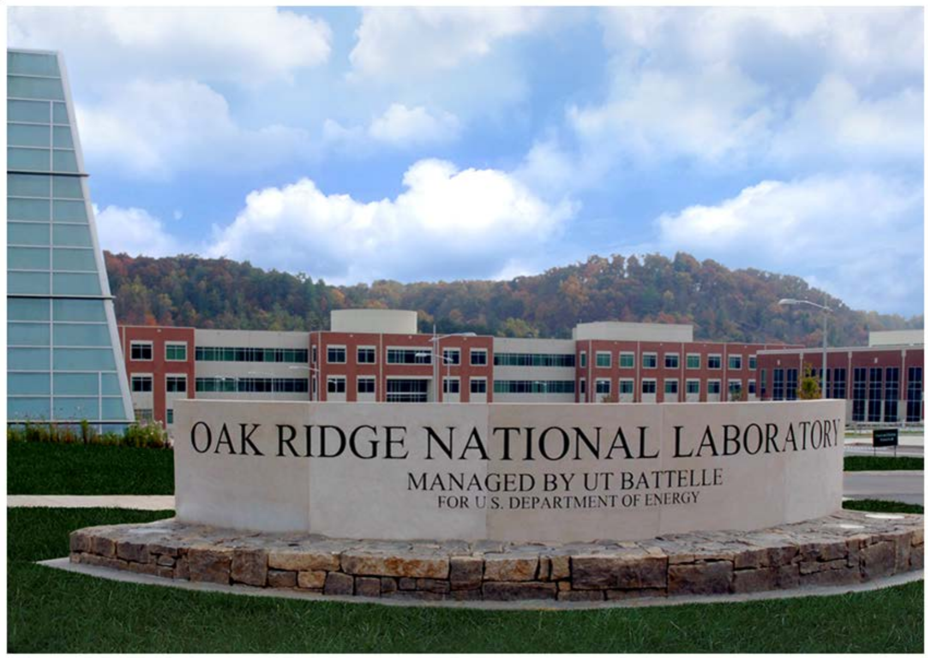
ORNL/TM-2017/037

Acceptance Test Data for the AGR-5/6/7 Irradiation Test Fuel TRISO Composite



Grant W. Helmreich
John D. Hunn
Darren J. Skitt
John A. Dyer
Austin T. Schumacher

May 2017

**Approved for public release.
Distribution is unlimited.**

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

ORNL/TM-2017/037

Fusion and Materials for Nuclear Systems Division

ACCEPTANCE TEST DATA FOR THE AGR-5/6/7 IRRADIATION TEST FUEL TRISO COMPOSITE

Defective IPyC Fraction and Pyrocarbon Anisotropy

Grant W. Helmreich
John D. Hunn
Darren J. Skitt
John A. Dyer
Austin T. Schumacher

Date Published: May 2017

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

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

CONTENTS

LIST OF FIGURES

LIST OF TABLES

ACRONYMS

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

ACKNOWLEDGMENTS

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

1 INTRODUCTION AND SUMMARY

Coated particle composite J52R-16-98005 was produced by Babcock and Wilcox Technologies (BWXT) 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). This composite was comprised of four coated particle fuel batches J52O-16-93165B (26%), 93168B (26%), 93169B (24%), and 93170B (24%), chosen based on the Quality Control (QC) data acquired for each individual candidate AGR-5/6/7 batch. 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). Secondary upgrading by sieving was performed on the A-designated batches to remove particles with missing or very-thin buffer layers that were identified during previous analysis of the individual batches for defective IPyC, as reported in the acceptance test data report for the AGR-5/6/7 production batches [Hunn et al. 2017]. The additionally-upgraded batches were designated by appending the letter B to the end of the batch number (e.g., 93165B).

Composite 98005 was blended by first riffling each chosen batch into four equal aliquots and then blending one aliquot from each chosen batch into a composite aliquot, resulting in four composite aliquots. Samples were riffled from two of the composite aliquots and shipped to the Oak Ridge National Laboratory (ORNL) for 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 composite 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 average IPyC and OPyC anisotropy are reported for the two samples from Composite 98005 in Sections 2.2 and 3.2; Table 1-1 is a summary of the results. Both analyzed sublots satisfied the AGR-5/6/7 Fuel Specification for pyrocarbon anisotropy, with average diattenuation values sufficiently 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 heated to even higher temperatures. For additional information, anisotropy was measured again on particles heated to 1800°C to simulate compacting as described above. Table 1-1 shows that the average anisotropy of both the IPyC and OPyC layers increased as expected. The fractional increase in the OPyC diattenuation (~44%) was greater than for the IPyC (~22–27%), and this was at least partially due to the fact that the OPyC had not already experienced the period of heating to around 1550°C during SiC deposition. However, the fact that the OPyC and IPyC diattenuation values did not converge after the 1800°C heat treatment indicates that there was probably some microstructural differences between these two layers.

Table 1-1. Summary of pyrocarbon anisotropy for coated particle composite J52R-16-98005

Sample	Average anisotropy of as-deposited TRISO		Average Anisotropy after heating to 1800°C	
	IPyC	OPyC	IPyC	OPyC
NP-C1498	N=0.0149 OPTAF=1.0303	N=0.0103 OPTAF=1.0208	N=0.0182 OPTAF=1.0370	N=0.0148 OPTAF=1.0300
NP-C1504	N=0.0157 OPTAF=1.0318	N=0.0100 OPTAF=1.0203	N=0.0200 OPTAF=1.0407	N=0.0144 OPTAF=1.0291
Combined*	N=0.0153 OPTAF=1.0311	N=0.0102 OPTAF=1.0205	N=0.0191 OPTAF=1.0388	N=0.0146 OPTAF=1.0296

*Inspection report form for combined results is provided in Appendix A.

