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

ORNL/TM-2008/193

Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Particle Batch LEU04-02DTF

John D. Hunn and James H. Miller

October 2009

UT-BATTELLE
ORNL-27 (4-00)

Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Particle Batch LEU04-02DTF

John D. Hunn and James H. Miller
Oak Ridge National Laboratory

This document is a compilation of coating and characterization data for the AGR-3/4 designed-to-fail (DTF) particles. The DTF coating is a high density, high anisotropy pyrocarbon coating of nominal 20 µm thickness that is deposited directly on the kernel. The purpose of this coating is to fail early in the irradiation, resulting in a controlled release of fission products which can be analyzed to provide data on fission product transport. A small number of DTF particles will be included with standard TRISO driver fuel particles in the AGR-3 and AGR-4 compacts.

The ORNL Coated Particle Fuel Development Laboratory 50-mm diameter fluidized bed coater was used to coat the DTF particles. The coatings were produced using procedures and process parameters that were developed in an earlier phase of the project as documented in "Summary Report on the Development of Procedures for the Fabrication of AGR-3/4 Design-to-Fail Particles," ORNL/TM-2008/161. Two coating runs were conducted using the approved coating parameters. NUCO425-06DTF was a final process qualification batch using natural enrichment uranium carbide/uranium oxide (UCO) kernels. After the qualification run, LEU04-02DTF was produced using low enriched UCO kernels.

Both runs were inspected and determined to meet the specifications for DTF particles in section 5 of the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1). Table 1 provides a summary of key properties of the DTF layer. For comparison purposes, an archive sample of DTF particles produced by General Atomics was characterized using identical methods. This data is also summarized in Table 1.

Table 1. Summary of DTF Coating Properties

Specified Parameters		NUCO425-06DTF	LEU04-02DTF	GA
Mean Coating Thickness (µm)	20 ± 5	23.4	20.9	20.2
Mean Deposition Rate (µm/min.)	~0.19	0.195	0.190	-
Sink/Float Coating Density (g/cc)	1.95 ± 0.05	1.928	1.937	1.944
Anisotropy (BAFo equivalent)	Not specified	1.391	1.428	1.250

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Material Flow

Low enrichment uranium carbide/uranium oxide (UCO) used for fuel particle batch

B&W identification : two samples NP-B8039 and NP-B8040 from composite G73I-14-69307

Two samples combined and renamed by ORNL : LEU04

Riffled sub-sample from LEU04 for coating : LEU04-02K

Renamed after coating : LEU04-02DTF

Natural enrichment uranium carbide/uranium oxide (UCO) used for qualification batch

B&W identification : sample NP-B7869 from composite G73Z-NU-69306

Renamed by ORNL : NUCO425

Riffled sub-sample from NUCO425 for coating : NUCO425-06K

Renamed after coating : NUCO425-06DTF

Gas Certification Sheets

This section contains copies of the certificates of analysis for the gases used to coat LEU04-02DTF and NUCO425-06DTF. Table 2 lists the cylinders used for each gas.

Table 2. Cylinder number(s) of gases used to produce DTF batches

Batch ID	Propylene	Helium	Argon
NUCO425-06DTF	9198633G	XGO00227/XGO00103	BM104495/F04959/EL22118
LEU04-02DTF	9198633G	XGO00227/XGO00103	BM104495/F04959/EL22118

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AIR LIQUIDE

TM

Customer	: A L A C Oakridge		
P.O. #	:072302	**Doc#:**	10685248-1A
Blend Type	: LIQUID CERTIFIED		
Cyl. Size	: 22LP	**Item #:**	
Cert. Date	: 7-25-2002	**Valve**	: CGA 510
Cylinder #	: **9198633G**		8992 grams

Mole	**Component**
Balance	PROPYLENE CP GRADE
	IMPURITIES:
.403 %	PROPANE

Valid Until: 24 July, 2004

NVCO425-02DTF
03DTF
04DTF
05DTF
06DTF
LEV04-02DTF

James H. Miller 10-7-08

installed 9-5-08

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AIR LIQUIDE

CERTIFICATE OF BATCH/LOT ANALYSIS

JG 8/1/08

Certification Of Batch/Lot #: ~~163AUG8625A~~ 169AUG8625A

Product: Helium **Grade: Ultra High Purity**

Customer:	OAKRIDGE	**Test Cylinder #:**	12/pk
Date of Certification:	6/10/2008	**Item Number:**	
P.O. Number:		**Valve:**	580
Document Number:		**Cylinder Size:**	44/12

ANALYSIS REPORT

Major Component	Specification	Purity
Helium Ultra High Purity	99.9990%	>99.9990%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.4 PPM
Oxygen	<2 ppm	0.9 PPM
Total Hydrocarbons	<0.5 ppm	N/D

Cylinder # XG0ØØ227

NUC0425-04DTF

Notes: 05DTF

06DTF

LEV04-02DTF

Certified By: [signature]
Name STEPHEN WILSON

Air Liquide America, L.P.
1311 New Savannah Rd. Augusta, GA 30901-3843
Phone: 706-724-8725

James H. Miller 10-7-08

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installed in parallel w/ other He 9-10-08

AIR LIQUIDE

CERTIFICATE OF BATCH/LOT ANALYSIS

Certification Of Batch/Lot #: 137AUG8628A

Product: Helium **Grade:** Ultra High Purity

Customer: UT Battelle
Date of Certification: 3-Jun
P.O. Number: 4800492868
Document Number: 30650563

Test Cylinder #: X535
Item Number: 0030A-3000
Valve: 580
Cylinder Size: 44

ANALYSIS REPORT

Major Component	Specification	Purity
Helium Ultra High Purity	99.9990%	> 99.999%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	0.5 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

Cylinder #
XG066103

NUCO 425-05DTF
NUCO 425-06DTF
LEU04-02DTF

Notes:

Certified By: Jamie Gilmore

Air Liquide America, L.P.

1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

10-7-08

ICSC-CYL-0307-W Revision: 2 Effective Date:

2

AIR LIQUIDE™

installed 9-5-08

CERTIFICATE OF ANALYSIS

Certification Of Cylinder #: **BM104495**

Product: Argon **Grade: Ultra High Purity**

Customer:	UT Battelle	**Batch/Lot #:**	216ORG8395A
Date of Certification:	8/14/2008	**Item Number:**	0013-1300
P.O. Number:	4800491160	**Valve:**	580
Document Number:	3058828	**Cylinder Size:**	44

ANALYSIS REPORT

Major Component	Specification	Purity
Argon Ultra High Purity	99.9990%	> 99.999%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.1 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

NUC0425-04DTF
-05DTF
06DTF
LEU04-02DTF

Notes:

Certified By: Jamie Gilmore

Air Liquide America, L.P.

1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

James H. Miller 10-7-08

3

installed 9-10-08

AIR LIQUIDE

CERTIFICATE OF ANALYSIS

Certification Of Cylinder #: **F04959**

Product: Argon **Grade: Ultra High Purity**

Customer:	UT Battelle	**Batch/Lot #:**	210ORG8395A
Date of Certification:	8/14/2008	**Item Number:**	0013-1300
P.O. Number:	4800491160	**Valve:**	580
Document Number:	3058828	**Cylinder Size:**	44

ANALYSIS REPORT

Major Component	Specification	Purity
Argon Ultra High Purity	99.9990%	> 99.999%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.3 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

05 DTF

NUC0425-05DTF

06 DTF

Notes:

LEU 04-02 DTF

Certified By: [signature]
Jamie Gilmore

Air Liquide America, L.P. 1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

[signature] 10-7-08

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AIR LIQUIDE

Bar Code # A31192A9701

CERTIFICATE OF ANALYSIS

Certification Of Cylinder #: **EL22118**

installed 9-19-08

Product: Argon **Grade: Ultra High Purity**

Customer:	UT Battelle	**Batch/Lot #:**	216ORG8395A
Date of Certification:	8/14/2008	**Item Number:**	0013-1300
P.O. Number:	4800491160	**Valve:**	580
Document Number:	3058828	**Cylinder Size:**	44

ANALYSIS REPORT

Major Componen	Specification	Purity
Argon Ultra High F	99.9990%	> 99.999%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.1 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

NCC0425-06DTF
LEU04-02DTF

Notes:

Certified By: Jamie Gilmore

Air Liquide America, L.P.

1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

ICSC-CYL-0307-W

Revision: 2

Effec

James H. Miller 10-7-08

Fabrication of LEU04-02DTF

ORNL AGR program coating procedure AGR-DTF-COAT-SOP-01 was used to fabricate a DTF coating on low enrichment UCO kernels using the ORNL Coated Particle Fuel Development Laboratory 50-mm-diameter fluidized bed coater. Table 3 gives a summary of the process conditions and the resulting properties of the DTF coated particles. The coating process conditions met the specifications for DTF particles in section 3.2 of the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1), with the exception of the coating gas fraction (CGF), for which a deviation request was approved (see note 1 below). A copy of the coating summary sheet from the laboratory log book is included in this section.

Table 3. Summary of DTF Coating Conditions and Results

Parameter	Specified Value	LEU04-02DTF
He (sccm)	Not specified	12,200
Propylene (sccm)	Not specified	130
TGF (sccm)	Not specified	12,330
CGF	0.011 ± 0.002 (note 1)	0.0105
Coating Temperature (°C)	1285 ± 25	1260.7
Coating Time (min.)	Not specified	110
Coating Thickness (μm)	20 ± 5	20.9
Deposition Rate (μm/min.)	~0.19	0.190
Coating Density (g/cc)	1.95 ± 0.05	1.937
Anisotropy (BAFo equiv.)	Not specified	1.428

Note 1. The coating gas fraction specification in the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1) was 0.015 ± 0.0015. A deviation request (DR-ORNL-AGR-08-01) to operate in the region 0.011 ± 0.002 was approved. This region was determined during development efforts to produce coatings of the desired properties. A copy of the approved deviation request is included in this section.

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APPENDIX K: COATING SUMMARY

Coating Run No.	LEU04-02DTF	
Description:	DTF particles	
Kernel Lot No.	LEU04-02K	
Kernel Composition	LEUCO	
Kernel Diameter (μm)		
Kernel Density (g/cm³)		
Kernel Batch Wt. (g)	76.4326	
Kernel surface area (cm²)		
Kernel volume (cm³)		
After Coating	JHM 9-26-08	
Coated Particle Batch Wt. (g)	~~76.93269~~ 79.0566	
Coating Wt. (g)	2.1240	
	DTF Parameter	**As-Processed**
Carbon		
Coating gases	He + C_3H_6	He + C_3H_6
TGF	Not specified	12330
CGF	0.015 ± 0.0015	0.0105
CGR	1 (Propylene only)	1
Temperature	1285±25°C	1260
Helium flow rate (cm³/min)	Not specified	12200
Propylene flow rate (cm³/min)	Not specified	130
Time	Not specified	110
Coating rate (μm/min)	~0.19	0.19
Coating thickness (μm)	20 ± 5	20.9
Coating Density (g/cm³)	1.95 ± 0.05	1.9372
Comments/Notes:		

Operator:	James H. Miller	Date:	10-23-08
Verified by:	[signature]	Date:	10/23/08
QAS:	[signature]	Date:	10/27/08

DEVIATION FORM

1. DEVIATION NO: **DR-** ORNL-AGR-08-01	**2. DIVISION:** Materials Science&Tech.

3. PROJECT TITLE: N/A	**4. PROGRAM:** Advanced Gas Reactor Fuel Development and Qualification
5. ITEM/ACTIVITY NAME: DTF Coating for AGR-3/4	**6. SPECIFICATION/PROCEDURE:** AGR Program/INL Specification #EDF-6638, Revision 1

7. DRAWING NO: N/A	**8. SHOP ORDER:** N/A	**9. WORK/PURCHASE:** N/A

10. Description of Deviation

Table 3.2 (Design to Fail Fuel Coating Conditions) of the referenced specification requires a coating gas fraction (CGF) for Designed to Fail (DTF) fuel particles of 0.015 ± 0.0015. The most recent NUCO pre-production run for AGR-3/4 DTF particles was actually run at 0.011, a parameter outside of the tolerance established for the CGF. Results of this run provided product within the tolerances established for DTF particles in Table 5.2 of the same specification: a coating thickness of 20 ± 5 μm and a density of 1.95 ± 0.05 Mg/m^3, as well as the desired high anisotropy. The purpose of this proposed deviation is to document the need for departing from the current CGF process parameter so that the desired coating and density properties can be consistently obtained in future runs.

