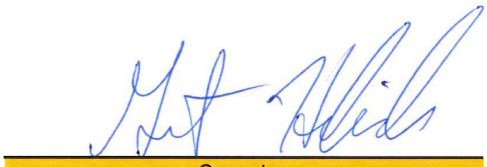
Figure 6-9. Data report for Batch 93170A IPyC anisotropy.

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:	M16121604
Sample ID:	NP-C1402
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93170A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17011101\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0088	0.0024	0.0013	1.0178	0.0049	0.0026
2	0.0081	0.0020	0.0013	1.0163	0.0041	0.0026
3	0.0089	0.0016	0.0011	1.0180	0.0033	0.0022
4	0.0089	0.0015	0.0012	1.0180	0.0031	0.0024
5	0.0088	0.0020	0.0012	1.0178	0.0041	0.0024
6	0.0097	0.0018	0.0012	1.0196	0.0037	0.0024
7	0.0089	0.0017	0.0011	1.0180	0.0035	0.0022
8	0.0093	0.0021	0.0012	1.0188	0.0043	0.0024
9	0.0084	0.0017	0.0012	1.0169	0.0035	0.0024
10	0.0093	0.0020	0.0012	1.0188	0.0041	0.0024
Average	0.0089	0.0019	0.0012	1.0180	0.0038	0.0024
St. Dev.	0.0005	0.0003	0.0001	0.0009	0.0005	0.0001

Comments



Operator

1-13-17

Date

Figure 6-10. Data report for Batch 93170A OPyC anisotropy.

7. BATCH 93172A

Sample NP-C1421 was a 130-gram sample riffled by BWXT from upgraded TRISO batch 93172A and shipped to ORNL for QC acceptance testing and analysis.

7.1 BATCH 93172A: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Batch 93172A Sample NP-C14212. 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 7-1) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93172A does not meet 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-C1421
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172A

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)	121383	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	10	fail	DRF-47

Comments

See NP-C1421-C01_DRF47R0 and NP-C1421-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample also failed Stage 1 testing with 6 particles with excessive U dispersion out of 64611.

QC Supervisor — Date: 2-15-17

QA Reviewer — Date: 2/15/17

Figure 7-1. Inspection report for Batch 93172A defective IPyC.

Figure 7-2 through Figure 7-7 are copies of the data report forms generated as part of the completion of Product Inspection Form PIP-28. Figure 7-2 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 7-1). Figure 7-3 is a record of the conditions of the particle heat treatment procedure. Figure 7-4 and Figure 7-6 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 7-5 and Figure 7-7 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 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. 2017].

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Grant Helmreich
Particle sample ID:	NP-C1421
Particle sample description:	Particles from BWXT coating batch J52O-16-93172A
Filename:	\\mc-agr\AGR\ParticleWeight\W17012602_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1354	0.1253	0.1551	0.1382	0.1457
Number of particles:	128	120	148	132	139
Average weight/particle (g):	1.06E-03	1.04E-03	1.05E-03	1.05E-03	1.05E-03

Mean average weight/particle (g):	1.049E-03
Standard error in mean average weight/particle (g):	2.31E-06

Operator

1/26/17

Date

Figure 7-2. Data report for Batch 93172A 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\H17012701_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	126.9315	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT coating batch J52O-16-93172A				

Furnace calibration due date	10/13/17

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-C1421-C01 (67.6002 g)	Crucible Marking	X
Sample ID	NP-C1421-D01 (59.3313 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

Reconnected the Eurotherm controller and ran furnace in auto mode. Ramp rates maintained as specified. Maximum temperature on optical pyrometer was 1820°C.
Optical pyrometer calibration due 5/31/17.

John Hunn — Operator

1-27-17 — Date

Figure 7-3. Data report for Batch 93172A particle heat treatment to simulate compact heat treatment.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1421-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170130\NP-C1421-C01_DRF47R0.xlsm

Weight of particles in sample (g):	67.6002
Number of particles in sample:	64611
Average weight/particle (g):	1.0463E-03

Number of particles with excessive U dispersion:	6

Comments

Tape mounts T17013001 thru T17013019.

Operator

2/15/17

Date

Figure 7-4. Data report for defective IPyC analysis of Batch 93172A subsample NP-1421-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-C1421-C01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170130\NP-C1421-C01_DRF47R0.xlsm

Number of particles in sample:	64611
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	6	9.29E-05	1.9E-04
White spots	16	2.48E-04	3.8E-04
Thin or low density SiC	4	6.19E-05	1.5E-04
Extra layers	0	0.00E+00	4.7E-05
Missing kernel	0	0.00E+00	4.7E-05
Kernel migration	0	0.00E+00	4.7E-05
Missing buffer	2	3.10E-05	9.8E-05
Particles with kernel anomalies	1211	1.87E-02	2.0E-02
Dimple or facet	*1079*	*1.67E-02*	*1.8E-02*
Severe dimple or facet	*47*	*7.27E-04*	*9.3E-04*
Notched kernel	*33*	*5.11E-04*	*6.9E-04*
Irregular kernel	*86*	*1.33E-03*	*1.6E-03*
Multi-kernel	*4*	*6.19E-05*	*1.5E-04*

Comments

Operator

2/15/17

Date

Figure 7-5. Summary of anomalies observed during defective IPyC analysis of Batch 93172A subsample NP-1421-C01.