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 composite has a defect fraction less than this limit. Acceptance testing was performed by riffling, per PIP-28, two random subsamples from each sample provided by BWXT containing the appropriate number of particles to apply two predetermined acceptance criteria derived using binomial distribution statistics. The Stage 1 acceptance criteria was ≤2 defects in a random group of at least 62956 particles. The Stage 2 acceptance criteria was ≤6 defects in a random group of at least 118422 particles. The analysis results from the first riffled subsample were used for Stage 1, and the combined results from both subsamples were used for Stage 2. Target weights for the riffled subsamples were determined based on the average particle weight, with a sufficient margin based on the uncertainty in the average particle weight to ensure the subsamples provided at least the required minimum number of particles, while minimizing overshoot. Minimizing overshoot is important because the probability that a sublot with an acceptable defect population will satisfy the acceptance criteria decreases as a function of increasing difference between the actual number of particles analyzed and the minimum required. The exact number of particles in each subsample was determined by using image analysis to count the particles in the x-ray radiographs acquired for the defective IPyC analysis.

As shown in Table 1-2, both samples passed the Stage 2 criteria (Sample NP-C1498 also passed the Stage 1 criteria). Detailed results of this analysis are reported in Sections 2.1 and 3.1. Since both samples were randomly-selected representative samples from the same source material (coated particle composite J52R-16-98005), the results may be combined for a single statistical analysis (see Appendix A for inspection report form for combined data). This has the effect of significantly reducing the statistical penalty involved in calculating the 95% confidence value by doubling the total number of particles that were sampled. The results for both samples combined are given in Table 1-2, and due to the reduction in the statistical penalty, the combined sample shows that the composite passes the upper limit on defective IPyC fraction with greater margin.

Table 1-2. Summary of Defective IPyC for coated particle composite J52R-16-98005

Sample	Number of particles in analyzed sample	Number of particles with Defective IPyC	Measured defect fraction in sample	Max defect fraction at 95% confidence
NP-C1498	121018	5 (pass)	4.13×10^{-5}	8.69×10^{-5}
NP-C1504	120804	6 (pass)	4.97×10^{-5}	9.81×10^{-5}
Combined*	241822	11 (pass)	4.55×10^{-5}	7.53×10^{-5}

*Inspection report form for combined results are provided in Appendix A.

Additional particles were identified with uranium dispersion that were not counted as particles with Defective IPyC and not included in the defect totals in Table 1-2. The AGR-5/6/7 Fuel Specification [Marshall 2016] defines particles with Defective IPyC using a visual standard that depicts the characteristic uranium dispersion which occurs in these defective particles after heating to 1800°C. Based on consensus of the technical leads, particles with uranium dispersion localized near the kernel or near an embedded kernel fragment do not meet the visual standard for Defective IPyC, which has a characteristic of radial symmetry. High-resolution x-ray tomography confirmed this localized uranium dispersion was associated with reactive fracture surfaces on the kernel or reactive kernel fragments embedded in the buffer. It was decided based on the additional analysis that only particles with radially-symmetric uranium dispersion characteristic of HCl infiltration through an unacceptably permeable IPyC layer would be counted as having Defective IPyC. Although particles with uranium dispersion due to reactive kernel fragments or surfaces were not counted for the defective IPyC specification, their presence may be of concern for fuel performance and should be considered for inclusion in the fuel specification.

As mentioned above, secondary upgrading by sieving was performed to remove particles with missing or very-thin buffer layers that were identified during previous analysis of the individual batches for defective IPyC [Hunn et al. 2017]. A population limit on particles with very-thin or missing buffer was not included in the AGR-5/6/7 Fuel Specification [Marshall 2016]. However, the observation of this anomaly in samples riffled from individual coater batches at fractional populations as high as 2.1×10^{-4} produced significant concern due to the high likelihood of TRISO-coating failure during the irradiation test. No particles with very-thin or missing buffer were found in either sample from Composite 98005 (Table 1-3), while approximately 12 would have been expected in each sample, based on the individual batch data [Hunn et al. 2017], had the secondary upgrading not been performed. This indicates that the additional sieving to upgrade each batch was effective in removing these anomalous particles.

Table 1-3. Summary of Missing Buffer* for coated particle composite J52R-16-98005

Sample	Number of particles in analyzed sample	Number of particles with Missing Buffer	Measured defect fraction in sample	Max defect fraction at 95% confidence
NP-C1498	121018	0	0	2.48×10^{-5} at 95%
NP-C1504	120804	0	0	2.48×10^{-5} at 95%
Combined	241822	0	0	1.24×10^{-5} at 95%

* 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.

The careful examination of the x-ray radiographs required for determination of defective IPyC fraction provided an opportunity to also inspect the two samples for other microstructural anomalies. A supplemental Data Report Form (DRF-47 Supplemental) is included in Sections 2.1 and 3.1 for each sample analyzed for defective IPyC. 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 gas 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 considered to be 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 are presented and the observed anomalies are discussed in greater detail in a separate report [Helmreich et al. 2017].

2 COMPOSITE 98005 SAMPLE NP-C1498

Sample NP-C1498 was a 130-gram sample riffled by BWXT from AGR-5/6/7 Composite J52R-16-98005 and shipped to ORNL for determination of Defective IPyC fraction and measurement of pyrocarbon anisotropy as part of the AGR-5/6/7 irradiation test fuel QC acceptance testing.