11. Justification and Limitations

Though the CGF of NUCO425-05DTF did not fall within the process condition tolerance provided in Table 3.2, the coating process is reproducible and has demonstrated the ability to produce particles with the desired coating thickness and density established in Table 5.2. Further coating development in an attempt to adhere to process conditions within the specification that produce particles that meet the product specification is not thought to be feasible, and is therefore not recommended.

It is recommended that the CGF process parameter of 0.011±0.002 be used in the planned NUCO verification run, and in subsequent runs to produce the LEUCO DTF particles.

12. Requested by: J. H. Miller	**13. Title:** Change to DTF Coating Gas Fraction	**14. Date:** 9/22/08

15. Drawing/Document is to be revised ☑ **NO** ☐ **YES (If yes, list):**

Approved by:	**16. Task Leader:** J. D. Hunn	**Date:** 9-22-08	**19. Item User:** N/A	**Date:**
As Appropriate	**17. Requirement Originator:** INL Rep.	**Date:** 9-22-08	**20. Other:** N/A	**Date:**
	18. QA Group M. C. Vance	**Date:** 9/22/08	**20. Other:** N/A	**Date:**

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QC Acceptance Test of LEU04-02DTF

Product inspection plan AGR-CHAR-PIP-08 was used to characterize the LEU04-02DTF particle batch. This section contains the inspection report form (IRF-08) and associated data report forms resulting from that analysis. The LEU04-02DTF particle batch was found to meet the specifications for DTF particles in section 5 of the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1).

Open porosity, which was specified for measurement only, could not be determined due to compression of the DTF coating. This is noted in DRF-31 and described in detail in the section "Effect of layer compression on mercury porosimeter measurements".

Inspection Report Form IRF-08: AGR-3/4 Designed to Fail Coated Particle Batches

Procedure:	AGR-CHAR-PIP-08 Rev. 0
Coated particle batch ID:	LEU04-02DTF
Coated particle batch description:	AGR-3/4 DTF Particle Batch

Property	Measured Data: Mean (x)	Measured Data: Std. Dev. (s)	Measured Data: # measured (n)	k or t value	Specification INL EDF-6638 Rev. 1	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
Average DTF thickness for each particle (μm)	20.9	1.1	192	1.653	mean 20 ± 5	A = x - ts/√n ≥ 15	20.8	pass	DRF-33 DRF-34
						B = x + ts/√n ≤ 25	21.0	pass	
				2.573	dispersion ≤0.01 ≤ 8	C = x - ks > 8	18.1	pass	
DTF sink/float density (Mg/m^3)	1.9372	0.0072	25	1.711	mean 1.95 ± 0.05	A = x - ts/√n ≥ 1.90	1.935	pass	DRF-03
						B = x + ts/√n ≤ 2.00	1.940	pass	
DTF anisotropy (BAFo equivalent)	1.428	0.022	10	1.833	Measurement Only				DRF-18
DTF open porosity (ml/m^2)					Measurement Only				DRF-22 DRF-31

Comments

Open porosity value unavailable due to compression of DTF layer.

10-22-08

QC Supervisor | Date

Accept coated particle batch (Yes or No):	Yes

10/24/08

QA Reviewer | Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	Andrew K. Kercher
Sample ID:	LEU04-02DTF
Sample description:	AGR-3/4 DTF Particle Batch
Mount ID number:	M08101301
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08101601\P0810160101\

DMR calibration expiration date:	9/15/2009
Calibrated pixels/micron:	4.4833
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 300 µm in stage micrometer image (µm):	300.0

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
174	179	184	188

Approximate layer width in polish plane (µm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
202	20			

Andrew K. Kercher — October 16, 2008

Operator	Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	Andrew K. Kercher
Sample ID:	LEU04-02DTF
Sample description:	AGR-3/4 DTF Particle Batch
Mount ID number:	M08101302
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08101601\P0810160102\

DMR calibration expiration date:	9/15/2009
Calibrated pixels/micron:	4.4833
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 300 µm in stage micrometer image (µm):	300.0

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
171	175	171	174

Approximate layer width in polish plane (µm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
202	20			

Operator

October 16, 2008

Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	Andrew K. Kercher
Sample ID:	LEU04-02DTF
Sample description:	AGR-3/4 DTF Particle Batch
Mount ID number:	M08101303
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08101601\P0810160103\

DMR calibration expiration date:	9/15/2009
Calibrated pixels/micron:	4.4833
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 300 μm in stage micrometer image (μm):	300.0

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
183	194	182	191

Approximate layer width in polish plane (μm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
209	20			

Andrew K. Kercher — October 16, 2008

Operator — Date

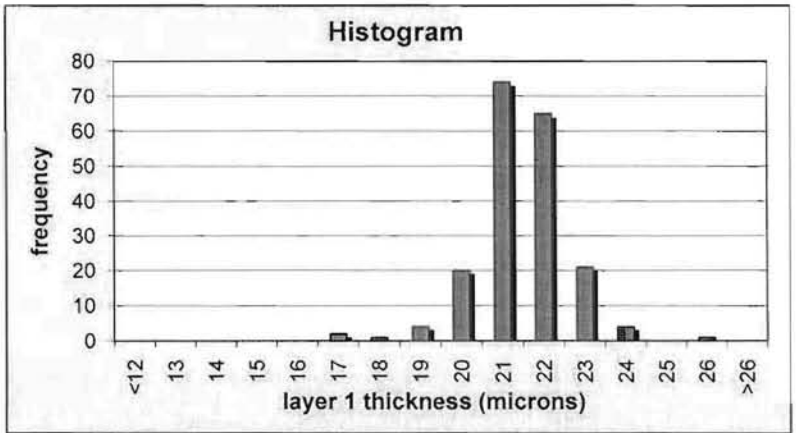
Data Report Form DRF-34A: Measurement of Layer 1 Thickness

Procedure:	AGR-CHAR-DAM-34 Rev. 0
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08101601\
Sample ID:	LEU04-02DTF
Sample Description:	AGR-3/4 DTF Particle Batch
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08101601_output\

Number of layers analyzed:	192
Mean of the average layer 1 thickness of each particle (μm):	20.9
Standard deviation in the average layer 1 thickness of each particle (μm):	1.1

Distribution of the average layer 1 thickness (top binned)

Layer 1 Thickness (μm)	*Frequency*
<12	0
13	0
14	0
15	0
16	0
17	2
18	1
19	4
20	20
21	74
22	65
23	21
24	4
25	0
26	1
>26	0



Andrew K. Kercher — October 17, 2008

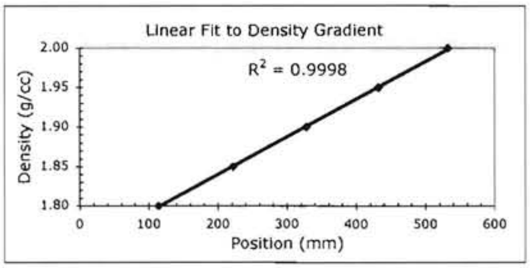
Operator | Date

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

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D080100701_DRF03R3.xls
Sample ID:	LEU04-02DTF-C01
Sample description:	AGR-3/4 DTF Particle Batch
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.0 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.800	109.87	118.05	113.96
1.850	218.06	225.78	221.92
1.900	324.75	330.69	327.72
1.950	429.02	435.89	432.46
2.000	528.67	535.71	532.19

Linear Fit			
slope	StDev	intercept	StDev
4.77E-04	2.97E-06	1.74E+00	9.74E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	381.31	1.9266	26			51		
2	381.31	1.9266	27			52		
3	387.59	1.9296	28			53		
4	381.37	1.9266	29			54		
5	388.43	1.9300	30			55		
6	389.90	1.9307	31			56		
7	392.36	1.9319	32			57		
8	387.51	1.9295	33			58		
9	391.68	1.9315	34			59		
10	398.68	1.9349	35			60		
11	403.31	1.9371	36			61		
12	406.45	1.9386	37			62		
13	409.18	1.9399	38			63		
14	410.23	1.9404	39			64		
15	403.59	1.9372	40			65		
16	406.43	1.9386	41			66		
17	406.76	1.9387	42			67		
18	409.38	1.9400	43			68		
19	410.32	1.9404	44			69		
20	419.04	1.9446	45			70		
21	422.68	1.9463	46			71		
22	422.48	1.9462	47			72		
23	421.92	1.9460	48			73		
24	426.56	1.9482	49			74		
25	431.44	1.9505	50			75		

Average density of PyC fragments:	1.9372
Standard deviation in density of PyC fragments:	0.0072
Uncertainty in calculated density of PyC fragments:	0.0016

Operator

Date

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08101301
Sample ID:	LEU04-02DTF
Sample Description:	AGR-3/4 DTF Particle Batch
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08102201\

Particle #	Grid Position	Diattenuation			Equivalent BAFo = 1+3N		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.1475	0.0174	0.0010	1.4425	0.0522	0.0030
2	4,5	0.1533	0.0127	0.0007	1.4599	0.0381	0.0021
3	4,6	0.1453	0.0192	0.0008	1.4359	0.0576	0.0024
4	5,4	0.1508	0.0129	0.0010	1.4524	0.0387	0.0030
5	5,5	0.1391	0.0156	0.0011	1.4173	0.0468	0.0033
6	5,6	0.1442	0.0196	0.0009	1.4326	0.0588	0.0027
7	6,4	0.1427	0.0131	0.0012	1.4281	0.0393	0.0036
8	6,5	0.1279	0.0118	0.0008	1.3837	0.0354	0.0024
9	6,6	0.1389	0.0187	0.0009	1.4167	0.0561	0.0027
10	5,7	0.1365	0.0169	0.0009	1.4095	0.0507	0.0027
Average		0.1426	0.0158	0.0009	1.4279	0.0474	0.0028

Mean of average BAFo per particle:	1.4279
Standard deviation of average BAFo per particle:	0.0222

Comments

G. E. Jellison
Operator

10/22/08
Date

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08101301
Sample ID:	LEU04-02DTF
Sample Description:	AGR-3/4 DTF Particle Batch
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08102201\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.1475	0.0174	0.0010	1.3460	0.0479	0.0028
2	4,5	0.1533	0.0127	0.0007	1.3621	0.0354	0.0020
3	4,6	0.1453	0.0192	0.0008	1.3400	0.0526	0.0022
4	5,4	0.1508	0.0129	0.0010	1.3552	0.0358	0.0028
5	5,5	0.1391	0.0156	0.0011	1.3232	0.0421	0.0030
6	5,6	0.1442	0.0196	0.0009	1.3370	0.0535	0.0025
7	6,4	0.1427	0.0131	0.0012	1.3329	0.0356	0.0033
8	6,5	0.1279	0.0118	0.0008	1.2933	0.0310	0.0021
9	6,6	0.1389	0.0187	0.0009	1.3226	0.0504	0.0024
10	5,7	0.1365	0.0169	0.0009	1.3162	0.0453	0.0024
Average		0.1426	0.0158	0.0009	1.3328	0.0430	0.0025

Mean of average BAFo per particle:	1.3328
Standard deviation of average BAFo per particle:	0.0201

Comments

G. E. Jellison
Operator

10/22/08
Date

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	LEU04-02DTF
Particle Lot Description:	AGR-3/4 DTF Particle Batch
Filename:	\\mc-agr\AGR\ParticleWeight\W08100801_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.0701	0.0749	0.0831	0.0862	0.0910
Number of particles:	151	160	181	188	197
Average weight/particle (g):	4.642E-04	4.681E-04	4.591E-04	4.585E-04	4.619E-04

Mean average weight/particle (g):	4.624E-04
Standard error in mean average weight/particle (g):	1.76E-06

Dixie Barker
Operator

10-8-08
Date

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

Procedure:	AGR-CHAR-DAM-31 Rev. 1
Operator:	S. D. Nunn
Coated particle batch ID:	LEU04-02DTF-E01
Batch Description:	AGR-3/4 DTF Particle Batch
Thermocouple Expiration Date:	5/15/09
Penetrometer Expiration Date:	7/10/09
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S08101401\S08101401_DRF31R1.xls

Mean average weight/particle (g):	4.62E-04
Standard error in mean average weight/particle (g):	1.76E-06

Weight of particles (g):	12.9123
Approximate number of particles:	27925
Uncertainty in number of particles:	106
Total envelope volume of sample (cc):	1.487
Average envelope volume/particle (cc):	5.33E-05
Sample envelope density (g/cc):	8.681

Average particle diameter (microns):	4.67E+02	
Average surface area/particle (cm2):	6.85E-03	
Total sample surface area (cm2):	1.91E+02	
Intruded mercury volume from 250-10,000 psia (cc):	2.38E-02	
Open porosity (ml/m2):	1.24E+00	See note

Comments
The measured intrusion volume and calculated open porosity was mostly a result of compression of the DTF coating rather than intrusion into open pores. The actual open porosity is much lower and can not be determined with this method.

