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FABRICATION AND CHARACTERIZATION OF SURROGATE TRISO PARTICLES USING 800µm ZrO_2 KERNELS

Fuel Cycle Research & Development Advanced Fuels Campaign

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FABRICATION AND CHARACTERIZATION OF SURROGATE TRISO PARTICLES USING 800µm ZrO_2 KERNELS

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INTRODUCTION

In support of fully ceramic microencapsulated (FCM) fuel development [1-2], coating development work is ongoing at the Oak Ridge National Laboratory (ORNL) to produce tri-structural isotropic (TRISO) coated fuel particles with both UN kernels [3] and surrogate (uranium-free) kernels. The nitride kernels are used to increase fissile density in these SiC-matrix fuel pellets with details described elsewhere [4]. The surrogate TRISO particles are necessary for separate effects testing and for utilization in the consolidation process development. This report focuses on the fabrication and characterization of surrogate TRISO particles which use 800µm in diameter ZrO_2 microspheres as the kernel.

The advanced gas reactor (AGR) program at ORNL used fluidized bed chemical vapor deposition (FBCVD) techniques for TRISO coating of UCO (two phase mixture of UO_2 and UC_x) kernels [5]. Similar techniques were employed for coating of the ZrO_2 kernels, however significant changes in processing conditions were required to maintain acceptable coating properties due to physical property and dimensional differences between the UCO and ZrO_2 kernels (Table 1).

Table 1: Comparison of UCO, UN, and ZrO_2 kernel properties

	AGR-1 (UCO)	UN kernels	ZrO_2 Surrogate
Nominal diameter (µm)	350	800	800
Density (g/cc)	10.92	12.53	6.0
Specific heat [J/g-K]	>0.3	0.25	0.45 - 0.65
Thermal conductivity (W/mK)	<5	15.5*	<4

*Literature value at 1000 °C. Actual values have not been measured.

The TRISO particles fabricated during the AGR-1 program have demonstrated excellent irradiation performance with no fission product release detected after 19.5% fissions per initial metal atom (FIMA). For this work, the target specifications for coating properties are similar to those of the AGR-1 program [6], with the only difference being a reduction of the buffer layer thickness from 100µm to 90µm. Twenty-nine process development experiments were performed to establish processing conditions enabling all four coating layers to meet the desired specifications. Details of the process development work have been previously reported [7]. The processing conditions established during the developmental work were used to produce a final surrogate TRISO batch named ZrX800-30T which met all required specifications that were measured. Details of the fabrication and characterization of ZrX800-30T are found in the following sections.

EXPERIMENTAL PROCEDURES

EQUIPMENT

The FBCVD coating furnace (Figure 1) used consists of a conical graphite coating chamber surrounded by a resistively heated graphite element which are both housed within a water cooled shell. Process gases are delivered through a water cooled injector which supports the coating chamber. Kernels are loaded through the top of the furnace at room temperature with argon gas flowing through the injector to maintain fluidization during heat up. The furnace is brought to coating temperature and the individual layers are deposited by switching between precursor gasses summarized in Table 2. Once the coating experiment is complete, the furnace is brought back to room temperature and gas flow is shut off allowing the kernels to drain through the injector and into the catch cup fixed to the bottom of the injector.

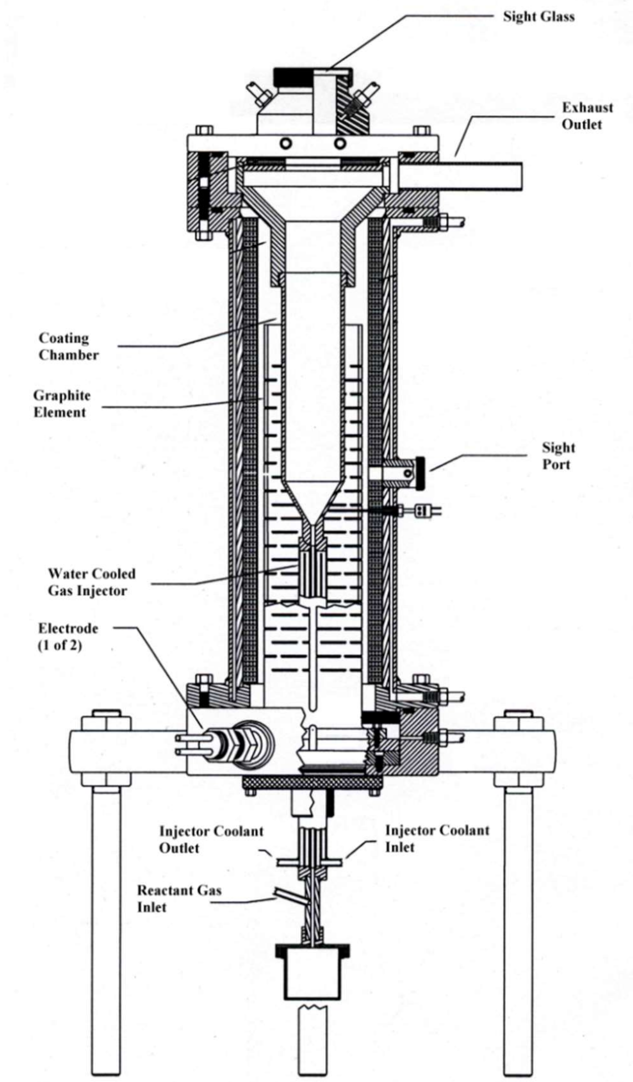

Figure 1: Schematic of FBCVD coating furnace used for TRISO coating of UN kernels.

Temperature of the fluidized bed is measured directly with a hand held optical pyrometer which looks through the top of the furnace via a sight glass and prism. Signal attenuation from the sight glass/prism set is measured and the appropriate correction factor applied. Gas delivery is controlled using electronic mass flow controllers which are periodically calibrated using a wet test meter. Calibration of critical process control equipment is performed using National Institute of Standards and Technology (NIST) traceable instruments and is typically performed annually.

RESULTS AND DISCUSSION

FABRICATION OF SURROGATE TRISO BATCH (ZRX800-30T)

After a series of 29 development runs to establish processing conditions which yielded the desired coating properties, a final batch of surrogate TRISO particles was produced and named ZrX800-30T. For this batch, all four coating layers were deposited onto nominally 800µm in diameter zirconia kernels according to AGR-SURROGATE-COAT-SOP-01.Rev0 which is the standard operating procedure for the surrogate FBCVD system. Key processing conditions for each coating layer are shown in Table 2. Coating summary sheets and gas certification sheets are shown in the appendix Figure 7 - Figure 17.