2.1 COMPOSITE 98005 SAMPLE NP-C1498: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Composite 98005 Sample NP-C1498. 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 composite satisfied the specified parameters for this property. Composite 98005 Sample NP-C1498 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-C1498
Coated particle sample description:	TRISO particles from BWXT composite J52R-16-98005

Property	Measured Data	Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	# of particles	INL SPC-1352				
Defective IPyC coating fraction (fraction of total particles)	121018	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	5	pass	DRF-47

Comments
See NP-C1498-C01_DRF47R0 and NP-C1498-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample NP-C1498-C01 also passed Stage 1 testing with 1 particle with excessive U dispersion indicative of Defective IPyC out of 64005. Three other particles were noted to have localized U dispersion associated with kernel debris trapped in the buffer layer near the kernel surface (in two cases) or with a notch in the kernel surface where a section of the kernel had broken away (in the third case). These were not counted as having Defective IPyC.

QC Supervisor	5-1-17 Date
QA Reviewer	5/1/17 Date

Figure 2-1. Inspection report for Composite 98005 Sample NP-C1498 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 indicative of particles with Defective IPyC. 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 composite, 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-C1498-B00
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
Filename:	\\mc-agr\AGR\ParticleWeight\W17030201_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1654	0.1364	0.1594	0.1648	0.1630
Number of particles:	164	134	156	161	162
Average weight/particle (g):	1.009E-03	1.018E-03	1.022E-03	1.024E-03	1.006E-03

Mean average weight/particle (g):	1.016E-03
Standard error in mean average weight/particle (g):	3.51E-06

3/2/17

Operator　　　　Date

Figure 2-2. Data report for Composite 98005 Sample NP-C1498 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\H17030201_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	122.5283	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT composite J52R-16-98005				

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-C1498-C01 (64.7924 g)	Crucible Marking	X
Sample ID	NP-C1498-D01 (57.7359 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 Eurotherm auto mode. Set controller temperature to 1780°C to get 1800°C optical pyrometer temperature.
Optical pyrometer calibration due 5/31/17.
0.0188 g removed from C1498-D01 for heated-particle anisotropy sample.

Operator

3-2-17

Date

Figure 2-3. Data report for Composite 98005 Sample NP-C1498 particle heat treatment to simulate compact heat treatment.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1498-C01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170303\NP-C1498-C01_DRF47R0.xlsm

Weight of particles in sample (g):	64.7924
Number of particles in sample:	64005
Average weight/particle (g):	1.0123E-03

Number of particles identified as having Defective IPyC:	1

Comments

Tape mounts T17030301 thru T17030319.

One additional particle was identified with uranium dispersion that was determined with high-resolution x-ray tomography to be associated with kernel debris trapped in the buffer layer near the kernel surface.

Operator

4/28/17

Date

Figure 2-4. Data report for defective IPyC analysis of Composite 98005 subsample NP-C1498-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-C1498-C01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170303\NP-C1498-C01_DRF47R0.xlsm

Number of particles in sample:	64005
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	2	3.12E-05	9.9E-05
Defective-IPyC related	*1*	*1.56E-05*	*7.5E-05*
Fractured-kernel related	*1*	*1.56E-05*	*7.5E-05*
White spots	4	6.25E-05	1.5E-04
Thin or low density SiC	5	7.81E-05	1.7E-04
Extra layers	0	0.00E+00	4.7E-05
Missing kernel	0	0.00E+00	4.7E-05
Kernel migration	2	3.12E-05	9.9E-05
Missing buffer	0	0.00E+00	4.7E-05
Particles with kernel anomalies	1158	1.81E-02	1.9E-02
Dimple or facet	*1092*	*1.71E-02*	*1.8E-02*
Severe dimple or facet	*8*	*1.25E-04*	*2.3E-04*
Notched kernel	*48*	*7.50E-04*	*9.6E-04*
Irregular kernel	*24*	*3.75E-04*	*5.3E-04*
Multi-kernel	*2*	*3.12E-05*	*9.9E-05*

Comments

Two bare kernels were found at T17030318_x1796_y7239 (partial) and T17030307_x2233_y3901 (whole).
Two particles were identified by x-ray radiography to have excessive uranium dispersion.
High-resolution x-ray tomography was used to determine that the uranium dispersion in one of these particles was localized around kernel debris trapped in the buffer layer near the kernel surface.
The uranium dispersion in the other particle was presumed to indicate defective IPyC, as it was more symmetric and not obviously associated with kernel debris.

Operator | Date: 4/28/17

Figure 2-5. Summary of anomalies observed during defective IPyC analysis of Composite 98005 subsample NP-C1498-C01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Austin Schumacher/Darren Skitt
Particle sample ID:	NP-C1498-D01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170303\NP-C1498-D01_DRF47R0.xlsm

Weight of particles in sample (g):	57.7359
Number of particles in sample:	57013
Average weight/particle (g):	1.0127E-03

Number of particles identified as having Defective IPyC:	4

Comments

Tape mounts T17030320 thru T17030336.

Two additional particles were identified with uranium dispersion that was determined with high-resolution x-ray tomography in one case to be associated with kernel debris trapped in the buffer layer near the kernel surface and in the other case to be associated with a notch in the surface where a section of kernel was missing.