S. D. Nunn
Operator

10/14/08
Date

For Information Only

The following sections provide additional information relevant to the LEU04-02DTF particle batch.

<u>Anomalies observed during inspection by optical microscopy</u>

Two anomalies were observed during optical microscopy analysis of the designed-to-fail (DTF) particle cross-sections. First, a gap was observed between the kernel and the coating. This had some impact on the image analysis for coating thickness and on the measurement of open porosity. Second, what appears to be low density soot inclusions were observed in the DTF layer.

Figure 1 and Figure 2 show typical cross-sectioned particles from LEU04-02DTF and NUCO425-06DTF batches. A gap between the kernel and DTF layer of 1-2 μm was observed on all the cross-sectioned particles. The image analysis software was not designed to account for the existence of this gap. On a few particles (~10% or less), the inner boundary of the DTF was correctly identified on the pyrocarbon side; on some particles (~40%), the boundary was almost completely identified on the kernel side; on the remaining particles (~50%), the identified boundary alternated from one side of the gap to the other. The result is that a positive bias was introduced to the average thickness measurement of approximately 1 μm. This bias does not impact the determination that the coating thickness was within the specified range. In fact, subtracting 1 μm to account for this bias brings the average thickness closer to the center of the specified range. Note that this gap was also observed in the GA DTF archive material (Figure 3). Compression of the DTF to close the gap during measurement in the mercury porosimeter made it impossible to determine the open porosity of the layer. This is discussed in the next section.

Figure 1 and Figure 2 also show a second anomaly, which was observed on most of the particles. There is a dark band close to the kernel/coating interface, which indicates an interruption in the pyrocarbon coating. This is probably a layer of lower density carbon "soot", similar to the anomalies sometimes seen in the layers of TRISO coated particles. In most case the layer was only 1-2 μm thick, although a few examples of thicker inclusions were also observed (Figure 4).

The GA archive DTF particles also showed what appear to be low density soot inclusions in the DTF layer, many of them much more severe than those observed in the ORNL particles (Figure 5 and Figure 6). Therefore, it is not surprising that these anomalies, which are related to fluidization problems, were observed in the ORNL particles, which were intentionally coated using conditions close to those use by GA, even though these conditions were determined to not be optimized for particle fluidization during preliminary testing with the ORNL 50 mm coater. Note that for the analysis of the DTF layer thickness for the GA particles, 8 particles with extremely thick inclusions were not included in the average thickness determination because this would bias the results.

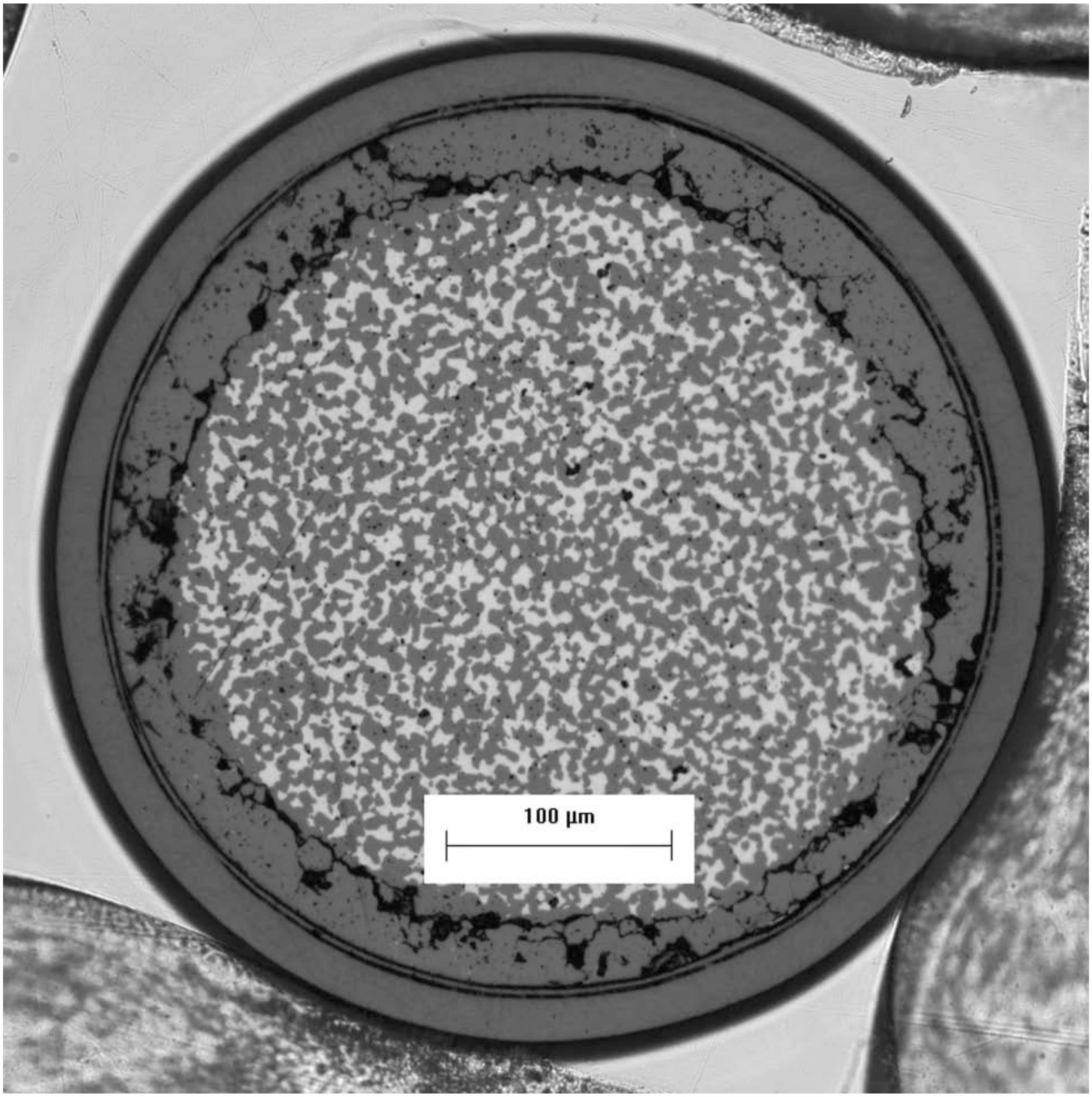


Figure 1. Cross-section of LEU04-02DTF particle.

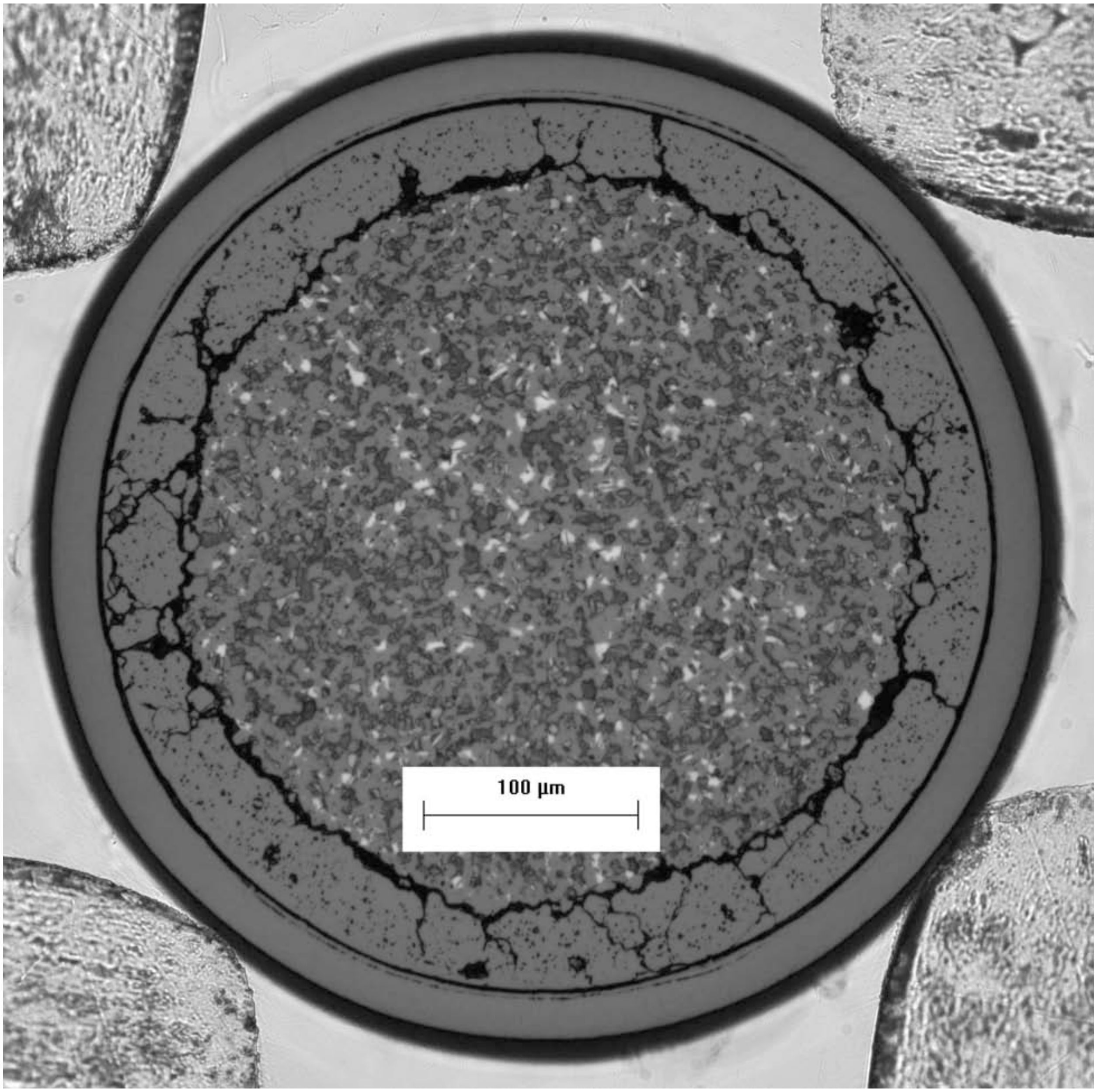


Figure 2. Cross-section of NUCO425-06DTF particle.

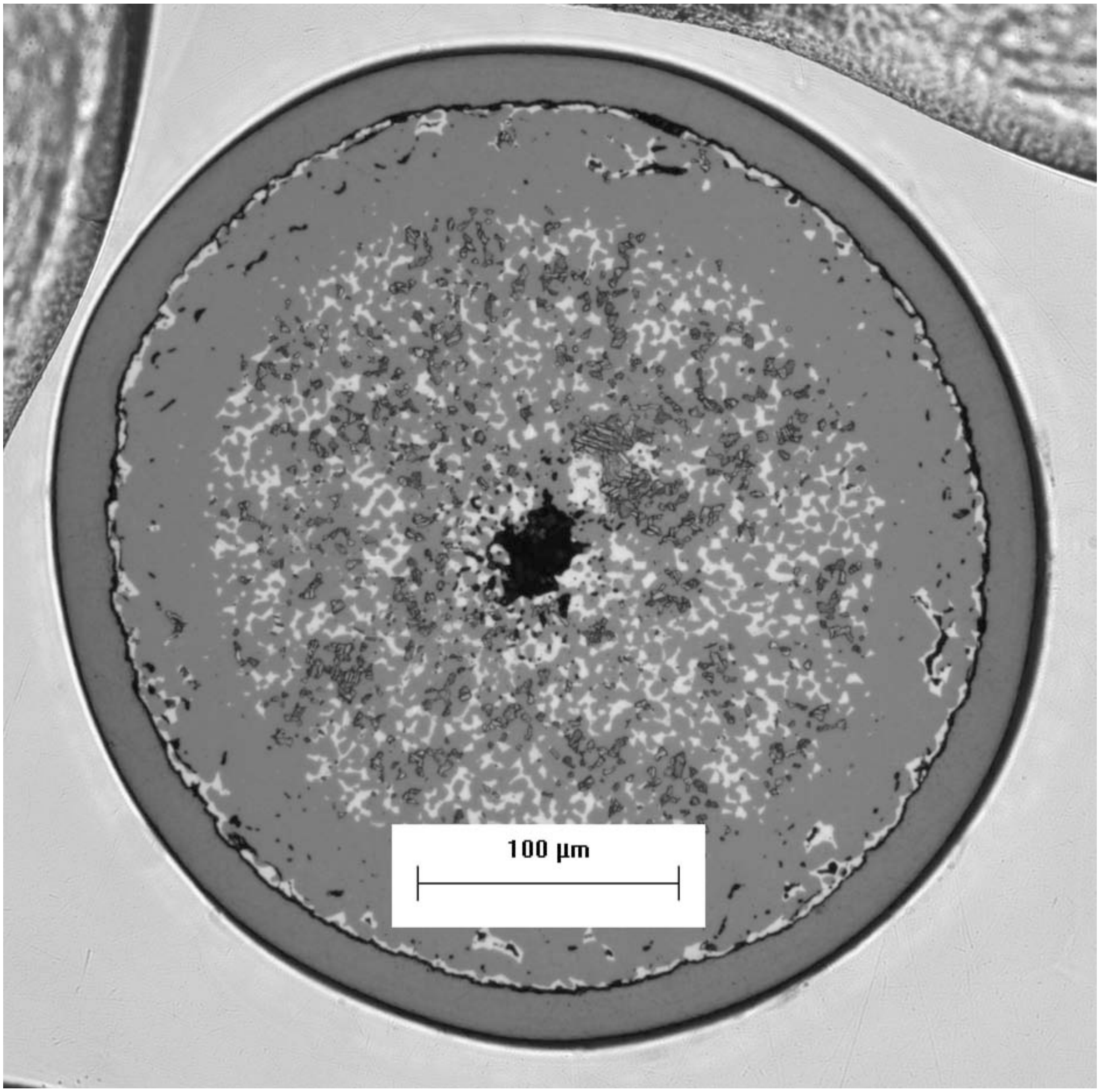


Figure 3. Cross-section of archive GA DTF particle.