Table 2: Processing conditions for ZrX800-30T

	Coating Layer			
Parameter	**Buffer**	**IPyC**	**SiC**	**OPyC**
Time (min)	1.15	8.6	123.87	7.6
Temp (°C)	1425	1265	1425	1290
Ar flow (sccm)	4,620	10,625	7,500	14,025
H_2 flow (sccm)	N/A	N/A	7,500	N/A
C_2H_2 flow (sccm)	6,380	1,014	N/A	1,338
C_3H_6 flow (sccm)	N/A	861	N/A	1,137
MTS flow (sccm)	N/A	N/A	71	N/A
Total flow (sccm)	11,000	12,500	15,071	16,500
CGF	.58	.15	.0047	.15
CGR	N/A	.85	N/A	.85

Note:
CGF is coating gas fraction and is defined as precursor gas flow divided by total flow
CGR is coating gas ratio and is defined as the ratio of propylene to acetylene

CHARACTERIZATION OF SURROGATE KERNELS (ZRX800-K)

The kernels used for the entire surrogate coating development work are nominally 800μm in diameter ZrO_2 microspheres supplied by the advanced ceramics division of TOSOH Corporation. The trade name for the ZrO_2 microspheres is YTZ Grinding Media and they are yttria stabilized with a composition of 95% ZrO_2, 5% Y_2O_3 [8]. A polished cross sectional image showing several of the typical 800μm ZrO_2 microspheres can be seen in Figure 2.

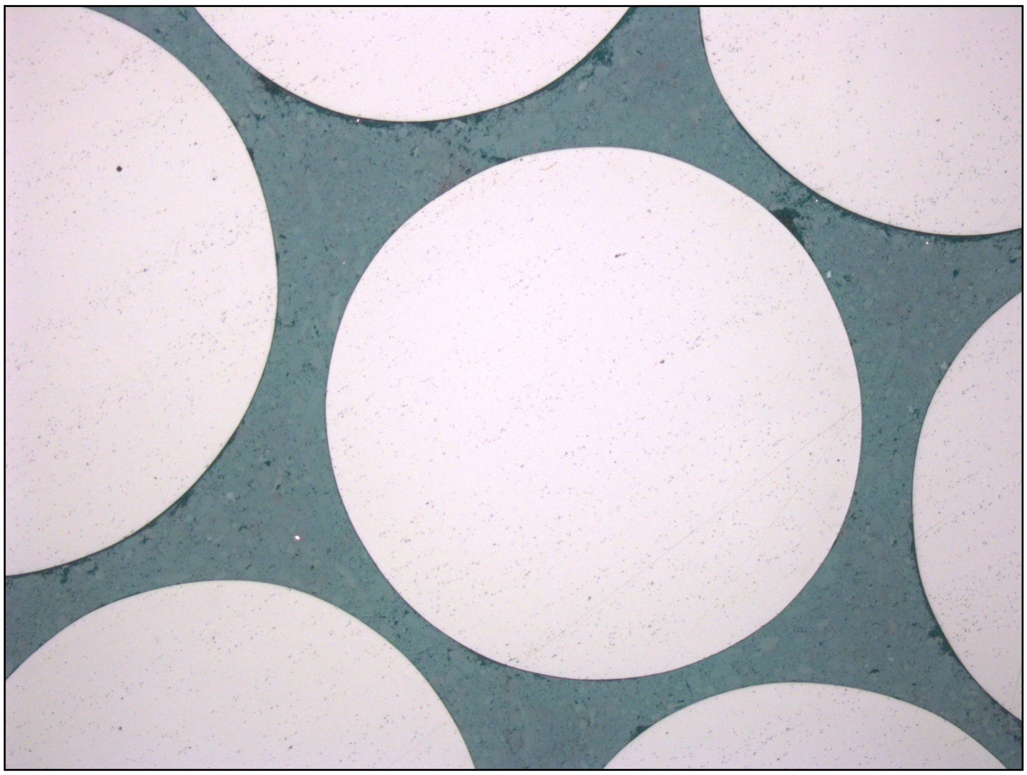

Figure 2: Optical cross sectional image of typical 800μm ZrO_2 microspheres used for the surrogate TRISO coating work.

The ZrO_2 microspheres are supplied in 1kg packages. For this work, 2kg of material was combined and named ZrX800-K. A sample of ZrX800-K was separated and characterized according to AGR-CHAR-PIP-25 Rev.0 which is the product inspection plan for surrogate kernels. The kernel characterization data generated is required for characterization of the coated particles, and is used in determining batch size for each coating experiment. A summary of the measurements made for ZrX800-K is shown in Table 3. Detailed information regarding each measurement is shown in the data report forms in the appendix Figure 18-Figure 21.

Table 3: Summary of measured kernel properties for ZrX800-K

Property	Mean	Std. Dev.
Diameter (μm)	813.7	25.0
Ellipticity (Dmax/Dmin)	1.015	
Kernel weight (g)	1.592E-03	2.95E-06
Density (g/cm^3)	6.032	0.030
Volume (cm^3)	2.639E-04	7.6E-07

CHARACTERIZATION OF SURROGATE TRISO BATCH (ZRX800-30T)

A standard product inspection plan, similar to AGR-1, was used to characterize all four coating layers of ZrX800-30T. A summary of the characterization data is found in Table 4, and detailed measurement information is found in the data report forms shown in the appendix Figure 22-Figure 25. Note that buffer layer density cannot be measured when subsequent layers are deposited on top of it. Consequently, data shown for buffer density is from previous buffer only coating experiments, which used the same buffer layer processing conditions, and not from ZrX800-30T. A cross sectional micrograph of a typical coated particle from ZrX800-30T is shown in Figure 3.

After characterization, all measured coating layer properties from ZrX800-30T were found to conform to the specifications listed in the AGR-1 Fuel Product Specification and Characterization Guidance document [6]. However, the density of the IPyC layer was unable to be measured due to difficulty in separating the IPyC layer from the buffer layer. The IPyC density is likely within specification based on previous work during the AGR-1 program [5] which found that the OPyC and IPyC densities were very similar (within .0052g/cm^3 of each other) when OPyC deposition temperature was 25°C higher than the IPyC deposition temperature. Also, density of both PyC layers appear similar from the cross sectional optical images, but this only provides a qualitative analysis. Modified buffer coatings were produced which had a thin lower density outer layer in order to promote a weak interface between the buffer and IPyC layers (details of this modified buffer coating are described in the following section). The engineered weak interface was designed to aid in buffer/IPyC separation, but only marginal improvements were noted. Difficulty in separating the SiC/OPyC layers was also encountered and a modified technique had to be employed where the bottom portion of the TRISO particle was glued in place so that the OPyC could be removed from the top portion with tweezers. It is hypothesized that the difficulty in separating the pyrocarbon layers from their preceding layers is related to the geometrical differences in the currently used larger 800μm kernels compared to previous 350μm kernels used during AGR-1. With the larger kernel, the coating layer interface approaches a flat on flat orientation rather than the more pronounced curvature present when using the previous smaller kernels.