Grant Helmreich — 4/28/17

Operator — Date

Figure 2-6. Data report for defective IPyC analysis of Composite 98005 subsample NP-C1498-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/Darren Skitt
Particle sample ID:	NP-C1498-D01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170303\NP-C1498-D01_DRF47R0.xlsm

Number of particles in sample:	57013
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	6	1.05E-04	2.1E-04
Defective-IPyC related	*4*	*7.02E-05*	*1.7E-04*
Fractured-kernel related	*2*	*3.51E-05*	*1.2E-04*
White spots	3	5.26E-05	1.4E-04
Thin or low density SiC	4	7.02E-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	0	0.00E+00	5.3E-05
Particles with kernel anomalies	1369	2.40E-02	2.6E-02
Dimple or facet	*1138*	*2.00E-02*	*2.1E-02*
Severe dimple or facet	*136*	*2.39E-03*	*2.8E-03*
Notched kernel	*19*	*3.33E-04*	*4.7E-04*
Irregular kernel	*86*	*1.51E-03*	*1.9E-03*
Multi-kernel	*4*	*7.02E-05*	*1.9E-04*

Comments

Six particles were identified by x-ray radiography to have excessive uranium dispersion.
High-resolution x-ray tomography was used to determine that the uranium dispersion in one of these particles was localized around kernel debris trapped in the buffer layer near the kernel surface and the uranium dispersion in another particle was localized around a notch in the surface where a section of kernel was missing.
The uranium dispersion in the other four particles was presumed to indicate defective IPyC, as it was more symmetric and not obviously associated with kernel debris.

Operator:

4/28/17
Date

Figure 2-7. Summary of anomalies observed during defective IPyC analysis of Composite 98005 subsample NP-C1498-D01.

2.2 COMPOSITE 98005 SAMPLE NP-C1498: 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 Composite 98005 Sample NP-C1498 according to the sampling instructions in Product Inspection Plan PIP-28. The average optical diattenuation values of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-28B (Figure 2-8) with a determination as to whether the particle composite satisfied the specified parameters for this property. Composite 98005 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-C1498-A01
Coated particle sample description:	TRISO particles from BWXT composite J52R-16-98005

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	0.0149	0.0008	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.015	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.018	pass	
OPyC diattenuation	0.0103	0.0006	10	1.833	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.011	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.013	pass	

Comments

See R17031601_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0303 (IPyC) and 1.0208 (OPyC).

QC Supervisor — Date: 5-1-17

QA Reviewer — Date: 5/1/17

Figure 2-8. Inspection report for Composite 98005 Sample NP-C1498 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.

The data report forms in Figure 2-11 and Figure 2-12 show the average anisotropy data for each particle cross section following heat treatment at 1800°C for one hour to simulate compacting. As expected based on prior research [Hunn et al. 2007], the anisotropy of both the IPyC and OPyC layers increased following this heat treatment. The AGR-5/6/7 Fuel Specification for pyrocarbon anisotropy is defined for particles which have not undergone further heat treatment, and as such these results are provided for information only.

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

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17030202
Sample ID:	NP-C1498-A01
Sample Description:	TRISO particles from BWXT composite J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17031601\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0154	0.0036	0.0011	1.0313	0.0074	0.0023
2	0.0141	0.0028	0.0010	1.0286	0.0058	0.0021
3	0.0147	0.0026	0.0010	1.0298	0.0054	0.0021
4	0.0146	0.0027	0.0011	1.0296	0.0056	0.0023
5	0.0147	0.0030	0.0010	1.0298	0.0062	0.0021
6	0.0164	0.0028	0.0011	1.0333	0.0058	0.0023
7	0.0145	0.0026	0.0010	1.0294	0.0054	0.0021
8	0.0138	0.0031	0.0011	1.0280	0.0064	0.0023
9	0.0157	0.0034	0.0011	1.0319	0.0070	0.0023
10	0.0152	0.0038	0.0012	1.0309	0.0078	0.0025
Average	0.0149	0.0030	0.0011	1.0303	0.0063	0.0022
St. Dev.	0.0008	0.0004	0.0001	0.0016	0.0009	0.0001

Comments

Grant Helmreich

Operator

4/11/17

Date

Figure 2-9. Data report for Composite 98005 Sample NP-C1498 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:	M17030202
Sample ID:	NP-C1498-A01
Sample Description:	TRISO particles from BWXT composite J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17031601\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0108	0.0029	0.0009	1.0218	0.0059	0.0018
2	0.0099	0.0024	0.0009	1.0200	0.0049	0.0018
3	0.0087	0.0024	0.0010	1.0176	0.0049	0.0020
4	0.0107	0.0026	0.0010	1.0216	0.0053	0.0020
5	0.0107	0.0027	0.0010	1.0216	0.0055	0.0020
6	0.0102	0.0028	0.0010	1.0206	0.0057	0.0020
7	0.0105	0.0022	0.0010	1.0212	0.0045	0.0020
8	0.0103	0.0023	0.0010	1.0208	0.0047	0.0020
9	0.0108	0.0032	0.0010	1.0218	0.0065	0.0020
10	0.0103	0.0040	0.0011	1.0208	0.0082	0.0022
Average	0.0103	0.0028	0.0010	1.0208	0.0056	0.0020
St. Dev.	0.0006	0.0005	0.0001	0.0013	0.0011	0.0001

Comments

Grant Helmreich

Operator

4/11/17

Date

Figure 2-10. Data report for Composite 98005 Sample NP-C1498 OPyC anisotropy.

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

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17030302
Sample ID:	NP-C1498-D01
Sample Description:	Heat-treated TRISO particles from BWXT composite J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17032201\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0190	0.0038	0.0014	1.0387	0.0079	0.0029
2	0.0154	0.0031	0.0013	1.0313	0.0064	0.0027
3	0.0184	0.0037	0.0014	1.0375	0.0077	0.0029
4	0.0175	0.0048	0.0015	1.0356	0.0099	0.0031
5	0.0197	0.0043	0.0015	1.0402	0.0089	0.0031
6	0.0196	0.0042	0.0015	1.0400	0.0087	0.0031
7	0.0181	0.0039	0.0015	1.0369	0.0081	0.0031
8	0.0194	0.0030	0.0015	1.0396	0.0062	0.0031
9	0.0173	0.0038	0.0015	1.0352	0.0079	0.0031
10	0.0171	0.0042	0.0015	1.0348	0.0087	0.0031
Average	0.0182	0.0039	0.0015	1.0370	0.0081	0.0030
St. Dev.	0.0014	0.0005	0.0001	0.0028	0.0011	0.0001

Comments

Particles were heat treated to 1800°C for one hour according to DAM-41.