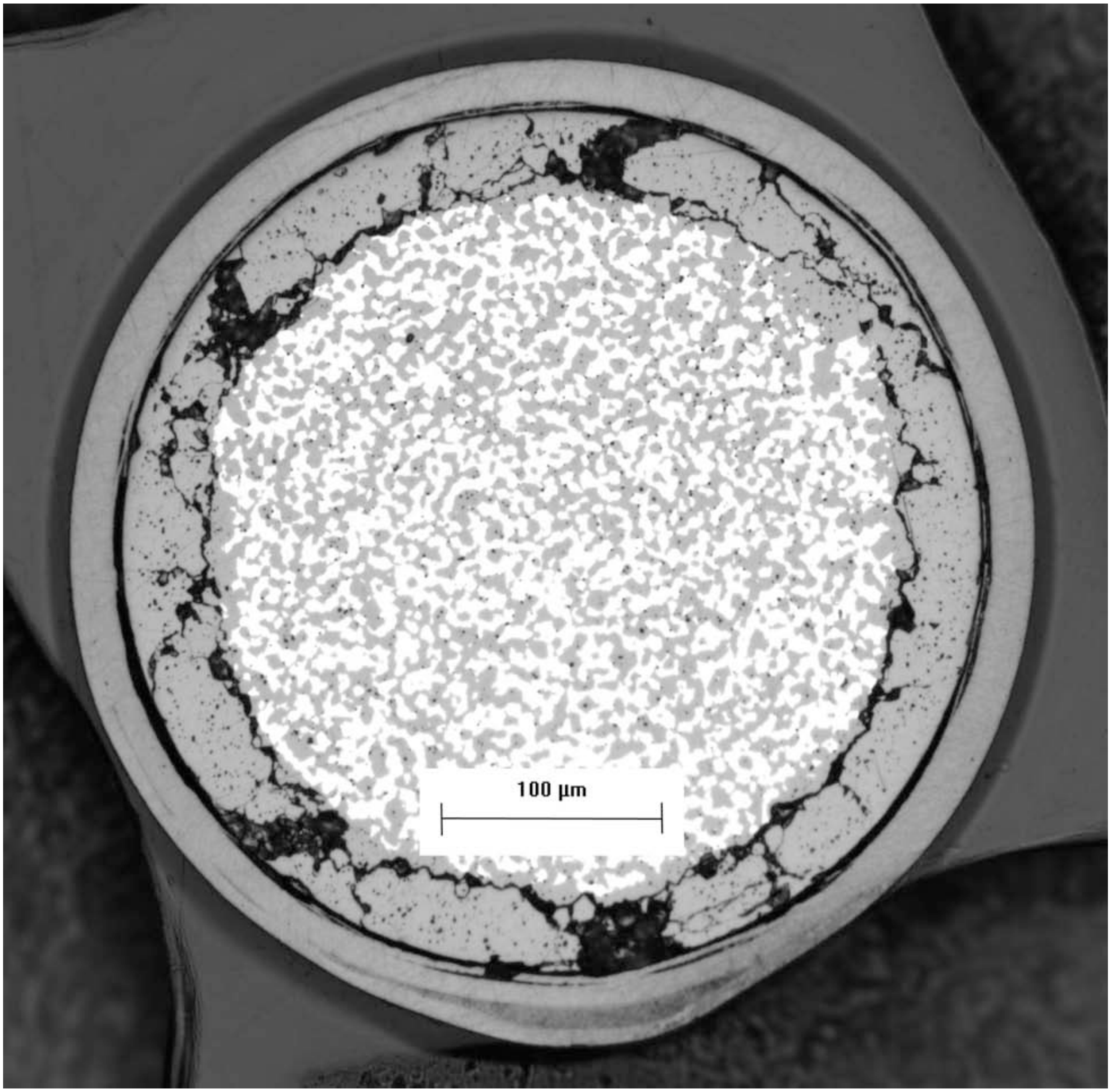


Figure 4. LEU04-02DTF particle cross section showing a thick inclusion in the DTF layer.

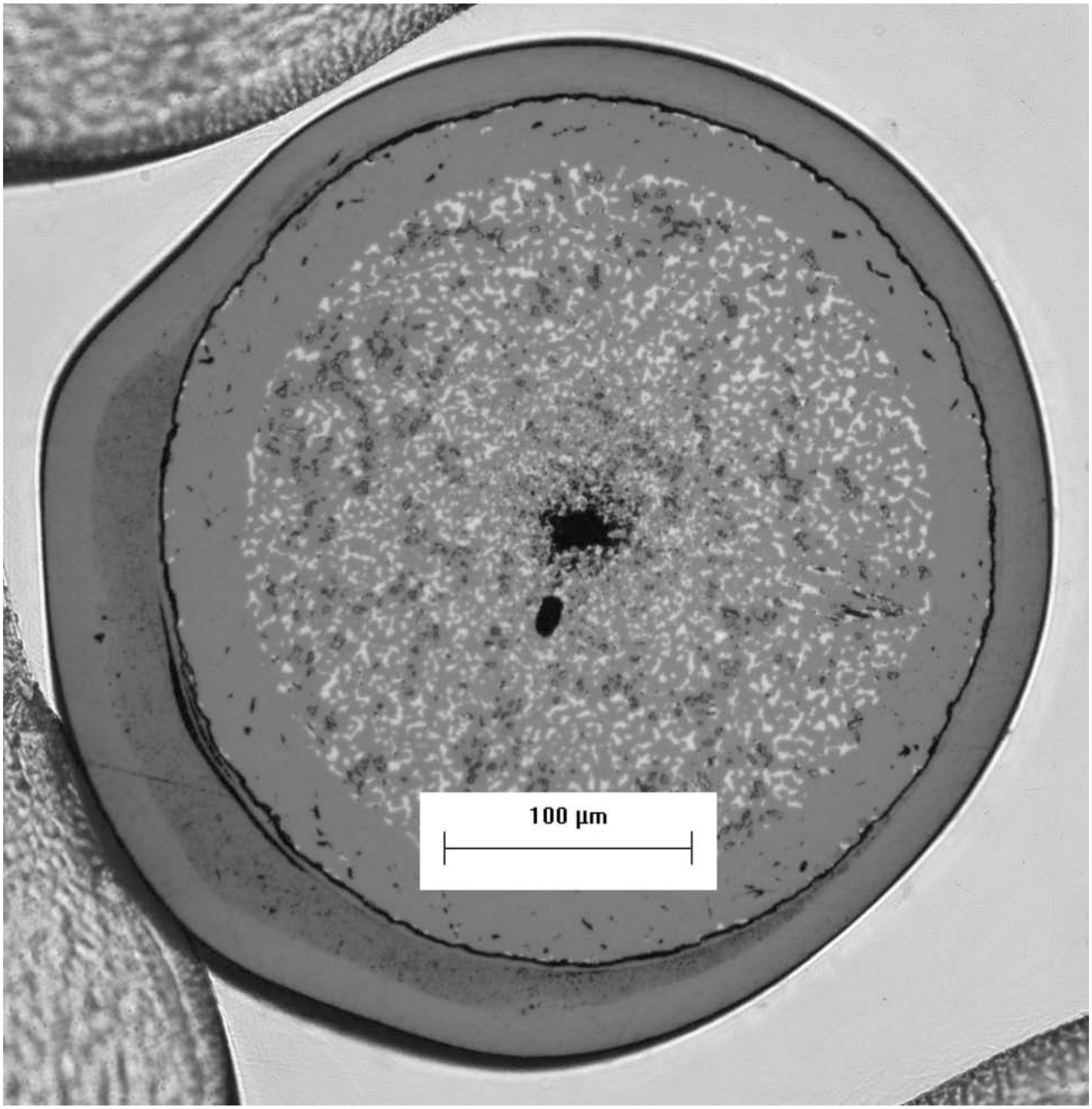


Figure 5. GA DTF with significant inclusion.

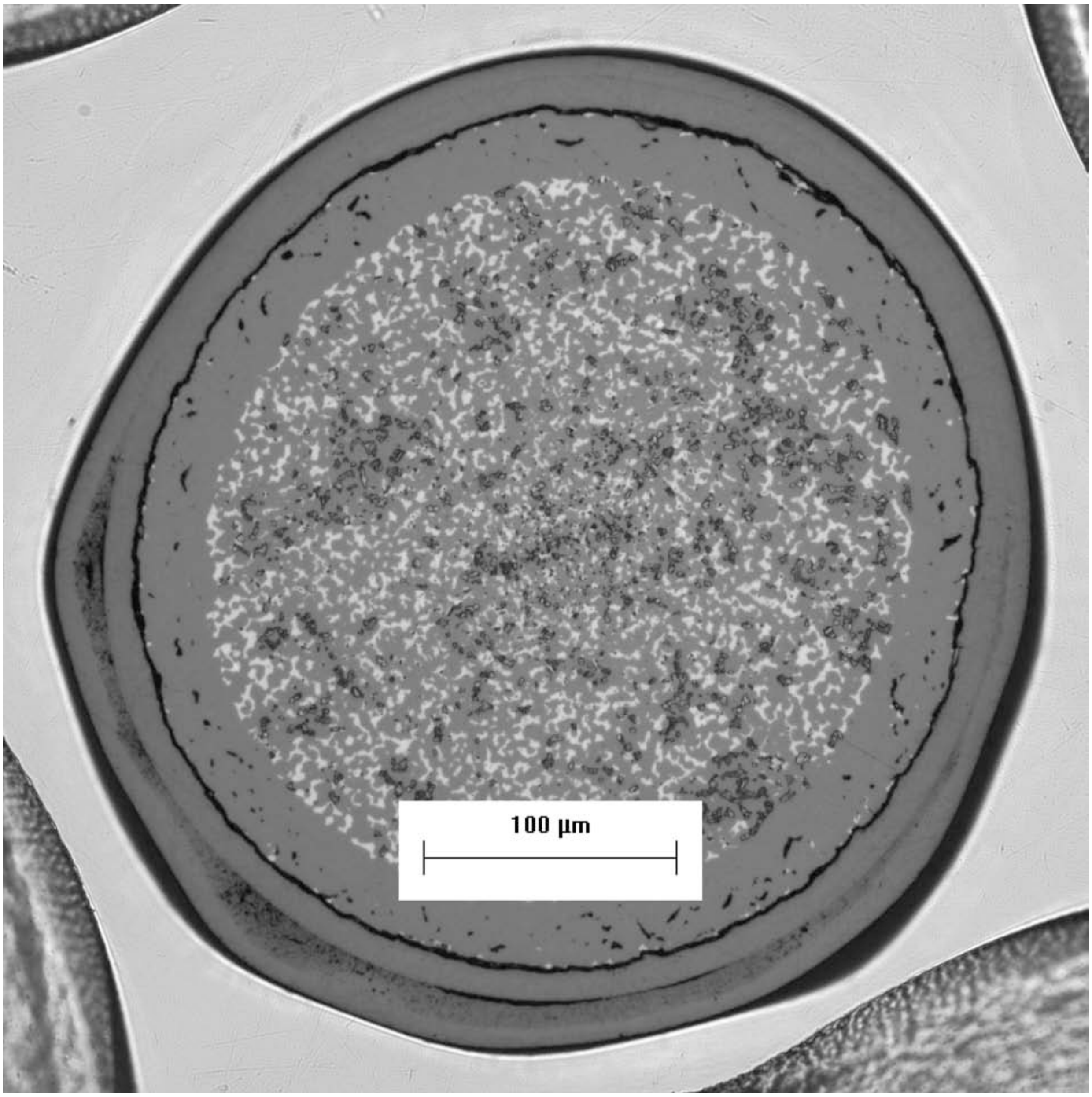


Figure 6. GA DTF with significant inclusion.