Table 4: Summary of key characterization data for ZrX800-30T

Layer Property	Specification (AGR-1)	Average for ZrX800-30T	Pass/Fail
Buffer thickness (μm)	90 ± 15*	98.7	PASS
IPyC thickness (μm)	40 ± 4	41.3	PASS
SiC thickness (μm)	35 ± 3	32.9	PASS
OPyC thickness (μm)	40 ± 4	43.1	PASS
Buffer density (g/cm^3)	1.03 ± 0.15	(1.01)**	PASS
IPyC density (g/cm^3)	1.90 ± 0.05	N/M	N/M
SiC density (g/cm^3)	≥ 3.19	3.1983	PASS
SiC microstructure (grain size)	Visual standard	Smaller than max allowable	PASS
OPyC density (g/cm^3)	1.90 ± 0.05	1.8937	PASS
IPyC anisotropy (BAF_o)	≤ 1.0350	1.0149	PASS
OPyC anisotropy (BAF_o)	≤ 1.035	1.0153	PASS

N/M indicates property not measured
*Note that buffer thickness target reduced from 100μm to 90μm for this work
**Buffer density shown is from previous buffer only coating run ZrX800-11B, ZrX800-30T used the same processing conditions for the buffer layer as ZrX800-11B

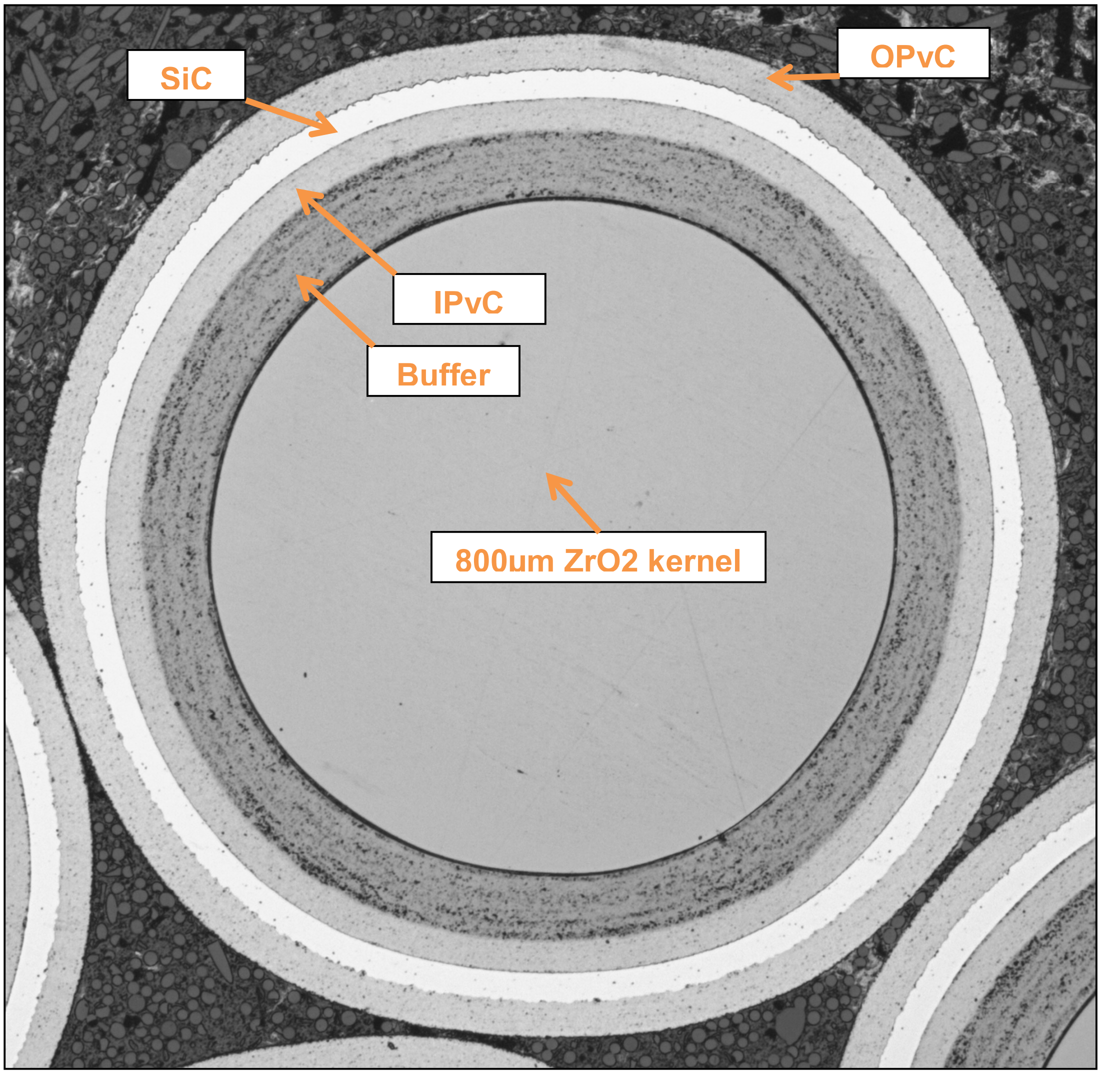

Figure 3: Optical cross sectional image of a typical TRISO particle from ZrX800-30T.

Back scatter SEM imaging of the SiC layer from ZrX800-30T was performed to observe the SiC microstructure (Figure 4). When compared to the AGR-1 visual standard for maximum allowable grain size shown in Figure 5, the grain size of the SiC from ZrX800-30T was found to be smaller and therefore meets the specification. It is interesting to note that the outer surface of the SiC layer from ZrX800-30T is significantly rougher than what is typically observed in previous AGR-1 TRISO particles. This increased SiC surface roughness isn't something that is currently captured in the specifications, and its impact on fuel performance is unknown. The increased roughness may have contributed to the difficulty in separating the SiC/OPyC layers which was previously discussed.

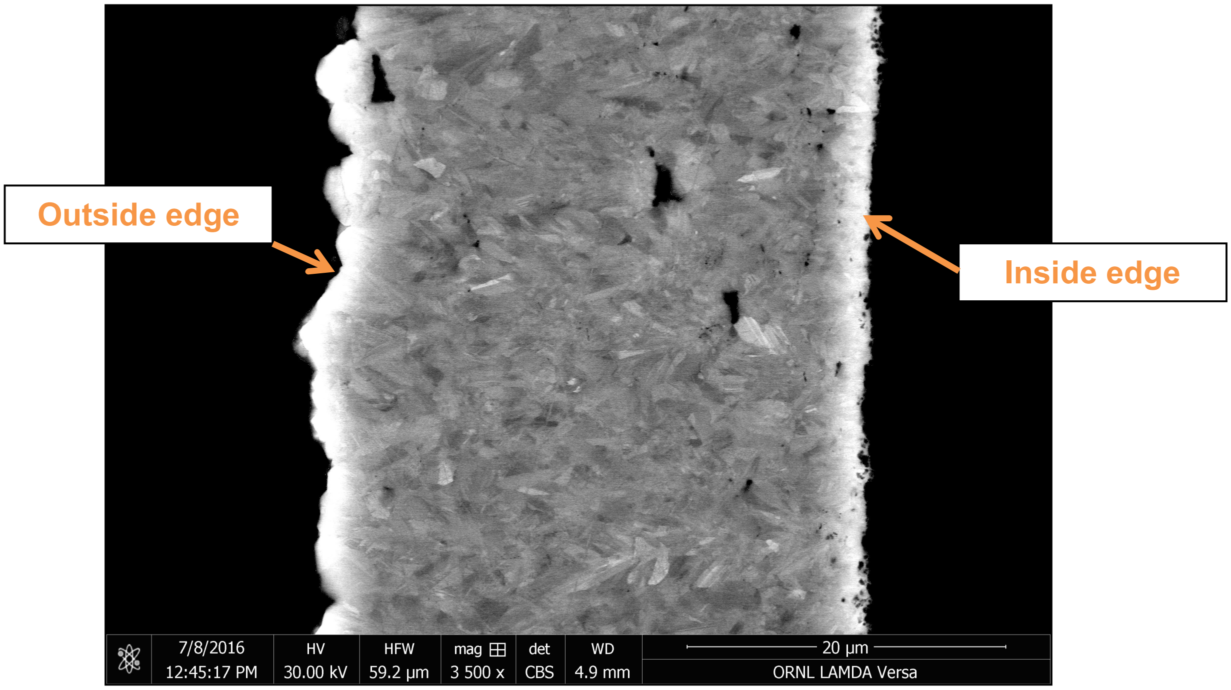

Figure 4: Back scatter SEM image of SiC layer from ZrX800-30T showing SiC grain size to be within AGR-1 specification.