Grant Helmreich

Operator

4/11/17

Date

Figure 2-11. Data report for Composite 98005 Sample NP-C1498 IPyC anisotropy after heating to 1800°C.

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:	M17030302
Sample ID:	NP-C1498-D01
Sample Description:	Heat-treated TRISO particles from BWXT composite J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17032201\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0155	0.0041	0.0014	1.0315	0.0085	0.0029
2	0.0136	0.0033	0.0013	1.0276	0.0068	0.0027
3	0.0157	0.0038	0.0016	1.0319	0.0078	0.0033
4	0.0160	0.0036	0.0014	1.0325	0.0074	0.0029
5	0.0154	0.0037	0.0015	1.0313	0.0076	0.0031
6	0.0150	0.0036	0.0016	1.0305	0.0074	0.0033
7	0.0156	0.0033	0.0014	1.0317	0.0068	0.0029
8	0.0139	0.0035	0.0014	1.0282	0.0072	0.0029
9	0.0135	0.0038	0.0014	1.0274	0.0078	0.0029
10	0.0137	0.0042	0.0016	1.0278	0.0086	0.0033
Average	0.0148	0.0037	0.0015	1.0300	0.0076	0.0030
St. Dev.	0.0010	0.0003	0.0001	0.0021	0.0006	0.0002

Comments
Particles were heat treated to 1800°C for one hour according to DAM-41.

Grant Helmreich
Operator

4/11/17
Date

Figure 2-12. Data report for Composite 98005 Sample NP-C1498 OPyC anisotropy after heating to 1800°C.

3 COMPOSITE 98005 SAMPLE NP-C1504

Sample NP-C1504 was a 130-gram sample riffled by BWXT from AGR-5/6/7 Composite J52R-16-98005 and shipped to ORNL for determination of Defective IPyC fraction and measurement of pyrocarbon anisotropy as part of the AGR-5/6/7 irradiation test fuel QC acceptance testing.

3.1 COMPOSITE 98005 SAMPLE NP-C1504: DEFECTIVE IPYC

The number of particles with defective IPyC was determined for two subsamples from Composite 98005 Sample NP-C1504. 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 composite satisfied the specified parameters for this property. Composite 98005 Sample NP-C1504 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-C1504
Coated particle sample description:	TRISO particles from BWXT composite J52R-16-98005

Property	Measured Data	Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	# of particles	INL SPC-1352				
Defective IPyC coating fraction (fraction of total particles)	120804	$\leq 1.0 \times 10^{-4}$	≤2 with excessive U dispersion in ≥62956 particles or ≤6 with excessive U dispersion in ≥118422 particles	6	pass	DRF-47

Comments
See NP-C1504-C01_DRF47R0 and NP-C1504-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography. Sample NP-C1504-C01 failed Stage 1 testing with 3 particles with excessive U dispersion indicative of Defective IPyC out of 63905. Two other particles were noted to have localized U dispersion associated with kernel debris trapped in the buffer layer near the kernel surface. These were not counted as having Defective IPyC.

John Hunn
QC Supervisor

5-1-17
Date

M.C.
QA Reviewer

5/1/17
Date

Figure 3-1. Inspection report for Composite 98005 Sample NP-C1504 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 indicative of particles with Defective IPyC. 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 composite, 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
Particle sample ID:	NP-C1504-B00
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
Filename:	\\mc-agr\AGR\ParticleWeight\W17030202_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1095	0.1313	0.1567	0.1427	0.1201
Number of particles:	108	129	156	141	118
Average weight/particle (g):	1.014E-03	1.018E-03	1.004E-03	1.012E-03	1.018E-03

Mean average weight/particle (g):	1.013E-03
Standard error in mean average weight/particle (g):	2.45E-06

Operator

03/02/2017

Date

Figure 3-2. Data report for Composite 98005 Sample NP-C1504 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\H17030301_DRF41R0.xls

Particle loading procedure	AGR-CHAR-PIP-28-R1				
Particle weight (g)	122.3283	Additional Material	Graphite	AM weight (g)	N/A
Details	TRISO particles from BWXT composite J52R-16-98005				

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-C1504-C01 (64.7085 g)	Crucible Marking	X
Sample ID	NP-C1504-D01 (57.6198 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 Eurotherm auto mode. Set controller temperature to 1780°C to get 1800°C optical pyrometer temperature.
Optical pyrometer calibration due 5/31/17.

John Hunn

Operator

3-3-17

Date

Figure 3-3. Data report for Composite 98005 Sample NP-C1504 particle heat treatment to simulate compact heat treatment.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1504-C01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170306\NP-C1504-C01_DRF47R0.xlsm

Weight of particles in sample (g):	64.7085
Number of particles in sample:	63905
Average weight/particle (g):	1.0126E-03

Number of particles identified as having Defective IPyC:	3

Comments

Tape mounts T17030601 thru T17030619.

One additional particle was identified with uranium dispersion that was determined with high-resolution x-ray tomography to be associated with kernel debris trapped in the buffer layer near the kernel surface.