Effect of layer compression on mercury porosimeter measurements

Two batches of designed-to-fail (DTF) pyrocarbon-coated kernels were evaluated for open porosity using a mercury porosimeter. The particle batches that were examined were NUCO425-06DTF and LEU04-02DTF. The kernels used for these two batches were nominally 425 μm in diameter and the DTF was nominally 20 μm thick. Examination of the intrusion and extrusion curves from the mercury porosimeter indicated that, in a particular pressure range, the particles underwent elastic compression. This elastic compression dominated the volume change and made it impossible to determine the open porosity.

The sample of particles is placed in a penetrometer cell, which is evacuated and back-filled with mercury. Volume reduction as pressure is increased in the penetrometer is interpreted by the porosimeter software as mercury intrusion into the open porosity of a sample. However, in some cases, a portion of this volume reduction may be due to compression of the sample. This effect is observed, for instance, when measuring the open porosity of the inner pyrocarbon, due to compression of the underlying buffer layer. In contrast, compression is negligible when measuring outer pyrocarbon, where the underlying SiC is not compressible. Elastic compression is indicated by the intruded volume vs. pressure data when a plot of the data shows a linear relationship of intrusion volume versus applied pressure. Elastic compression will also show a recovery in the volume change as pressure is lowered. This extrusion curve should have about the same slope as the intrusion curve in the compression region, with an offset that is related to the actual mercury intrusion or other volume reducing effects such as inelastic compression.

For the NUCO425-06DTF particles, the volume change was approximately linear over a pressure range of about 30 to 8000 psi (Figure 7 and Figure 8). Above 8000 psi, the volume change tapered off, probably due to the cessation of compression for an increasing population of particles in the sample. Above 16000 psi, no more volume change was observed. For the LEU04-02DTF particles, the linear compression range was about 150 to 7000 psi (Figure 9 and Figure 10). Again, the volume change tapered off at higher pressure and stopped at about 16000 psi. In both cases, the linear elastic compression occurred in a pressure range that overlapped the pressure span that is used in determining the open porosity, namely 250 to 10,000 psi. Therefore, the open porosity values that would be reported using the standard procedure would be in error because they include both real open porosity and sample compression, the later being the dominant effect. For the NUCO425-06DTF sample, open porosity based on the standard procedure was 1.18 ml/m^2, for LEU04-02DTF it was 1.24 ml/m^2. However, most of this appears due to compression and the open porosity is believed to be much lower. The dense, shiny surface appearance of the DTF also indicates a low surface porosity.

The particle compression behavior that was observed in the mercury porosimeter can be explained by compression of the DTF against the kernel. Materialographic cross-sections show a narrow gap between the pyrocarbon layer and the kernel (Figure 11 and Figure 12). This gap develops because of a thermal expansion mismatch as the particles are cooled from the coating temperature. It is hypothesized that the coating layer was compressed as pressure was applied in the mercury porosimeter until the gap was closed and the coating came into direct contact with the underlying kernel surface. To corroborate this explanation, calculations were made to see if the observed compression volume can be accounted for by the volume of the gap between the coating and the kernel.

The total measured volume reduction, after low pressure filling of the space between the particles in the sample, was about 0.03 cm^3 for the NUCO425-06DTF sample and about 0.025 cm^3 for the LEU04-02DTF sample. For a kernel diameter of about 425 μm and given the number of particles in each sample, this corresponds to a gap between the outer surface of the kernel and the inner surface of the DTF of about 1.8 μm for the NUCO425-06DTF sample and about 1.5 μm for the LEU04-02DTF sample. These values are reasonable when compared to the observed gap between the kernel and DTF in the polished cross-sections.

As an additional measurement validation, the particle volume determined using the mercury porosimeter can be compared to results of other measurements. Average measured particle envelope volume was 5.50E-5 cm^3 for the NUCO425-06DTF sample and 5.33E-5 cm^3 for the LEU04-02DTF sample. For a NUCO kernel diameter of 421 μm (reported by B&W for lot G73Z-NU-69306), a gap of 1.8 μm and a coating thickness of 23.4 μm (measured by ORNL), the expected average particle volume is 5.48E-5 cm^3 for the NUCO425-06DTF sample. For a LEUCO kernel diameter of 426.5 μm (reported by B&W for lot G73I-14-69307), a gap of 1.5 μm and a coating thickness of 20.9 μm (measured by ORNL), the expected average particle volume is 5.46E-5 cm^3 for the LEU04-02DTF sample. Both these values agree within the expected uncertainties of the measurements.

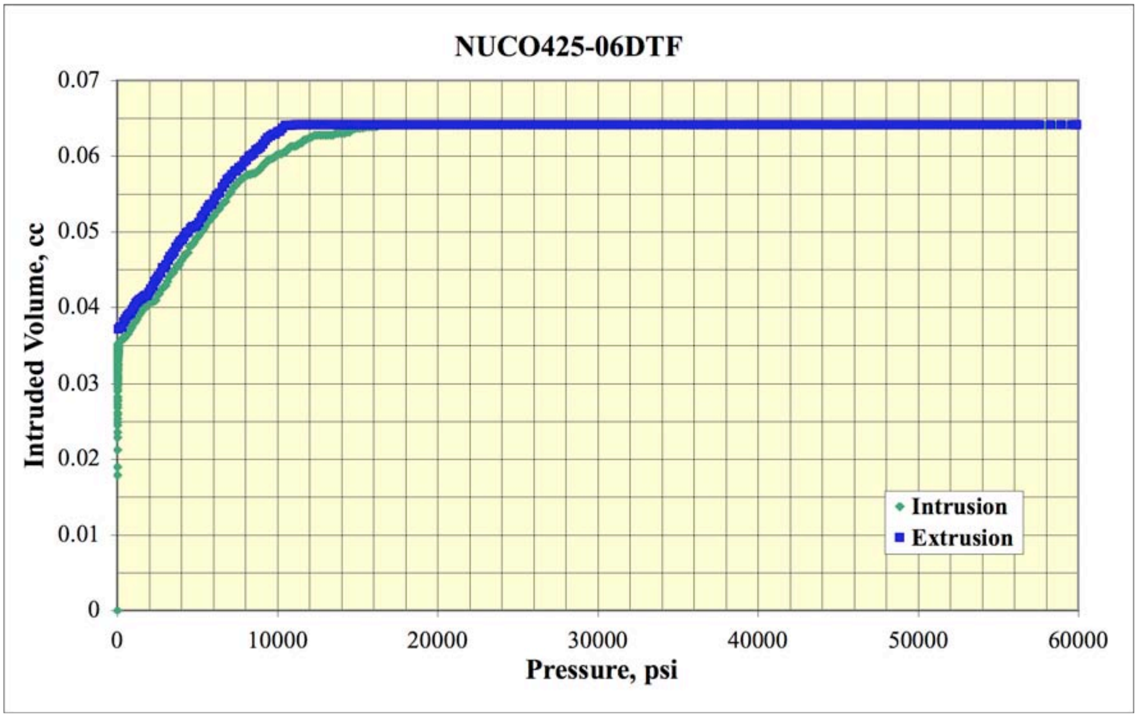


Figure 7. **Plot of the mercury porosimeter data for NUCO425-06DTF showing the linear elastic response of the intrusion and extrusion curves over the pressure range of ~30 to 8000 psi.**

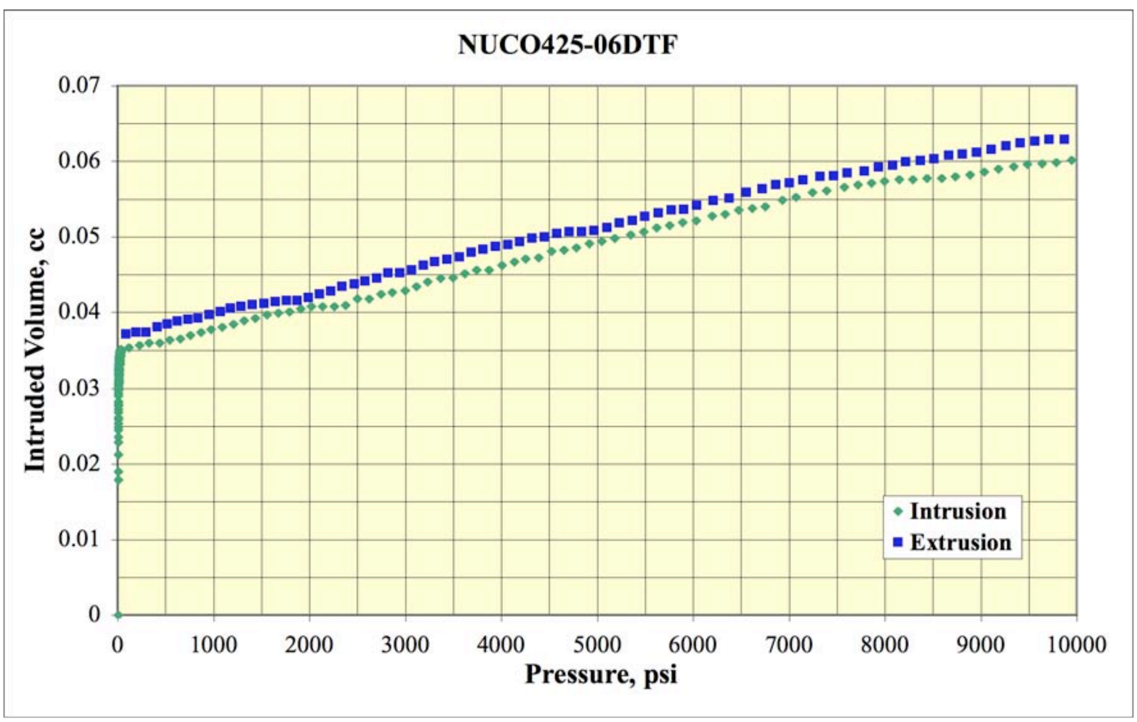


Figure 8. **Detail plot of the mercury porosimeter data for NUCO425-06DTF in the linear elastic response range.**

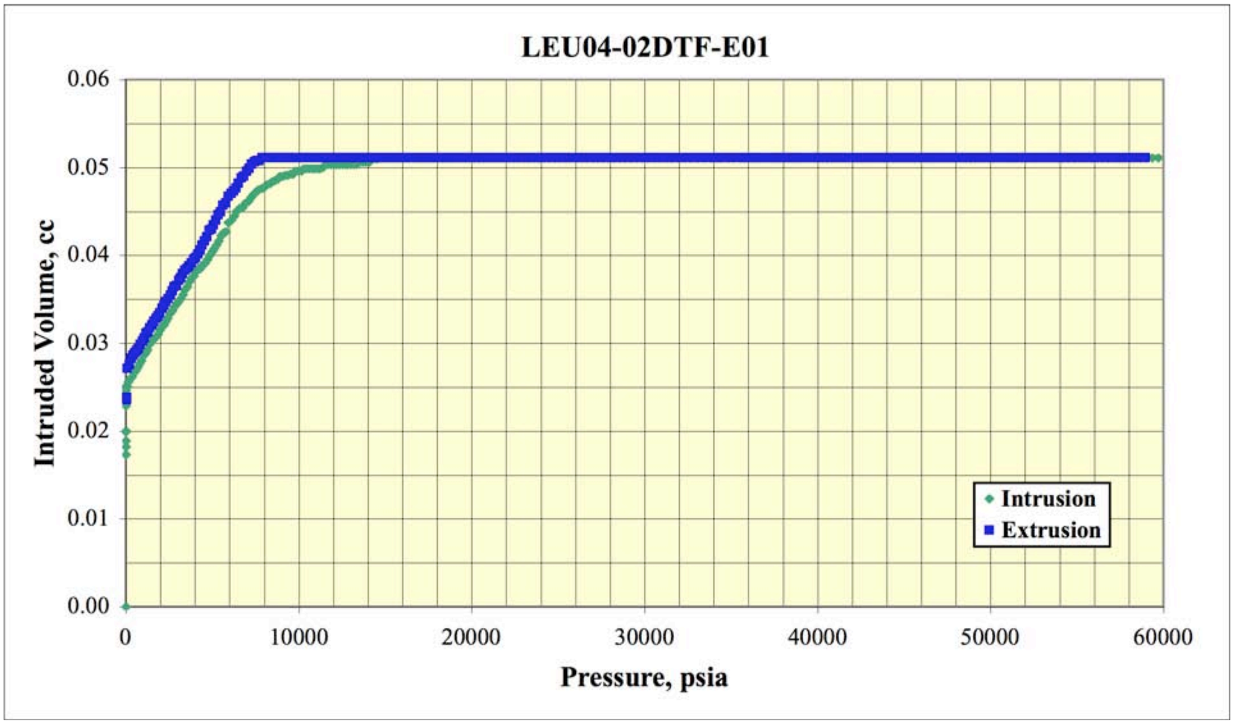


Figure 9. Plot of the mercury porosimeter data for LEU04-02DTF showing the linear elastic response of the intrusion and extrusion curves over the pressure range of ~150 to 6600 psi.