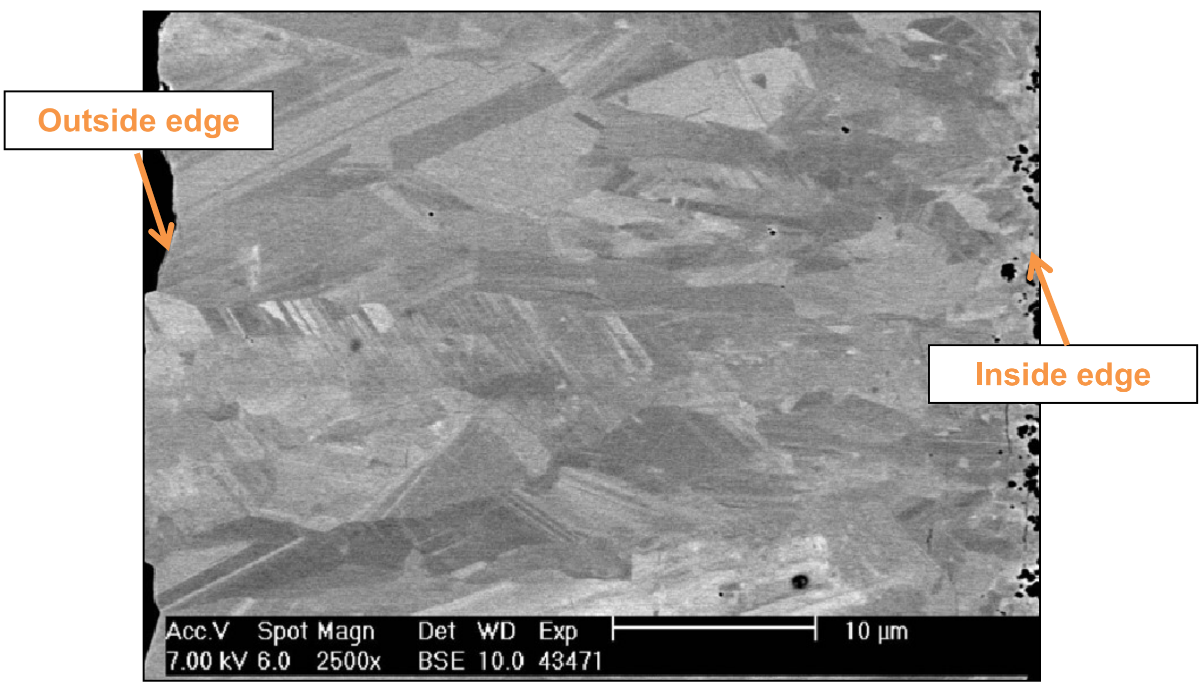

Figure 5: Visual standard for maximum allowable SiC grain size.

MODIFIED BUFFER LAYER (LOWER DENSITY OUTER REGION)

In order to measure the IPyC density using a gradient density column, the IPyC layer must be removed from the buffer layer. During the surrogate TRISO coating development work, which used 800µm kernels, separating the buffer/IPyC layer was extremely difficult. To aid in the buffer/IPyC separation, a modified buffer layer was developed which incorporated a thin lower density outer region to create a weak interface (Figure 6). The lower density outer region was created by first depositing the standard buffer layer, per the processing conditions in Table 2, then increasing the temperature by 100°C and making an additional deposition for 5 seconds.

While a thin lower density interface between the buffer and IPyC layers was successfully produced, little improvement was seen when attempting to separate the layers, and this modified buffer layer configuration was not used in the subsequent TRISO particle batches. However, this type of modified buffer/IPyC interface may be of interest to future coated particle work since a weak buffer/IPyC interface is desirable so that the buffer will de-bond without tearing the IPyC layer during irradiation.

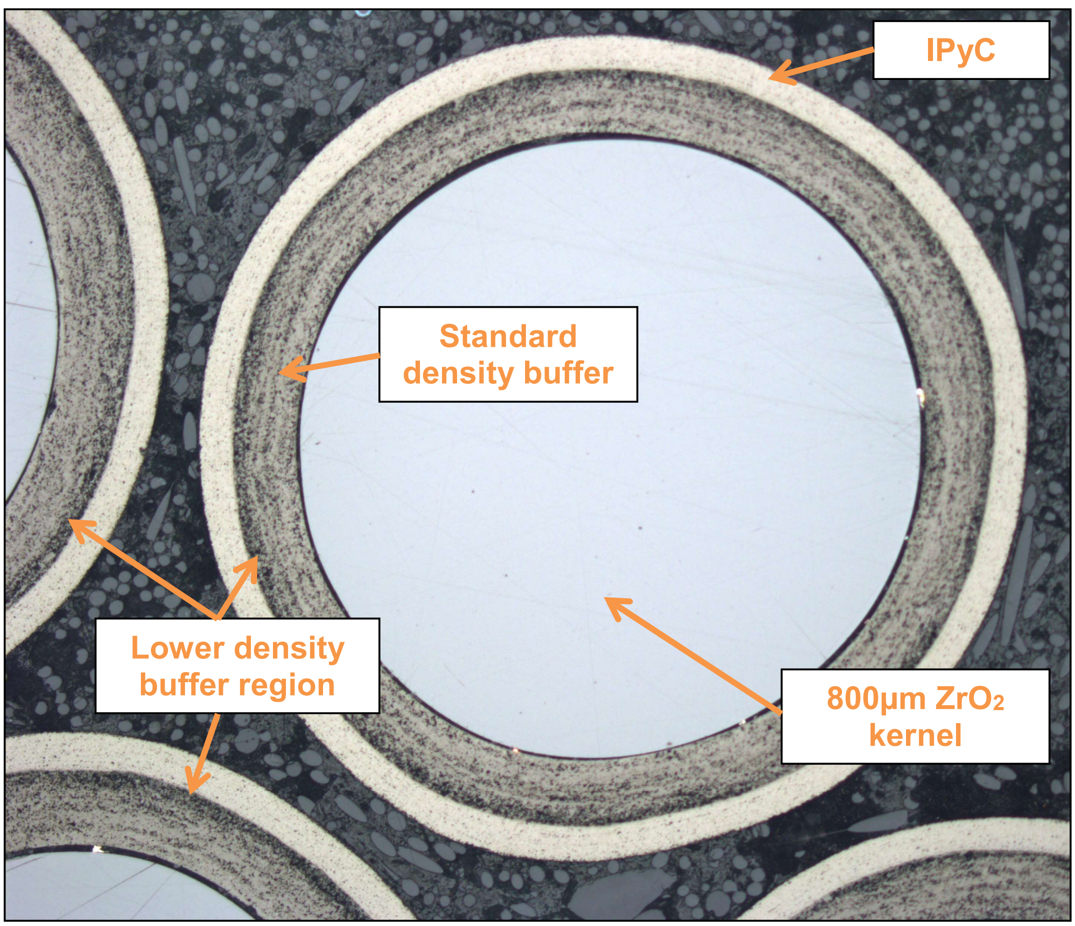

Figure 6: Optical cross sectional image of ZrX800-19I showing thin lower density outer region in the buffer layer.