4/28/17

Operator | Date

Figure 3-4. Data report for defective IPyC analysis of Composite 98005 subsample NP-C1504-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-C1504-C01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170306\NP-C1504-C01_DRF47R0.xlsm

Number of particles in sample:	63905
Number of radiographs analyzed:	19

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	4	6.26E-05	1.5E-04
Defective-IPyC related	*3*	*4.69E-05*	*1.3E-04*
Fractured-kernel related	*1*	*1.56E-05*	*7.5E-05*
White spots	1	1.56E-05	7.5E-05
Thin or low density SiC	7	1.10E-04	2.1E-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	0	0.00E+00	4.7E-05
Particles with kernel anomalies	675	1.06E-02	1.2E-02
Dimple or facet	*638*	*9.98E-03*	*1.1E-02*
Severe dimple or facet	*10*	*1.56E-04*	*2.7E-04*
Notched kernel	*10*	*1.56E-04*	*2.7E-04*
Irregular kernel	*17*	*2.66E-04*	*4.0E-04*
Multi-kernel	*1*	*1.56E-05*	*7.5E-05*

Comments

Four particles were identified by x-ray radiography to have excessive uranium dispersion.
High-resolution x-ray tomography was used to determine that the uranium dispersion in one of these particles was localized around kernel debris trapped in the buffer layer near the kernel surface.
The uranium dispersion in the other three particles was presumed to indicate defective IPyC, as it was more symmetric and not obviously associated with kernel debris.

Grant Helmreich | 4/28/17

Operator | Date

Figure 3-5. Summary of anomalies observed during defective IPyC analysis of Composite 98005 subsample NP-C1504-C01.

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

Procedure:	AGR-CHAR-DAM-47 Rev. 0
Operator:	Grant Helmreich/Darren Skitt
Particle sample ID:	NP-C1504-D01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170306\NP-C1504-D01_DRF47R0.xlsm

Weight of particles in sample (g):	57.6198
Number of particles in sample:	56899
Average weight/particle (g):	1.0127E-03

Number of particles identified as having Defective IPyC:	3

Comments

Tape mounts T17030620 thru T17030636.

One additional particle was identified with uranium dispersion that was determined with high-resolution x-ray tomography to be associated with kernel debris trapped in the buffer layer near the kernel surface.

Operator

4/28/17

Date

Figure 3-6. Data report for defective IPyC analysis of Composite 98005 subsample NP-C1504-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-C1504-D01
Particle sample description:	TRISO particles from BWXT composite J52R-16-98005
DRF filename:	\\mc-agr\AGR\DefectiveIPyC\T170306\NP-C1504-D01_DRF47R0.xlsm

Number of particles in sample:	56899
Number of radiographs analyzed:	17

Defect or anomaly classification	Number observed	Sample fraction	Maximum source fraction at 95% confidence
Uranium dispersion	4	7.03E-05	1.4E-04
Defective-IPyC related	*3*	*5.27E-05*	*1.4E-04*
Fractured-kernel related	*1*	*1.76E-05*	*8.4E-05*
White spots	1	1.76E-05	8.4E-05
Thin or low density SiC	2	3.52E-05	1.2E-04
Extra layers	0	0.00E+00	5.3E-05
Missing kernel	0	0.00E+00	5.3E-05
Kernel migration	2	3.52E-05	1.2E-04
Missing buffer	0	0.00E+00	5.3E-05
Particles with kernel anomalies	993	1.75E-02	1.9E-02
Dimple or facet	*938*	*1.65E-02*	*1.8E-02*
Severe dimple or facet	*12*	*2.11E-04*	*3.5E-04*
Notched kernel	*37*	*6.50E-04*	*8.6E-04*
Irregular kernel	*22*	*3.87E-04*	*5.6E-04*
Multi-kernel	*3*	*5.27E-05*	*1.4E-04*

Comments

Four particles were identified by x-ray radiography to have excessive uranium dispersion.
High-resolution x-ray tomography was used to determine that the uranium dispersion in one of these particles was localized around kernel debris trapped in the buffer layer near the kernel surface.
The uranium dispersion in the other three particles was presumed to indicate defective IPyC, as it was more symmetric and not obviously associated with kernel debris.

Grant Helmreich — 4/28/17

Operator	Date

Figure 3-7. Summary of anomalies observed during defective IPyC analysis of Composite 98005 subsample NP-C1504-D01.

3.2 COMPOSITE 98005 SAMPLE NP-C1504: 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 Composite 98005 Sample NP-C1504, 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 composite satisfied the specified parameters for this property. Composite 98005 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-C1504-A01
Coated particle sample description:	TRISO particles from BWXT composite J52R-16-98005

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	0.0157	0.0011	10	1.833	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.016	pass	DRF-18
				3.981	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.020	pass	
OPyC diattenuation	0.0100	0.0007	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	0.013	pass	

Comments

See R17032701_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0318 (IPyC) and 1.0203 (OPyC).

QC Supervisor — Date: 5-1-17

QA Reviewer — Date: 5/1/17

Figure 3-8. Inspection report for Composite 98005 Sample NP-C1504 pyrocarbon anisotropy.

The data report forms in Figure 3-9 and Figure 3-10 show the average anisotropy data for each particle cross section in terms of both the diattenuation and the OPTAF. Note that the standard deviation in the measured anisotropy within each layer was greater than the standard deviation in the distribution of measured values for the ten-particle sample. This illustrates that even though there is significant localized variation in the PyC microstructure within each layer, the average PyC anisotropy is relatively consistent from particle to particle.

The data report forms in Figure 3-11 and Figure 3-12 show the average anisotropy data for each particle cross section following heat treatment at 1800°C for one hour to simulate compacting. As expected based on prior research [Hunn et al. 2007], the anisotropy of both the IPyC and OPyC layers increased following this heat treatment. The AGR-5/6/7 Fuel Specification for pyrocarbon anisotropy is defined for particles which have not undergone further heat treatment, and as such these results are provided for information only.