Data Report Form DRF-47: Counting of Particles with Excessive Uranium Dispersion Inside SiC

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt/Austin Schumacher
Particle sample ID:	NP-C1421-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170130\NP-C1421-D01_DRF47R0.xlsm

Weight of particles in sample (g):	59.3313
Number of particles in sample:	56772
Average weight/particle (g):	1.0451E-03

Number of particles with excessive U dispersion:	4

Comments

Tape mounts T17013020 thru T17013036.

Operator

2/15/17

Date

Figure 7-6. Data report for defective IPyC analysis of Batch 93172A subsample NP-1421-D01.

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/Austin Schumacher
Particle sample ID:	NP-C1421-D01
Particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172A
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170130\NP-C1421-D01_DRF47R0.xlsm

Number of particles in sample:	56772
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	4	7.05E-05	1.7E-04
White spots	6	1.06E-04	2.1E-04
Thin or low density SiC	4	7.05E-05	1.7E-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	2	3.52E-05	1.2E-04
Particles with kernel anomalies	887	1.56E-02	1.7E-02
Dimple or facet	*743*	*1.31E-02*	*1.4E-02*
Severe dimple or facet	*41*	*7.22E-04*	*9.4E-04*
Notched kernel	*36*	*6.34E-04*	*8.4E-04*
Irregular kernel	*77*	*1.36E-03*	*1.7E-03*
Multi-kernel	*3*	*5.28E-05*	*1.4E-04*

Comments

Operator

2/15/17

Date

Figure 7-7. Summary of anomalies observed during defective IPyC analysis of Batch 93172A subsample NP-1421-D01.

7.2 BATCH 93172A: 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 Batch 93172A Sample NP-C1421. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 7-8) with a determination as to whether the particle batch satisfied the specified parameters for this property. Batch 93172A 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-C1421
Coated particle sample description:	TRISO particles from BWXT coating batch J52O-16-93172A

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	0.0129	0.0005	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.013	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.015	pass	
OPyC diattenuation	0.0086	0.0007	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.009	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.011	pass	

Comments

See R17020701_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0262 (IPyC) and 1.0173 (OPyC).

QC Supervisor — 2-15-17 Date

QA Reviewer — 2/15/17 Date

Figure 7-8. Inspection report for Batch 93172A pyrocarbon anisotropy.

The data report forms in Figure 7-9 and Figure 7-10 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.

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:	M17013002
Sample ID:	NP-C1421
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93172A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17020701\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0134	0.0022	0.0013	1.0272	0.0045	0.0027
2	0.0133	0.0019	0.0014	1.0270	0.0039	0.0029
3	0.0130	0.0029	0.0014	1.0263	0.0060	0.0029
4	0.0130	0.0028	0.0015	1.0263	0.0057	0.0031
5	0.0136	0.0029	0.0015	1.0276	0.0060	0.0031
6	0.0129	0.0021	0.0014	1.0261	0.0043	0.0029
7	0.0122	0.0023	0.0015	1.0247	0.0047	0.0031
8	0.0125	0.0021	0.0016	1.0253	0.0043	0.0033
9	0.0121	0.0019	0.0015	1.0245	0.0039	0.0031
10	0.0133	0.0025	0.0015	1.0270	0.0051	0.0031
Average	0.0129	0.0024	0.0015	1.0262	0.0048	0.0030
St. Dev.	0.0005	0.0004	0.0001	0.0011	0.0008	0.0002

Comments

Operator

2/10/17

Date

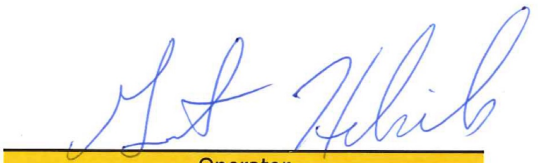
Figure 7-9. Data report for Batch 93172A IPyC anisotropy.

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:	M17013002
Sample ID:	NP-C1421
Sample Description:	TRISO particles from BWXT coating batch J52O-16-93172A
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17020701\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0089	0.0020	0.0013	1.0180	0.0041	0.0026
2	0.0079	0.0021	0.0013	1.0159	0.0043	0.0026
3	0.0092	0.0022	0.0014	1.0186	0.0045	0.0029
4	0.0077	0.0022	0.0015	1.0155	0.0045	0.0030
5	0.0092	0.0027	0.0014	1.0186	0.0055	0.0029
6	0.0084	0.0018	0.0013	1.0169	0.0037	0.0026
7	0.0091	0.0024	0.0014	1.0184	0.0049	0.0029
8	0.0075	0.0022	0.0015	1.0151	0.0045	0.0030
9	0.0083	0.0022	0.0014	1.0167	0.0045	0.0028
10	0.0094	0.0024	0.0015	1.0190	0.0049	0.0031
Average	0.0086	0.0022	0.0014	1.0173	0.0045	0.0028
St. Dev.	0.0007	0.0002	0.0001	0.0014	0.0005	0.0002

Comments



Operator

Date

Figure 7-10. Data report for Batch 93172A OPyC anisotropy.

8. 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 93165A, 93166RA, 93168A, 93169A, 93170A, 93172A. 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. All batches met the AGR-5/6/7 Fuel Specification requirements for IPyC and OPyC anisotropy, but only Batches 93165 and 93169 met the specified requirements for defective IPyC. See Table 1-1 and discussion in Section 1 for a summary of the measured values and Sections 2–7 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 2–7. Details of this additional analysis is provided in a separate report [Helmreich et al. 2017].

9. REFERENCES

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