SUMMARY

After significant process development work, a batch of TRISO particles (ZrX800-30T) was produced using 800μm in diameter ZrO_2 microspheres as the kernel. The ZrX800-30T particle batch was characterized and found to have met all the AGR-1 coating property specifications measured. These surrogate (uranium-free) particles will be used for separate effects testing and for consolidation process development in support of the FCM fuel development program.

Now that processing conditions have been identified which yield surrogate TRISO particles with acceptable coating properties, additional surrogate particles can be fabricated for future work much more readily.

ACKNOWLEDGMENTS

The work presented in this transaction was supported by the Advanced Fuels Campaign of the Fuel Cycle R&D program in the Office of Nuclear Energy, US Department of Energy.

REFERENCES

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[2] K. A. Terrani; L. L. Snead; J. C. Gehin, Journal of Nuclear Materials 2012, 427 (1-3), 209-224.

[3] T. B. Lindemer, C. M. Silva,J. J. Henry, J. W. McMurray, S. L. Voit, R. D. Hunt, “Carbothermic Synthesis of ~820-μm UN Kernels: Investigation of Process Variables” ORNL/TM-2015/301 (2015).

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[5] J. D. Hunn; R. A. Lowden, Data compilation for AGR-1 baseline coated particle composite LEU01-46T, ORNL/TM-2006/019 Oak Ridge National Laboratory: 2006.

[6] C. M. Barnes, AGR-1 Fuel Product Specification and Characterization Guidance, EDF-4380, Rev. 8: 4/21/2006

[7] B. C. Jolly, T. B. Lindemer, K. A. Terrani, “TRISO Coating Development Progress for Uranium Nitride Kernels”, ORNL/LTR-2015/432

[8] TOSOH Corporation, “Typical Properties of Zirconia Grinding and Dispersion Media”, http://www.tosoh.com/our-products/advanced-materials/zirconia-grinding--dispersion-media

Appendix:

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Surface processing and Mechanics Group
Materials Science & Technology Division
Oak Ridge National Laboratory

AGR-Surrogate-Coat-SOP-01 Rev. 0
Issue Date: July-2009
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Standard Operating Procedure
Surrogate Fluidized Bed Chemical Vapor Deposition System

Appendix G: Run Summary

Run Number	**ZRX800-30T**	
Description	Surrogate TRISO run using ZrO_2 kernels	
Particle batch description	~ 800 μm ZrO_2	
Particle initial batch wt. (g)	40.0019	
Coated particle batch wt. (g)	72.423	
	Target Parameters	As-Processes
Buffer		
Coating gases	C2H2	C_2H_2
TGF	11,000sccm	11,000
CGF	.58	.58
Temperature (°C)	1425	1425
Time (min)	1.15	1.15
IPyC		
Coating gases	C2H2 + C3H6	$C_2H_2 + C_3H_6$
TGF	12,500sccm	12,501
CGF	.15	.15
CGR	.85	.85
Temperature (°C)	1265	1265
Time (min)	8.6	8.6
SiC		
Coating gases	MTS	MTS
TGF	15,114sccm	15,071
CGF	.0075	.0047
Temperature (°C)	1425	1425
Time (min)	~116*	123.87
OPyC		
Coating gases	C2H2 + C3H6	$C_2H_2 + C_3H_6$
TGF	16,500sccm	16,507
CGF	.15	.15
CGR	.85	.85
Temperature (°C)	1290	1290
Time (min)	7.6	7.6
Comments		
Operator/Date	[signature] 6-30-16	
Verify/Date		

* Actual SiC run time determined by bubbler weight change which is measured in situ
– target weight change is 59g

Figure 7: Coating conditions summary sheet for ZrX800-30T

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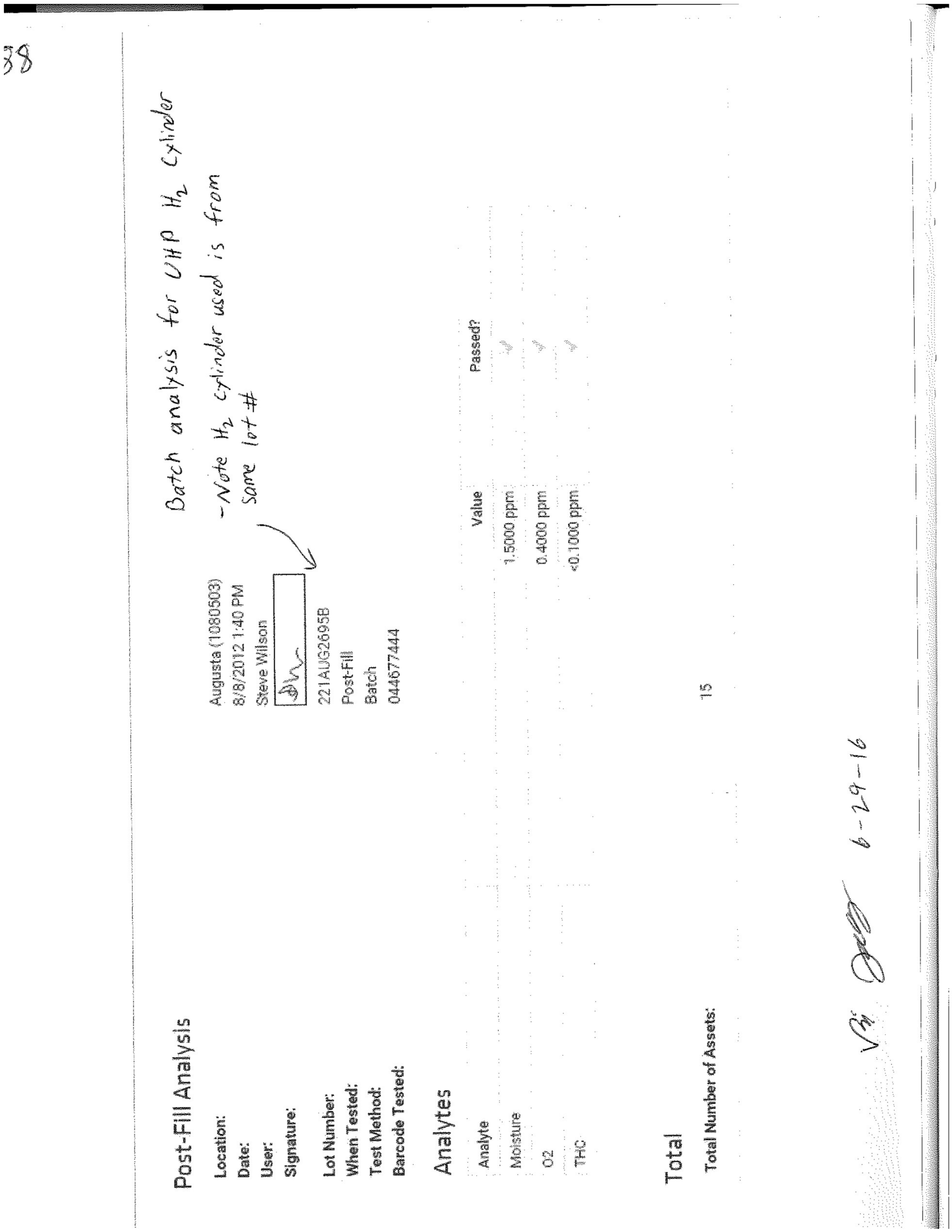
Appendix D: Precursor Summary