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

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17030203
Sample ID:	NP-C1504-A01
Sample Description:	TRISO particles from BWXT composite J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17032701\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0153	0.0034	0.0011	1.0311	0.0070	0.0023
2	0.0166	0.0043	0.0012	1.0338	0.0089	0.0025
3	0.0146	0.0043	0.0012	1.0296	0.0089	0.0025
4	0.0145	0.0028	0.0011	1.0294	0.0058	0.0023
5	0.0163	0.0030	0.0011	1.0331	0.0062	0.0023
6	0.0148	0.0030	0.0011	1.0300	0.0062	0.0023
7	0.0153	0.0030	0.0011	1.0311	0.0062	0.0023
8	0.0173	0.0028	0.0012	1.0352	0.0058	0.0025
9	0.0147	0.0022	0.0011	1.0298	0.0045	0.0023
10	0.0173	0.0026	0.0012	1.0352	0.0054	0.0025
Average	0.0157	0.0031	0.0011	1.0318	0.0065	0.0024
St. Dev.	0.0011	0.0007	0.0001	0.0023	0.0014	0.0001

Comments

Grant Helmreich

Operator

4/11/17

Date

Figure 3-9. Data report for Composite 98005 Sample NP-C1504 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:	M17030203
Sample ID:	NP-C1504-A01
Sample Description:	TRISO particles from BWXT composite J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17032701\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0106	0.0032	0.0011	1.0214	0.0065	0.0022
2	0.0103	0.0033	0.0011	1.0208	0.0067	0.0022
3	0.0104	0.0033	0.0010	1.0210	0.0067	0.0020
4	0.0103	0.0041	0.0011	1.0208	0.0084	0.0022
5	0.0088	0.0030	0.0011	1.0178	0.0061	0.0022
6	0.0095	0.0022	0.0011	1.0192	0.0045	0.0022
7	0.0094	0.0029	0.0011	1.0190	0.0059	0.0022
8	0.0103	0.0028	0.0011	1.0208	0.0057	0.0022
9	0.0098	0.0028	0.0011	1.0198	0.0057	0.0022
10	0.0110	0.0025	0.0011	1.0222	0.0051	0.0022
Average	0.0100	0.0030	0.0011	1.0203	0.0061	0.0022
St. Dev.	0.0007	0.0005	0.0000	0.0013	0.0011	0.0001

Comments

Grant Helmreich (signature) — 4/11/17

Operator — Date

Figure 3-10. Data report for Composite 98005 Sample NP-C1504 OPyC anisotropy.

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

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	Grant Helmreich
Mount ID:	M17030601
Sample ID:	NP-C1504-D01
Sample Description:	Heat-treated TRISO particles from BWXT coating batch J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17032001\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0205	0.0050	0.0013	1.0419	0.0104	0.0027
2	0.0181	0.0032	0.0013	1.0369	0.0066	0.0027
3	0.0219	0.0035	0.0013	1.0448	0.0073	0.0027
4	0.0212	0.0043	0.0014	1.0433	0.0090	0.0029
5	0.0215	0.0041	0.0016	1.0439	0.0086	0.0033
6	0.0185	0.0044	0.0015	1.0377	0.0091	0.0031
7	0.0207	0.0051	0.0016	1.0423	0.0106	0.0033
8	0.0212	0.0039	0.0015	1.0433	0.0081	0.0031
9	0.0172	0.0044	0.0016	1.0350	0.0091	0.0033
10	0.0187	0.0049	0.0014	1.0381	0.0102	0.0029
Average	0.0200	0.0043	0.0015	1.0407	0.0089	0.0030
St. Dev.	0.0017	0.0006	0.0001	0.0035	0.0013	0.0003

Comments
Particles were heat treated to 1800°C for one hour according to DAM-41.

Grant Helmreich
Operator

4/11/17
Date

Figure 3-11. Data report for Composite 98005 Sample NP-C1504 IPyC anisotropy after heating to 1800°C.

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:	M17030601
Sample ID:	NP-C1504-D01
Sample Description:	Heat-treated TRISO particles from BWXT coating batch J52R-16-98005
Folder containing data:	\\mc-agr\AGR\2-MGEM\R17032001\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0128	0.0049	0.0013	1.0259	0.0101	0.0027
2	0.0141	0.0037	0.0013	1.0286	0.0076	0.0027
3	0.0144	0.0040	0.0013	1.0292	0.0082	0.0027
4	0.0138	0.0039	0.0013	1.0280	0.0080	0.0027
5	0.0149	0.0031	0.0014	1.0303	0.0064	0.0029
6	0.0149	0.0040	0.0013	1.0303	0.0082	0.0027
7	0.0150	0.0034	0.0013	1.0305	0.0070	0.0027
8	0.0153	0.0043	0.0014	1.0311	0.0089	0.0029
9	0.0135	0.0041	0.0015	1.0274	0.0084	0.0031
10	0.0149	0.0034	0.0013	1.0303	0.0070	0.0027
Average	0.0144	0.0039	0.0013	1.0291	0.0080	0.0028
St. Dev.	0.0008	0.0005	0.0001	0.0016	0.0011	0.0001

Comments
Particles were heat treated to 1800°C for one hour according to DAM-41.

Grant Helmreich — Operator

4/11/17 — Date

Figure 3-12. Data report for Composite 98005 Sample NP-C1504 IPyC anisotropy after heating to 1800°C.