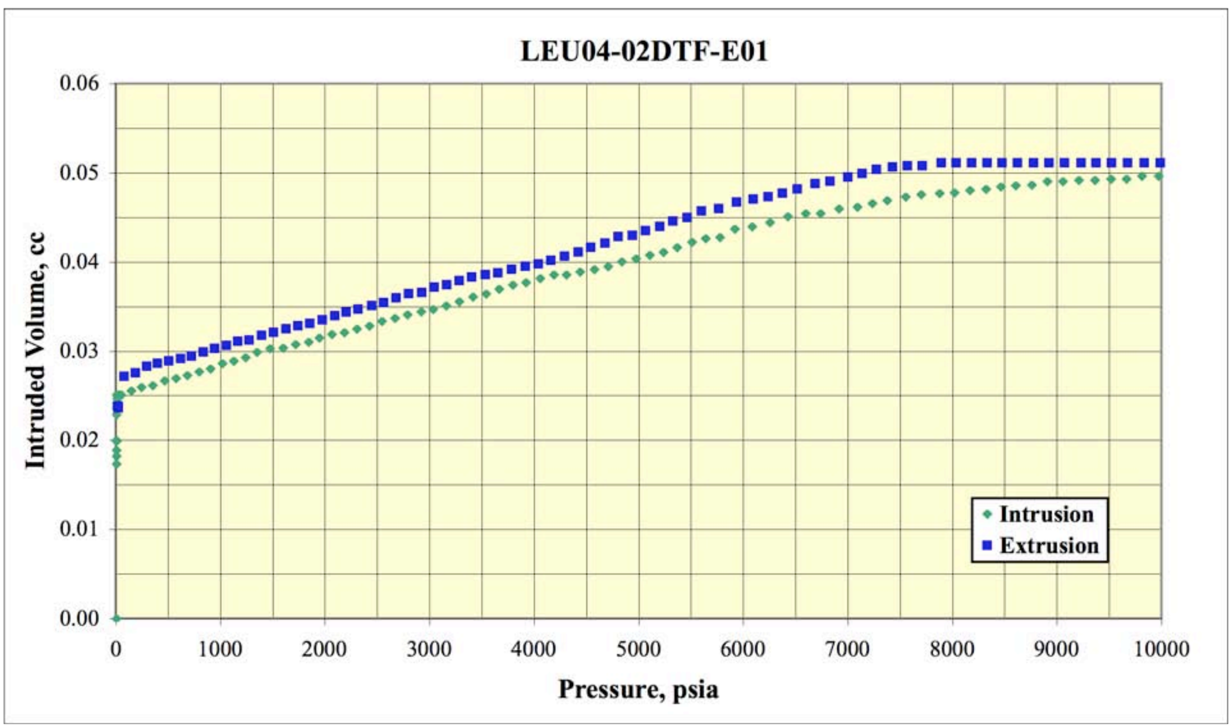


Figure 10. Detail plot of the mercury porosimeter data for LEU04-02DTF in the linear elastic response range.

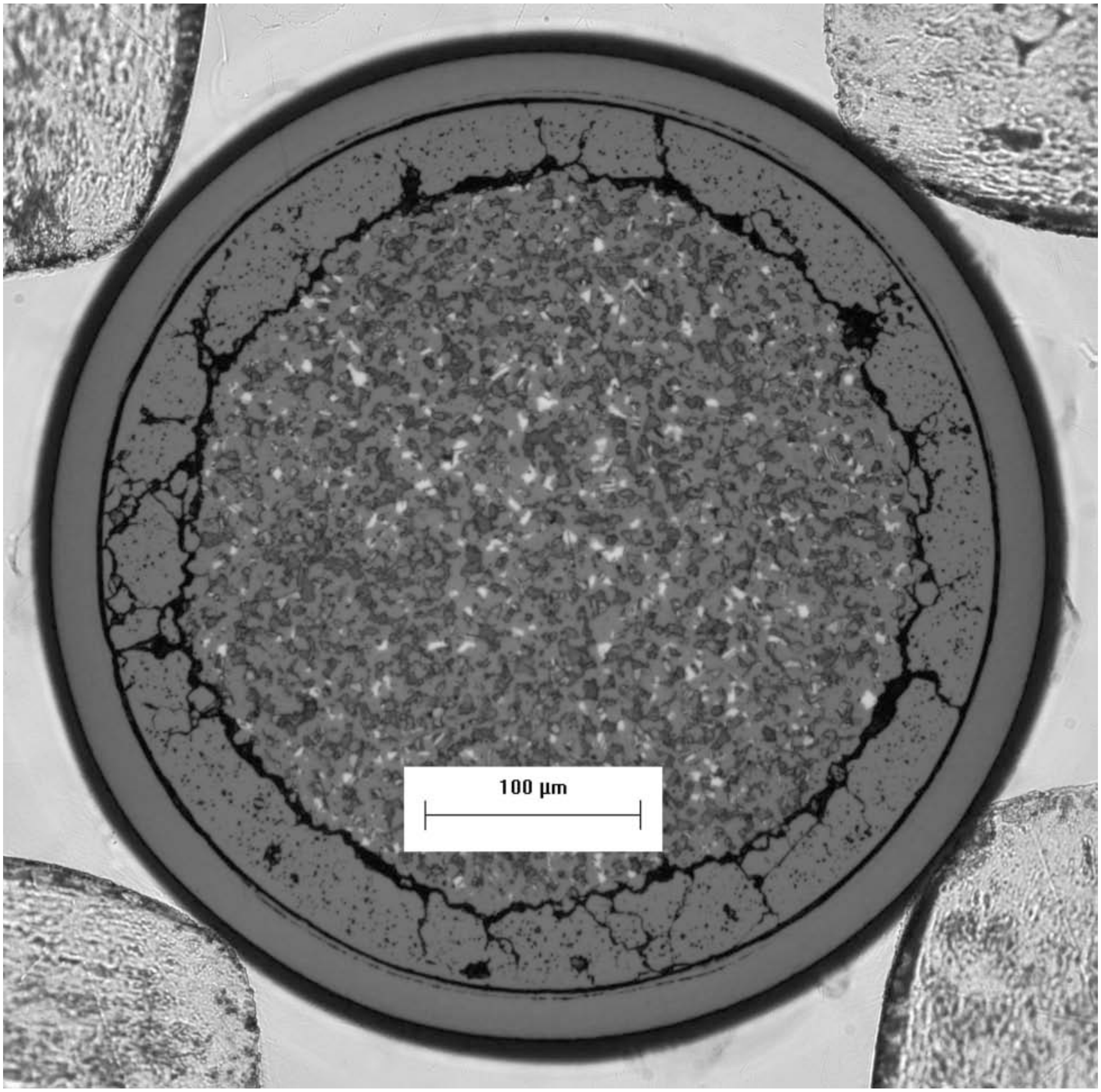


Figure 11. Cross-section of a particle from NUCO425-06DTF. A gap is evident between the kernel and the coating.

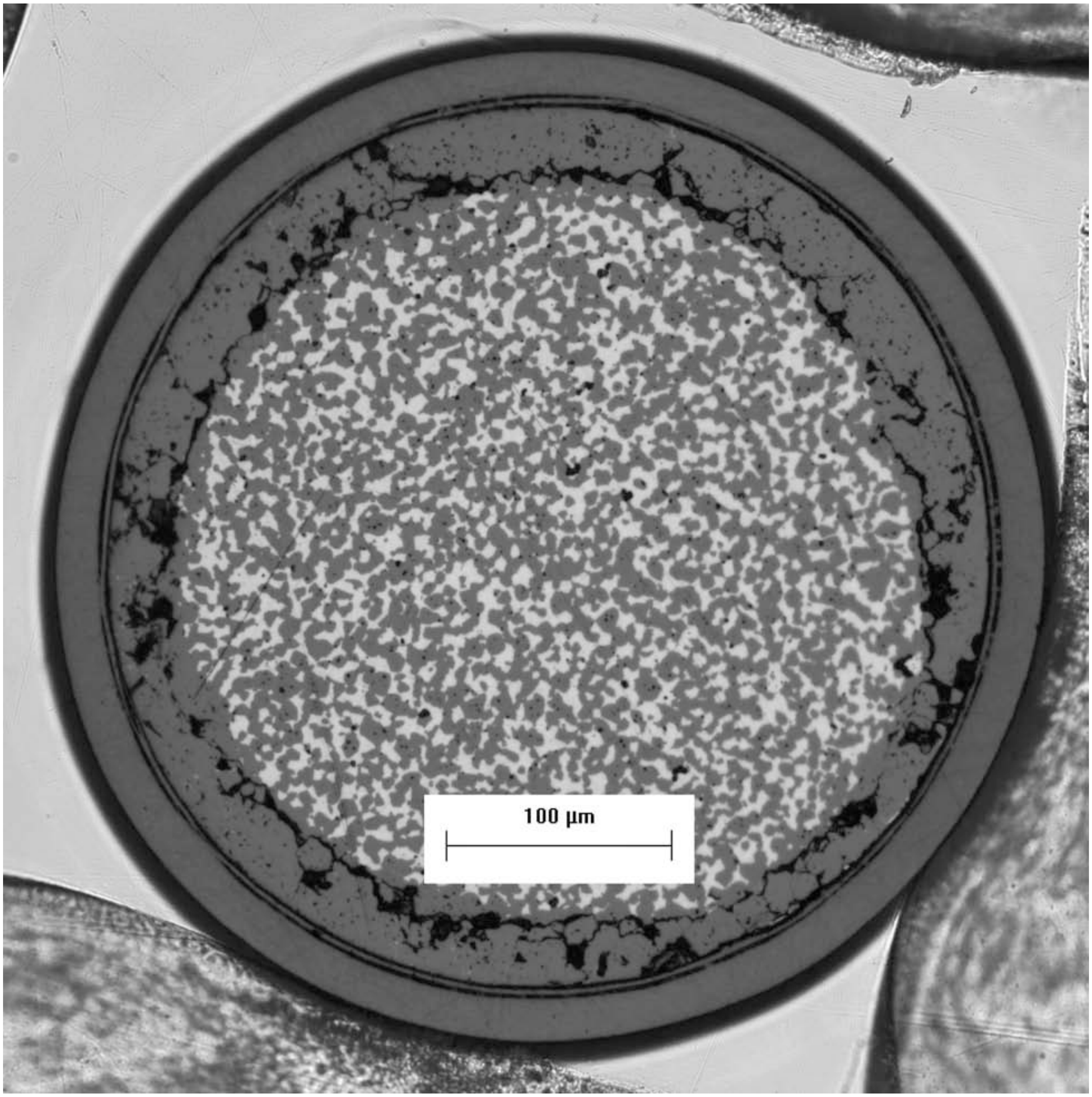


Figure 12. Cross-section of a particle from LEU04-02DTF. A gap is evident between the kernel and the coating.

Fabrication of NUCO425-06DTF

ORNL AGR program coating procedure AGR-DTF-COAT-SOP-01 was used to fabricate a DTF coating on natural enrichment UCO kernels using the ORNL Coated Particle Fuel Development Laboratory 50-mm-diameter fluidized bed coater. Table 4 gives a summary of the process conditions and the resulting properties of the DTF coated particles. The coating process conditions met the specifications for DTF particles in section 3.2 of the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1), with the exception of the coating gas fraction (CGF), for which a deviation request was approved (see note 1 below). A copy of the coating summary sheet from the laboratory log book is included in this section.