Run Number	ZRX800-30T		
Gas	**Cylinder number (stamped on cylinder)**	**Tracking number (bar code number)**	**Batch analysis or material cert. (logbook and page number)**
Hydrogen	W435389	052909180	pg 88 this logbook
Argon	H7030	011655116	pg 89 this logbook
Argon	H1269589	* Gov. owned cylinder # X6001677	pg 90 this logbook
Acetylene	Y11507	034847512	pgs 91+92 this logbook
Propylene	C01876HV	No tracking # on cylinder	pgs 93+94 this logbook
MTS	Product code M85301-500G	Batch # 32296CPV	pg 95 this logbook
MTS	Product code M85301-500G	Batch # 00128HH	pg 96 this logbook
Comments	* no tracking number present on cylinder		
Operator/Date	[signature] 6-30-16		
Verify/date			

Note that these are the last two batches of MTS precursor added to the bubbler prior to run ZrX800-30T. However, there was residual MTS in the bubbler from previous refills. The batch # for these previous refills was not documented, but the same 99% grade of MTS from Sigma-Aldrich was used.

Figure 8: Precursor summary sheet for ZrX800-30T



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Post-Fill Analysis

Location: Augusta (1080503)
Date: 8/8/2012 1:40 PM
User: Steve Wilson
Signature:

Lot Number: 221AUG2695B
When Tested: Post-Fill
Test Method: Batch
Barcode Tested: 044677444

Analytes

Analyte	Value	Passed?
Moisture	1.5000 ppm	✓
O2	0.4000 ppm	✓
THC	<0.1000 ppm	✓

Total

Total Number of Assets: 15

Batch analysis for UHP H_2 Cylinder
- Note H_2 cylinder used is from same lot #

6-29-16

Figure 9: Batch analysis for H_2 cylinder used for ZrX800-30T

AIR LIQUIDE

CERTIFICATES FOR PURE SPECIALTY GASES

2B-ALPCYL-OPS-0028-F
Rev. 0
Effective Date:06/15/2008

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CERTIFICATE OF ANALYSIS

Ar used for ZrX 800-30T

Certification Of Cylinder #: H7030

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

Customer:	X10	**Batch/Lot #:**	070OAK6395A
Date of Certification:	3/10/16	**Item Number:**	0013-1300
P.O. Number:	4800947221	**Valve:**	580
Document Number:	2362513	**Cylinder Size:**	44

ANALYSIS REPORT

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

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.2 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

Notes:

Certified By: *Jamie Gilmore*
Name Jamie Gilmore

Air Liquide America Specialty Gases LLC
1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

MANDATORY DOCUMENT

6-29-16

Figure 10: Certificate of analysis for Ar cylinder used for ZrX800-30T

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AIR LIQUIDE

CERTIFICATES FOR PURE SPECIALTY GASES

2B-ALPCYL-OPS-0028-F
Rev. 0
Effective Date:06/15/2008

CERTIFICATE OF ANALYSIS

Ar used for ZrX800-30T

Certification Of Cylinder #: H1269589

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

Customer:	**X10**	**Batch/Lot #:**	**070OAK6395A**
Date of Certification:	**3/10/16**	**Item Number:**	**0013-1300**
P.O. Number:	**4800947221**	**Valve:**	**580**
Document Number:	**2362513**	**Cylinder Size:**	**44**

ANALYSIS REPORT

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

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

Notes:

Certified By: *Jamie Gilmore*
Name Jamie Gilmore

Air Liquide America Specialty Gases LLC
1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

MANDATORY DOCUMENT

6-29-16

Figure 11: Certificate of analysis for Ar cylinder used for ZrX800-30T

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Certificate of conformance for C_2H_2 cylinder on following pg.

Asset Details	
Barcode:	034847512
Serial Number:	Not Set
Category:	Spec Gas
Group:	Acetylene
Type:	LG
Product Code:	A0211675
Description:	ACETYLENE, C2H2, ALPHAGAZ 1, LG, CGA-510
Ownership:	Air Liquide
Location:	**Customer:** 56905 View **Department:** UT BATTELLE LLC-1 (272184) View
Volume:	320.00 Liters
Capacity:	LG
DOT Pressure Rating:	240
Last Test Date:	11/06/2014 18:00:00
Manufacture Date:	03-2015
Manufacturer:	Dummy
Thread:	Tapered
Valve Type:	Standard
Working Pressure:	LG
OwnerType:	AL
Status:	Delivered
Unset Asset Details:	Show Details

6-29-16

Figure 12: Info sheet for C_2H_2 cylinder used for ZrX800-30T

2

AIR LIQUIDE

CERTIFICATE OF CONFORMANCE

Product: Acetylene **Grade: Alphagaz 1**

Major Component	Specification
Acetylene Alphagaz 1	99.6000%

Impurities	Specification
NItrogen	0.40%
Phosphine	<50 ppm

Air Liquide America, L.P. guarantees this product meets the above listed specifications.

Air Liquide America, L.P.

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

ICSC-CYL-0307-W [signature] 6-29-16 Revision: 2 Effective Date:

Figure 13: Certificate of conformance for C_2H_2 cylinder used for ZrX800-30T

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Certificate of conformance for C_3H_6 cylinder on following page.