4 CONCLUSION

The analyses called out in the ORNL Product Inspection Plan for AGR-5/6/7 Coated Particles, PIP-28, were completed as part of the acceptance testing of BWXT TRISO Composite 98005. 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. The results of the QC analysis are that Composite 98005 met the AGR-5/6/7 Fuel Specification requirements for Defective IPyC and for IPyC and OPyC anisotropy. See Table 1-1 and discussion in Section 1 for a summary of the measured values and Sections 2–3 for the associated inspection report forms and data report forms that contain the detailed data.

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

5 REFERENCES

Helmreich, G.W., J.D. Hunn, D.J. Skitt, J.A. Dyer, and A.T. Schumacher. 2017. *X-ray Analysis of Defects and Anomalies in AGR-5/6/7 TRISO Particles.* ORNL/TM-2017-039. Oak Ridge, Tennessee: Oak Ridge National Laboratory.

Hunn, J.D., G.E. Jellison Jr., and R.A. Lowden. 2007. "Increase in pyrolytic carbon optical anisotropy and density during processing of coated particle fuel due to heat treatment." J. Nucl. Mater. 374: 445-452.

Hunn, J.D. 2013. *Data Acquisition Method for Counting of TRISO Particles with Excessive Uranium Dispersion Inside SiC*. AGR-CHAR-DAM-47, Revision 0. Oak Ridge, Tennessee: Oak Ridge National Laboratory.

Hunn, J.D. 2016. *Product Inspection Plan for AGR-5/6/7 Coated Particles.* AGR-CHAR-PIP-28, Revision 1. Oak Ridge, Tennessee: Oak Ridge National Laboratory.

Hunn, J.D. and G.E. Jellison Jr. 2016. *Data Acquisition Method for Measurement of Pyrocarbon Anisotropy Using the Second Generation Two-Modulator Generalized Ellipsometry Microscope*. AGR-CHAR-DAM-18, Revision 3. Oak Ridge, Tennessee: Oak Ridge National Laboratory.

Hunn, J.D., G.W. Helmreich, D.J. Skitt, J.A. Dyer, and A.T. Schumacher. 2017. *Acceptance Test Data for Candidate AGR-5/6/7 TRISO Particle Batches.* ORNL/TM-2017-036. Oak Ridge, Tennessee: Oak Ridge National Laboratory.

Jellison, G.E. Jr. and J.D. Hunn. 2008. "Optical Anisotropy Measurements of TRISO Nuclear Fuel Particle Cross sections: The Method." J. Nucl. Mater. 372: 36–44.

Kercher, A.K. 2010. *Data Acquisition Method for Heat Treatment of Loose Particles Using a Graphite Furnace*. AGR-CHAR-DAM-41, Revision 0. Oak Ridge, Tennessee: Oak Ridge National Laboratory.

Lybeck, N.J. 2016. *Statistical Sampling Plan for AGR-5/6/7 Fuel Materials.* PLN-4352, Revision 5. Idaho Falls, Idaho: Idaho National Laboratory.

Marshal, D.W. 2016. *AGR-5/6/7 Fuel Specification.* SPC-1352, Revision 7. Idaho Falls, Idaho: Idaho National Laboratory.

APPENDIX A. COMBINED RESULTS

Data from the determination of Defective IPyC and measurement of pyrocarbon anisotropy for the two samples from Composite 98005 (Samples NP-C1498 and NP-C1504) have been combined and are reported in the following inspection report forms.

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

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1498 and NP-C1504
Coated particle sample description:	TRISO particles from BWXT composite J52R-16-98005

Property	Measured Data	Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	# of particles	INL SPC-1352				
Defective IPyC coating fraction (fraction of total particles)	241822	≤ 1.0 x 10^{-4}	≤15 with excessive U dispersion in ≥230968 particles	11	pass	DRF-47

Comments

See NP-C1498-C01_DRF47R0, NP-C1498-D01_DRF47R0, NP-C1504-C01_DRF47R0, and NP-C1504-D01_DRF47R0 for individual results of defective IPyC measurement and summary of other anomalies observed by x-ray radiography.
Five other particles were noted to have localized U dispersion associated with kernel debris trapped in the buffer layer near the kernel surface (in four cases) or with a notch in the kernel surface where a section of the kernel had broken away (in the fifth case). These were not counted as having Defective IPyC.

QC Supervisor — Date: 5-1-17

QA Reviewer — Date: 5/1/17

Figure A-1. Defective IPyC Inspection Report Form IRF28-A for Composite 98005, where results from Samples NP-C1498 and NP-C1504 have been combined.

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

Procedure:	AGR-CHAR-PIP-28 Rev. 1
Coated particle sample ID:	NP-C1498-A01 and NP-C1504-A01
Coated particle sample description:	TRISO particles from BWXT composite J52R-16-98005

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	0.0153	0.0010	20	1.729	mean ≤ 0.0170	B = x + ts/√n ≤ 0.0170	0.016	pass	DRF-18
				3.295	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.019	pass	
OPyC diattenuation	0.0102	0.0006	20	1.729	mean ≤ 0.0122	B = x + ts/√n ≤ 0.0122	0.010	pass	DRF-18
				3.295	dispersion ≤0.01 ≥0.0242	D = x + ks < 0.0242	0.012	pass	

Comments

See R17031601_DRF18R3 and R17032701_DRF18R3 for full diattenuation results.
Mean OPTAF=(1+N)/(1-N) was 1.0311 (IPyC) and 1.0205 (OPyC).

QC Supervisor — Date: 5-1-17

QA Reviewer — Date: 5/1/17

Figure A-2. Pyrocarbon anisotropy Inspection Report Form IRF28-B for Composite 98005, where results from Samples NP-C1498 and NP-C1504 have been combined.