Table 4. Summary of DTF Coating Conditions and Results

Parameter	Specified Value	NUCO425-06DTF
He (sccm)	Not specified	12,200
Propylene (sccm)	Not specified	130
TGF (sccm)	Not specified	12,330
CGF	0.011 ± 0.002 (note 1)	0.0105
Coating Temperature (°C)	1285 ± 25	1260.7
Coating Time (min.)	Not specified	120
Coating Thickness (μm)	20 ± 5	23.4
Deposition Rate (μm/min.)	~0.19	0.195
Coating Density (g/cc)	1.95 ± 0.05	1.928
Anisotropy (BAFo equiv.)	Not specified	1.391

Note 1. The coating gas fraction specification in the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1) was 0.015 ± 0.0015. A deviation request (DR-ORNL-AGR-08-01) to operate in the region 0.011 ± 0.002 was approved. This region was determined during development efforts to produce coatings of the desired properties. A copy of the approved deviation request is included in this section.

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Surface Processing & Mechanics Group
Materials Science & Technology Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

AGR-DTF-COAT-SOP-01 Rev. 0
Issue Date 09/15/08
Expire Date 09/15/11
Page 20 of 20

Standard Operating Procedure
Fluidized Bed Chemical Vapor Deposition System - DTF

APPENDIX K: COATING SUMMARY

Coating Run No.	NVCO425-06DTF	
Description:	DTF particles	
Kernel Lot No.	NVCO425-06K	
Kernel Composition	NVCO	
Kernel Diameter (μm)	420.7	
Kernel Density (g/cm^3)	10.97	
Kernel Batch Wt. (g)	~~76.9639~~ 76.5762 JHM 9-24-08	
Kernel surface area (cm^2)	995.6	
Kernel volume (cm^3)	11.44	
After Coating		
Coated Particle Batch Wt. (g)	78.9730 g	
Coating Wt. (g)	2.3968 g	
	DTF Parameter	**As-Processed**
Carbon		
Coating gases	He + C_3H_6	He + C_3H_6
TGF	Not specified	12330
CGF	0.015 ± 0.0015	0.0105
CGR	1 (Propylene only)	1
Temperature	1285±25°C	1260.7
Helium flow rate (cm^3/min)	Not specified	12200
Propylene flow rate (cm^3/min)	Not specified	130
Time	Not specified	120 min
Coating rate (μm/min)	~0.19	0.195
Coating thickness (μm)	20 ± 5	23.4
Coating Density (g/cm^3)	1.95 ± 0.05	1.9279
Comments/Notes:		

Operator:	James H. Miller	Date:	9-26-08
Verified by:	[signature]	Date:	10/27/08
QAS:	[signature]	Date:	10/27/08

DEVIATION FORM

1. DEVIATION NO: **DR-** ORNL-AGR-08-01	**2. DIVISION:** Materials Science&Tech.

3. PROJECT TITLE: N/A	**4. PROGRAM:** Advanced Gas Reactor Fuel Development and Qualification
5. ITEM/ACTIVITY NAME: DTF Coating for AGR-3/4	**6. SPECIFICATION/PROCEDURE:** AGR Program/INL Specification #EDF-6638, Revision 1

7. DRAWING NO: N/A	**8. SHOP ORDER:** N/A	**9. WORK/PURCHASE:** N/A

10. Description of Deviation

Table 3.2 (Design to Fail Fuel Coating Conditions) of the referenced specification requires a coating gas fraction (CGF) for Designed to Fail (DTF) fuel particles of 0.015 ± 0.0015. The most recent NUCO pre-production run for AGR-3/4 DTF particles was actually run at 0.011, a parameter outside of the tolerance established for the CGF. Results of this run provided product within the tolerances established for DTF particles in Table 5.2 of the same specification: a coating thickness of 20 ± 5 μm and a density of 1.95 ± 0.05 Mg/m, as well as the desired high anisotropy. The purpose of this proposed deviation is to document the need for departing from the current CGF process parameter so that the desired coating and density properties can be consistently obtained in future runs.

11. Justification and Limitations

Though the CGF of NUCO425-05DTF did not fall within the process condition tolerance provided in Table 3.2, the coating process is reproducible and has demonstrated the ability to produce particles with the desired coating thickness and density established in Table 5.2. Further coating development in an attempt to adhere to process conditions within the specification that produce particles that meet the product specification is not thought to be feasible, and is therefore not recommended.

It is recommended that the CGF process parameter of 0.011±0.002 be used in the planned NUCO verification run, and in subsequent runs to produce the LEUCO DTF particles.

12. Requested by: J. H. Miller	**13. Title:** Change to DTF Coating Gas Fraction	**14. Date:** 9/22/08

15. Drawing/Document is to be revised ☑ **NO** ☐ **YES (If yes, list):**

Approved by:	**16. Task Leader:** J. D. Hunn	**Date:** 9-22-08	**19. Item User:** N/A	**Date:**
As Appropriate	**17. Requirement Originator:** INL Rep.	**Date:** 9-22-08	**20. Other:** N/A	**Date:**
	18. QA Group M. C. Vance	**Date:** 9/22/08	**20. Other:** N/A	**Date:**

ORNL-313/Page 1 of 3 (4-2003)

QC Acceptance Test of NUCO425-06DTF

Immediately prior to fabrication of the LEU04-02DTF particle batch on the low enrichment UCO kernels, NUCO425-06DTF was fabricated with natural enrichment UCO kernels using identical conditions, in order to qualify that the process would produce a product that would likely meet the specification. The full AGR-CHAR-PIP-08 product inspection plan was used to characterize the NUCO425-06DTF particle batch. This section contains the inspection report form (IRF-08) and associated data report forms resulting from that analysis. The NUCO425-06DTF particle batch was found to meet the specifications for DTF particles in section 5 of the AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev.1).

Open porosity, which was specified for measurement only, could not be determined due to compression of the DTF coating. This is noted in DRF-31 and described in detail in the section “Effect of layer compression on mercury porosimeter measurements”.

Inspection Report Form IRF-08: AGR-3/4 Designed to Fail Coated Particle Batches

Procedure:	AGR-CHAR-PIP-08 Rev. 0
Coated particle batch ID:	NUCO425-06DTF
Coated particle batch description:	AGR-3/4 DTF Particle Qualification Batch

Property	Measured Data				Specification INL EDF-6638 Rev. 1	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value					
Average DTF thickness for each particle (μm)	23.4	1.0	74	1.666	mean 20 ± 5	A = x - ts/√n ≥ 15	23.2	pass	DRF-33 DRF-34
						B = x + ts/√n ≤ 25	23.6	pass	
				2.753	dispersion ≤0.01 ≤ 8	C = x - ks > 8	20.6	pass	
DTF sink/float density (Mg/m^3)	1.9279	0.0105	30	1.699	mean 1.95 ± 0.05	A = x - ts/√n ≥ 1.90	1.925	pass	DRF-03
						B = x + ts/√n ≤ 2.00	1.931	pass	
DTF anisotropy (BAFo equivalent)	1.391	0.016	10	1.833	Measurement Only				DRF-18
DTF open porosity (ml/m^2)					Measurement Only				DRF-22 DRF-31

Comments

Open porosity value unavailable due to compression of DTF layer.

[signature] 10-07-08

QC Supervisor — Date

Accept coated particle batch (Yes or No):	Yes

[signature] 10/24/08

QA Reviewer — Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	Andrew K. Kercher
Sample ID:	NUCO425-06DTF
Sample description:	AGR-3/4 DTF Particle Qualification Batch
Mount ID number:	M08092401
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08092501\P0809250101\

DMR calibration expiration date:	9/15/2009
Calibrated pixels/micron:	4.4833
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 300 µm in stage micrometer image (µm):	299.8

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
185	183	180	171

Approximate layer width in polish plane (µm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
206	23			

Andrew K. Kercher 9/9 — September 25, 2008

Operator — Date

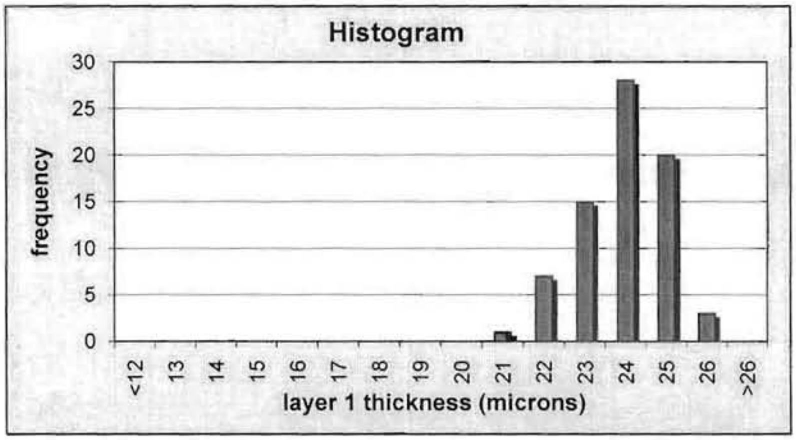
Data Report Form DRF-34A: Measurement of Layer 1 Thickness

Procedure:	AGR-CHAR-DAM-34 Rev. 0
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08092501\
Sample ID:	NUCO425-06DTF
Sample Description:	AGR-3/4 DTF Particle Qualification Batch
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08092501_output\

Number of layers analyzed:	74
Mean of the average layer 1 thickness of each particle (µm):	23.4
Standard deviation in the average layer 1 thickness of each particle (µm):	1.0

Distribution of the average layer 1 thickness (top binned)

Layer 1 Thickness (µm)	*Frequency*
<12	0
13	0
14	0
15	0
16	0
17	0
18	0
19	0
20	0
21	1
22	7
23	15
24	28
25	20
26	3
>26	0



Andrew K. Kercher
Operator

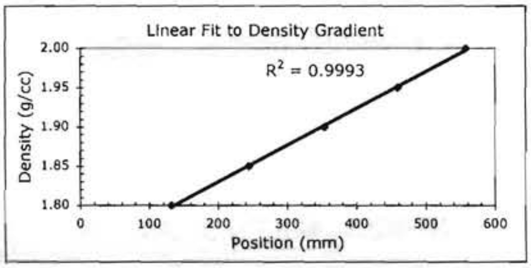
September 26, 2008
Date

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

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08092501_DRF03R3.xls
Sample ID:	NUCO425-06DTF-C01
Sample description:	AGR-3/4 DTF Particle Qualification Batch
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	22.8 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.800	127.76	137.28	132.52
1.850	240.53	248.99	244.76
1.900	351.02	356.65	353.84
1.950	455.45	462.77	459.11
2.000	553.69	560.79	557.24

Linear Fit			
slope	StDev	intercept	StDev
4.70E-04	2.85E-06	1.74E+00	1.07E-03



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	386.72	1.9175	26	422.23	1.9342	51		
2	388.02	1.9181	27	428.75	1.9372	52		
3	391.39	1.9197	28	455.78	1.9499	53		
4	392.94	1.9204	29	478.08	1.9604	54		
5	393.43	1.9206	30	468.56	1.9559	55		
6	394.92	1.9213	31			56		
7	394.99	1.9214	32			57		
8	395.85	1.9218	33			58		
9	396.15	1.9219	34			59		
10	394.94	1.9213	35			60		
11	396.76	1.9222	36			61		
12	397.61	1.9226	37			62		
13	399.91	1.9237	38			63		
14	399.91	1.9237	39			64		
15	401.11	1.9242	40			65		
16	402.43	1.9249	41			66		
17	403.88	1.9255	42			67		
18	404.33	1.9258	43			68		
19	407.29	1.9271	44			69		
20	408.27	1.9276	45			70		
21	408.81	1.9279	46			71		
22	409.67	1.9283	47			72		
23	411.79	1.9293	48			73		
24	412.06	1.9294	49			74		
25	417.55	1.9320	50			75		

Average density of PyC fragments:	1.9279
Standard deviation in density of PyC fragments:	0.0105
Uncertainty in calculated density of PyC fragments:	0.0017

Dixie Barker — Operator

9-25-08 — Date

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08092401
Sample ID:	NUCO425-06DTF
Sample Description:	AGR-3/4 DTF Particle Qualification Batch
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08100201\

Particle #	Grid Position	Diattenuation			Equivalent BAFo = 1+3N		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.1243	0.0118	0.0009	1.3729	0.0354	0.0027
2	4,5	0.1253	0.0119	0.0009	1.3759	0.0357	0.0027
3	4,6	0.1243	0.0135	0.0008	1.3729	0.0405	0.0024
4	5,4	0.1310	0.0206	0.0011	1.3930	0.0618	0.0033
5	5,5	0.1317	0.0150	0.0011	1.3951	0.0450	0.0033
6	5,6	0.1376	0.0132	0.0011	1.4128	0.0396	0.0033
7	6,4	0.1296	0.0182	0.0011	1.3888	0.0546	0.0033
8	6,5	0.1371	0.0129	0.0010	1.4113	0.0387	0.0030
9	6,6	0.1364	0.0145	0.0011	1.4092	0.0435	0.0033
10	5,7	0.1246	0.0146	0.0011	1.3738	0.0438	0.0033
Average		0.1302	0.0146	0.0010	1.3906	0.0439	0.0031