GAS INNOVATIONS

PROPYLENE, REASEARCH

Cylinder Number:	001876HV
Gross Weight:	53 LBS
Scale Weight:	26 LBS
Net Weight:	27 LBS
Fill Date / Filler:	05/27/16 CR
Lot Number:	PRY-031416-R
SO Number:	00074982

Info Tag included with C_3H_6 cylinder

[illegible] 6-29-16

Figure 14: Info tag for C_3H_6 cylinder used for ZrX800-30T

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GAS INNOVATIONS

CERTIFICATE OF CONFORMANCE

PROPYLENE
RESEARCH PURITY

Guaranteed Specifications		Lot Analysis
99.99 % +	Propylene	99.995 % +
Sum of all impurities to	Ethane (None Detected)	< 0.50 PPM
be < 100 PPM	Total saturated C4's (None Detected)	< 5 PPM
Sulfur < 1PPM	Propane	48 PPM
Water < 2 PPM	Carbon Dioxide	< 1 PPM
Liquid Phase Analysis	Water	< 2 PPM
	Oxygen	< 1 PPM
	Nitrogen	< 5 PPM
	Sum of Other Hydrocarbons (None Detected)	< 5 PPM
	Sulfur	< 0.50 PPM

Certified by: Stanely Chatham Date: 6-1-16

SO# 00074982

Cylinder Serial Number(s)
01271HV, 001805HV, 001876HV

Batch Number
PRY-052716-R

Customer PO# **82003708-0**
Lot Number: PRY-031416-R
18005 E. Highway 225, La Porte, TX 77571 Tel- 281-471-2200 Fax-281-471-2201 www.gasinnovations.com

6-29-16

Figure 15: Certificate of conformance for C_3H_6 cylinder used for ZrX800-30T

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SIGMA-ALDRICH®

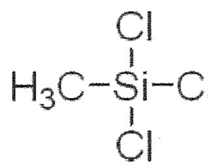
sigma-aldrich.com

3050 Spruce Street,Saint Louis,MO 63103,USA
Website: www.sigmaaldrich.com
Email USA: techserv@sial.com
Outside USA: eurtechserv@sial.com

Certificate of Analysis

Product Name:
Methyltrichlorosilane 99%

Product Number:	**M85301**
Lot Number:	**32296CPV**
Brand:	ALDRICH
CAS Number:	75-79-6
MDL Number:	MFCD00000481
Formula:	CH3Cl3Si
Formula Weight:	149.48 g/mol
Quality Release Date:	18 MAR 2011



Test	Specification	Result
Appearance (Color)	Colorless	Colorless
Appearance (Form)	Liquid	Liquid
Infrared spectrum	Conforms to Structure	Conforms
Purity (GC)	$\geq$ 98.5%	98.9%

Jennifer Baughman, Manager
Quality Control
Sheboygan Falls, WI US

Version Number: 1 Page 1 of 1

6-29-16

Figure 16: Batch analysis for MTS used for ZrX800-30T

Certificate of Analysis

SIGMA-ALDRICH

Product Name	Methyltrichlorosilane, 99%
Product Number	M85301
Product Brand	ALDRICH
CAS Number	75-79-6
Molecular Formula	CH_3SiCl_3
Molecular Weight	149.48

TEST	SPECIFICATION	LOT 00128HH RESULTS
APPEARANCE	COLORLESS LIQUID	COLORLESS LIQUID
INFRARED SPECTRUM	CONFORMS TO STRUCTURE.	CONFORMS TO STRUCTURE.
GAS LIQUID	98.5% (MINIMUM)	98.6% *
	REVISED SEPTEMBER 30, 2005 JAD	* SUPPLIER DATA
QUALITY CONTROL		JULY 2007

Barbara Rajzer, Supervisor
Quality Control
Milwaukee, Wisconsin USA

6-29-16

Figure 17: Batch analysis for MTS used for ZrX800-30T

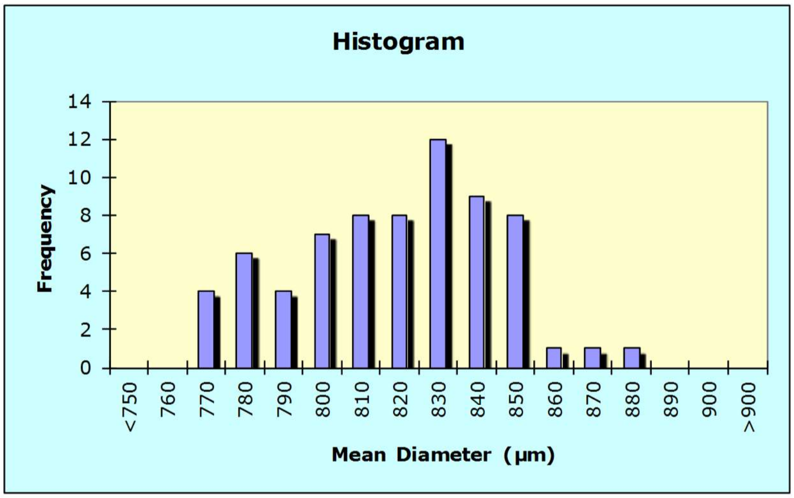
Data Report Form DRF-10A: Measurement of Particle Diameter

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Chinthaka M. Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P13010202
Sample ID:	ZrX800-K
Sample Description:	nominal 800 micron ZrO2 microspheres
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P13010202_output

Number of particles analyzed:	69
Mean of the average diameter of each particle (µm):	813.7
Standard deviation in the average diameter of each particle (µm):	25

Distribution of the average particle diameter (top binned)

Mean Diameter (µm)	*Frequency*
<750	0
760	0
770	4
780	6
790	4
800	7
810	8
820	8
830	12
840	9
850	8
860	1
870	1
880	1
890	0
900	0
>900	0



Operator	Date

Figure 18: Data report for particle diameter measurement of ZrX800-K.

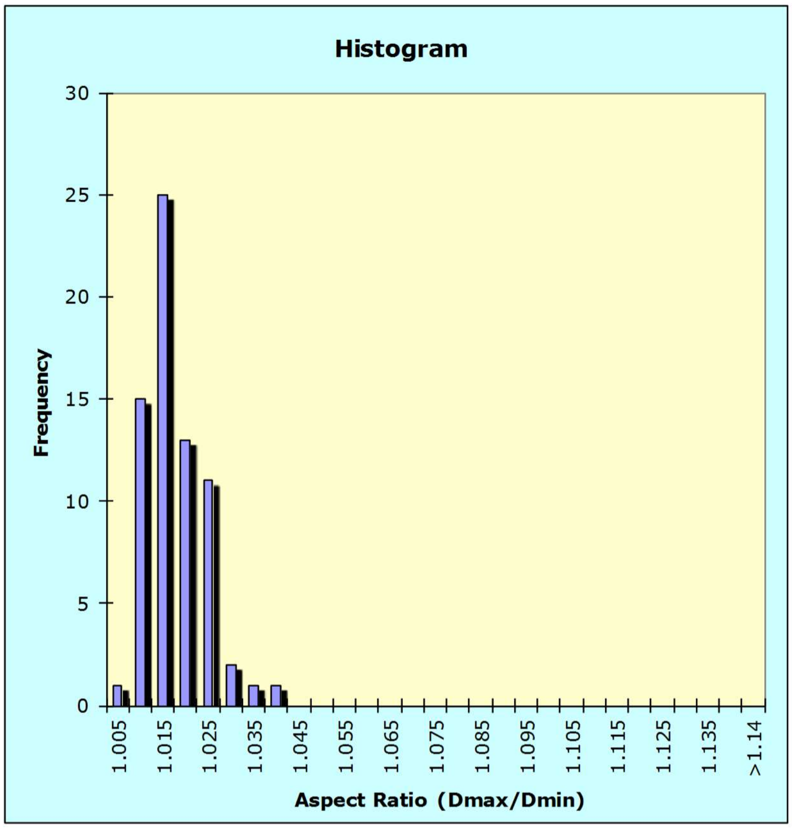
Data Report Form DRF-10B: Measurement of Particle Aspect Ratio (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-10 Rev. 2
Operator:	Grant Helmreich
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P0
Sample ID:	ZrX800-K
Sample Description:	nominal 800 micron ZrO2 microspheres
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P