Mean of average BAFo per particle:	1.3906
Standard deviation of average BAFo per particle:	0.0164

Comments

G. E. Jellison
Operator

10-6-2008
Date

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08092401
Sample ID:	NUCO425-06DTF
Sample Description:	AGR-3/4 DTF Particle Qualification Batch
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08100201\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.1243	0.0118	0.0009	1.2839	0.0308	0.0023
2	4,5	0.1253	0.0119	0.0009	1.2865	0.0311	0.0024
3	4,6	0.1243	0.0135	0.0008	1.2839	0.0352	0.0021
4	5,4	0.1310	0.0206	0.0011	1.3015	0.0546	0.0029
5	5,5	0.1317	0.0150	0.0011	1.3034	0.0398	0.0029
6	5,6	0.1376	0.0132	0.0011	1.3191	0.0355	0.0030
7	6,4	0.1296	0.0182	0.0011	1.2978	0.0480	0.0029
8	6,5	0.1371	0.0129	0.0010	1.3178	0.0346	0.0027
9	6,6	0.1364	0.0145	0.0011	1.3159	0.0389	0.0029
10	5,7	0.1246	0.0146	0.0011	1.2847	0.0381	0.0029
Average		0.1302	0.0146	0.0010	1.2994	0.0387	0.0027

Mean of average BAFo per particle:	1.2994
Standard deviation of average BAFo per particle:	0.0145

Comments

G. E. Jellison
Operator

10-6-2008
Date

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	NUCO425-06DTF
Particle Lot Description:	AGR-3/4 DTF Particle Qualification Batch
Filename:	\\mc-agr\AGR\ParticleWeight\W08100802_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1110	0.1193	0.1120	0.1164	0.1106
Number of particles:	245	262	248	257	244
Average weight/particle (g):	4.531E-04	4.553E-04	4.516E-04	4.529E-04	4.533E-04

Mean average weight/particle (g):	4.532E-04
Standard error in mean average weight/particle (g):	6.00E-07

Dixie Barker
Operator

10-8-08
Date

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

Procedure:	AGR-CHAR-DAM-31 Rev. 1
Operator:	S. D. Nunn
Coated particle batch ID:	NUCO425-06DTF-E01
Batch Description:	AGR-3/4 DTF Particle Qualification Batch
Thermocouple Expiration Date:	5/15/09
Penetrometer Expiration Date:	7/10/09
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S08100601\S08100601_DRF31R1.xls

Mean average weight/particle (g):	4.53E-04
Standard error in mean average weight/particle (g):	6.00E-07

Weight of particles (g):	13.3968
Approximate number of particles:	29560
Uncertainty in number of particles:	39
Total envelope volume of sample (cc):	1.625
Average envelope volume/particle (cc):	5.50E-05
Sample envelope density (g/cc):	8.244

Average particle diameter (microns):	4.72E+02	
Average surface area/particle (cm2):	6.99E-03	
Total sample surface area (cm2):	2.07E+02	
Intruded mercury volume from 250-10,000 psia (cc):	2.44E-02	
Open porosity (ml/m2):	1.18E+00	See note

Comments
The measured intrusion volume and calculated open porosity was mostly a result of compression of the DTF coating rather than intrusion into open pores. The actual open porosity is much lower and can not be determined with this method.

S. D. Nunn
Operator

10/6/08
Date

Characterization of GA Archive DTF Particles

Product inspection plan AGR-CHAR-PIP-08 was used as a guideline to characterize an archive sample of DTF particles. These particles were identified as having been produced by General Atomics (GA). An identification number of 8662-133 was on the label of the container of particles. This section contains the inspection report form (IRF-08) and associated data report forms resulting from that analysis.

Open porosity was not measured on this sample due to the limited number of particles available and the previous results on other DTF particles, which indicated that the measurement was not possible. Communication with John Saurwein at GA determined that no historical data was available on open porosity for these particles, and that such analysis was probably not performed.

Inspection Report Form IRF-08: AGR-3/4 Designed to Fail Coated Particle Batches

Procedure:	AGR-CHAR-PIP-08 Rev. 0
Coated particle batch ID:	8662-133
Coated particle batch description:	GA Archive DTF particles

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 EDF-6638 Rev. 1				
Average DTF thickness for each particle (μm)	20.2	1.0	72	1.667	mean 20 ± 5	$A = x - ts/\sqrt{n} \geq 15$	20.0	pass	DRF-33 DRF-34
						$B = x + ts/\sqrt{n} \leq 25$	20.4	pass	
				2.753	dispersion ≤0.01 ≤ 8	$C = x - ks > 8$	17.4	pass	
DTF sink/float density (Mg/m^3)	1.9441	0.0064	25	1.711	mean 1.95 ± 0.05	$A = x - ts/\sqrt{n} \geq 1.90$	1.942	pass	DRF-03
						$B = x + ts/\sqrt{n} \leq 2.00$	1.946	pass	
DTF anisotropy (BAFo equivalent)	1.250	0.016	10	1.833	Measurement Only				DRF-18
DTF open porosity (ml/m^2)					Measurement Only				DRF-22 DRF-31

Comments

Open porosity not measured because insufficient archive material was available.

[signature] 10-22-08

QC Supervisor **Date**

Accept coated particle batch (Yes or No): Not Applicable

[signature] 10/24/08

QA Reviewer **Date**

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	Andrew K. Kercher
Sample ID:	8662-133
Sample description:	GA Archive DTF Particles
Mount ID number:	M08091201
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P08100801\P0810080101\

DMR calibration expiration date:	9/15/2009
Calibrated pixels/micron:	4.4833
Stage micrometer calibration expiration date:	2/13/2009
Measured value for 300 µm in stage micrometer image (µm):	299.8

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
169	166	162	157

Approximate layer width in polish plane (µm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
169	20			

Andrew K. Kercher
Operator

October 8, 2008
Date

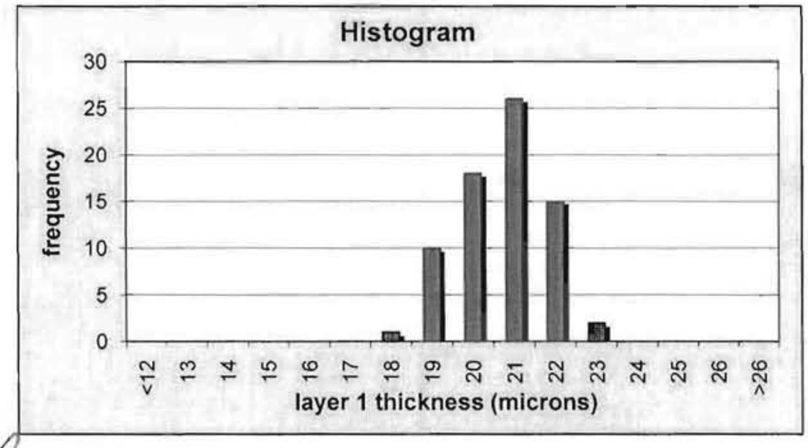
Data Report Form DRF-34A: Measurement of Layer 1 Thickness

Procedure:	AGR-CHAR-DAM-34 Rev. 0
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100801\
Sample ID:	8662-133
Sample Description:	GA Archive DTF particles
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P08100801_output\

Number of layers analyzed:	72
Mean of the average layer 1 thickness of each particle (μm):	20.2
Standard deviation in the average layer 1 thickness of each particle (μm):	1.0

Distribution of the average layer 1 thickness (top binned)

Layer 1 Thickness (μm)	*Frequency*
<12	0
13	0
14	0
15	0
16	0
17	0
18	1
19	10
20	18
21	26
22	15
23	2
24	0
25	0
26	0
>26	0



Operator

October 9, 2008

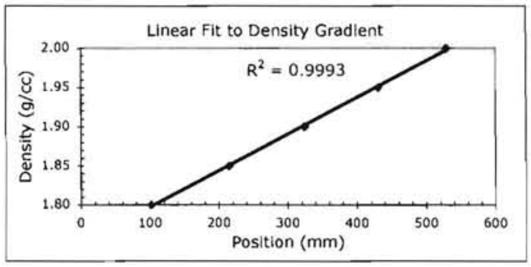
Date

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

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D08100901_DRF03R3.xls
Sample ID:	8662-133
Sample description:	GA Archive DTF Particles
Float expiration date:	07/2012
Gauge expiration date:	11/2008
Bath temperature:	23.5 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.800	97.71	106.10	101.91
1.850	210.35	218.52	214.44
1.900	320.91	326.88	323.90
1.950	427.13	434.08	430.61
2.000	525.13	532.00	528.57

Linear Fit			
slope	StDev	intercept	StDev
4.67E-04	2.82E-06	1.75E+00	9.76E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	384.01	1.9300	26			51		
2	392.58	1.9340	27			52		
3	395.69	1.9354	28			53		
4	397.41	1.9362	29			54		
5	401.54	1.9381	30			55		
6	405.54	1.9400	31			56		
7	405.10	1.9398	32			57		
8	405.95	1.9402	33			58		
9	407.79	1.9411	34			59		
10	410.97	1.9426	35			60		
11	412.25	1.9432	36			61		
12	414.98	1.9444	37			62		
13	414.98	1.9444	38			63		
14	416.77	1.9453	39			64		
15	418.13	1.9459	40			65		
16	421.53	1.9475	41			66		
17	425.47	1.9493	42			67		
18	426.45	1.9498	43			68		
19	425.73	1.9495	44			69		
20	427.60	1.9503	45			70		
21	432.73	1.9527	46			71		
22	432.58	1.9527	47			72		
23	436.87	1.9547	48			73		
24	425.25	1.9492	49			74		
25	421.30	1.9474	50			75		

Average density of PyC fragments:	1.9441
Standard deviation in density of PyC fragments:	0.0064
Uncertainty in calculated density of PyC fragments:	0.0016

Dixie Barker
Operator

10-5-08
Date

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08091201
Sample ID:	8862-133
Sample Description:	GA Archive DTF particles
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08091901\

Particle #	Grid Position	Diattenuation			Equivalent BAFo = 1+3N		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0919	0.0124	0.0009	1.2757	0.0372	0.0027
2	4,5	0.0851	0.0132	0.0010	1.2553	0.0396	0.0030
3	4,6	0.0839	0.0138	0.0008	1.2517	0.0414	0.0024
4	4,7	0.0920	0.0112	0.0008	1.2760	0.0336	0.0024
5	5,4	0.0778	0.0167	0.0011	1.2334	0.0501	0.0033
6	5,5	0.0775	0.0109	0.0010	1.2325	0.0327	0.0030
7	5,6	0.0813	0.0111	0.0009	1.2439	0.0333	0.0027
8	5,7	0.0813	0.0108	0.0009	1.2439	0.0324	0.0027
9	6,5	0.0849	0.0160	0.0011	1.2547	0.0480	0.0033
10	6,6	0.0791	0.0157	0.0010	1.2373	0.0471	0.0030
Average		0.0835	0.0132	0.0010	1.2504	0.0395	0.0029

Mean of average BAFo per particle:	1.2504
Standard deviation of average BAFo per particle:	0.0157

Comments

G. E. Jellison
Operator

9/19/08
Date

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M08091201
Sample ID:	8862-133
Sample Description:	GA Archive DTF particles
Folder containing data:	\\mc-agr\AGR\2-MGEM\R08091901\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0919	0.0124	0.0009	1.2024	0.0301	0.0022
2	4,5	0.0851	0.0132	0.0010	1.1860	0.0315	0.0024
3	4,6	0.0839	0.0138	0.0008	1.1832	0.0329	0.0019
4	4,7	0.0920	0.0112	0.0008	1.2026	0.0272	0.0019
5	5,4	0.0778	0.0167	0.0011	1.1687	0.0393	0.0026
6	5,5	0.0775	0.0109	0.0010	1.1680	0.0256	0.0024
7	5,6	0.0813	0.0111	0.0009	1.1770	0.0263	0.0021
8	5,7	0.0813	0.0108	0.0009	1.1770	0.0256	0.0021
9	6,5	0.0849	0.0160	0.0011	1.1856	0.0382	0.0026
10	6,6	0.0791	0.0157	0.0010	1.1718	0.0370	0.0024
Average		0.0835	0.0132	0.0010	1.1822	0.0314	0.0023

Mean of average BAFo per particle:	1.1822
Standard deviation of average BAFo per particle:	0.0125

Comments

G. E. Jellison
Operator

9/19/08
Date