Number of particles analyzed:	69
Number of particles with aspect ratio ≥ 1.14	0
Average particle aspect ratio:	1.015

Distribution of the aspect ratio (top binned)

Aspect Ratio (D)	*Frequency*
1.005	1
1.010	15
1.015	25
1.020	13
1.025	11
1.030	2
1.035	1
1.040	1
1.045	0
1.050	0
1.055	0
1.060	0
1.065	0
1.070	0
1.075	0
1.080	0
1.085	0
1.090	0
1.095	0
1.100	0
1.105	0
1.110	0
1.115	0
1.120	0
1.125	0
1.130	0
1.135	0
1.140	0
>1.14	0



Operator	Date

Figure 19: Data report for particle aspect ratio measurement of ZrX800-K.

Data Report Form DRF-22: Estimation of Average Particle Weight					
Procedure:	AGR-CHAR-DAM-22 Rev. 1				
Operator:	Grant Helmreich				
Particle Lot ID:	ZrX800-K				
Particle Lot Description:	Zirconia kernels				
Filename:	\\mc-agr\AGR\ParticleWeight\Wyymmddnn_DRF22R1.xls				

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.3910	0.4269	0.4064	0.4222	0.4005
Number of particles:	247	269	255	264	251
Average weight/particle (g):	1.583E-03	1.587E-03	1.594E-03	1.599E-03	1.596E-03

Mean average weight/particle (g):	1.592E-03
Standard error in mean average weight/particle (g):	2.95E-06

Operator	Date

Figure 20: Data report for average particle weight of ZrX800-K.

Data Report Form DRF-15: Measurement of Average Kernel Envelope Density using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-15 Rev. 3
Operator:	Grant Helmreich
Kernel Lot ID:	ZrX800-K
Kernel Lot Description:	ZrO_2 kernels of ~800 micron diameter
Thermocouple Expiration Date:	3/9/2016
Penetrometer Expiration Date:	5/7/2016
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\Syymmddnn\Syymmddnn_DRF15R3.xls

Mean average weight/kernel (g):	1.59E-03
Standard error in mean average weight/kernel (g):	2.95E-06

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Porosimeter data file number:	S15072101L	S15072102L	S15072103L		
Weight of kernels (g):	5.0890	5.5025	5.5332		
Approximate number of kernels:	3197	3457	3476		
Uncertainty in number of kernels:	6	6	6		
Envelope volume of sample (cc):	0.840	0.911	0.923		
Average envelope volume/kernel (cc):	2.63E-04	2.63E-04	2.65E-04		
Sample envelope density (g/cc):	6.055	6.043	5.998		

Mean average envelope volume/kernel (cc):	2.639E-04
Standard error in mean envelope volume/kernel (cc):	7.6E-07
Mean sample envelope density (g/cc):	6.032
Standard deviation in sample envelope density (g/cc):	0.030

Comments

Operator	Date

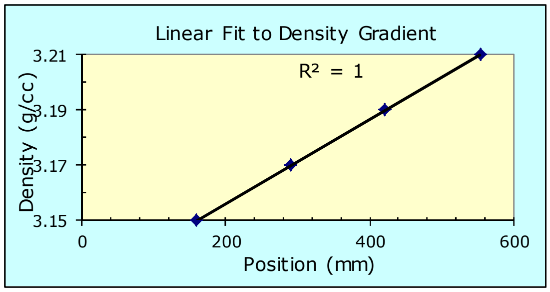
Figure 21: Data report for average kernel density and volume of ZrX800-K.

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

Procedure:	AGR-CHAR-DAM-02 Rev. 5
Operator:	Grant Helmreich/John Dyer
Filename:	\\mc-agr\AGR\DensityColumn\D16070701_DRF02R5.xls
Sample ID:	ZrX800-30T
Sample description:	SiC fragments
Float expiration date:	12/2023
Gauge expiration date:	06/2017
Bath temperature:	23.1 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Volume
3.150	147.92	172.39	160.16
3.170	277.04	301.75	289.40
3.190	408.67	433.31	420.99
3.210	540.91	564.91	552.91

Linear Fit			
slope	StDev	intercept	StDev
1.53E-04	2.70E-06	3.13E+00	9.63E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	417.48	3.1894	26	480.16	3.1990	51		
2	432.04	3.1916	27	480.62	3.1990	52		
3	444.07	3.1935	28	480.79	3.1991	53		
4	467.50	3.1970	29	481.21	3.1991	54		
5	468.63	3.1972	30	481.59	3.1992	55		
6	469.26	3.1973	31	482.11	3.1993	56		
7	469.41	3.1973	32	482.76	3.1994	57		
8	471.06	3.1976	33	483.23	3.1994	58		
9	471.89	3.1977	34	483.39	3.1995	59		
10	474.12	3.1981	35	484.24	3.1996	60		
11	474.32	3.1981	36	485.05	3.1997	61		
12	474.78	3.1982	37	485.05	3.1997	62		
13	475.29	3.1982	38	485.87	3.1999	63		
14	475.97	3.1983	39	488.13	3.2002	64		
15	477.11	3.1985	40	488.66	3.2003	65		
16	477.84	3.1986	41	488.93	3.2003	66		
17	477.84	3.1986	42	489.31	3.2004	67		
18	478.40	3.1987	43			68		
19	478.40	3.1987	44			69		
20	478.40	3.1987	45			70		
21	478.83	3.1988	46			71		
22	478.83	3.1988	47			72		
23	478.83	3.1988	48			73		
24	479.48	3.1989	49			74		
25	479.77	3.1989	50			75		

Average density of SiC fragments:	3.1983
Standard deviation in density of SiC fragments:	0.0021
ncertainty in calculated density of SiC fragments:	0.0016

Operator	Date

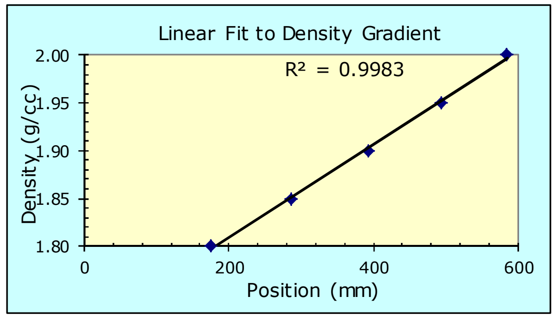
Figure 22: Data report for SiC density of ZrX800-30T.

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

Procedure:	AGR-CHAR-DAM-03 Rev. 4
Operator:	John Dyer
Filename:	\\mc-agr\AGR\DensityColumn\D16071301_DRF03R4.xls
Sample ID:	ZrX800-30T
Sample description:	OPyC fragments from ZrX800-30T
Float expiration date:	07/2017
Gauge expiration date:	06/2017
Bath temperature:	23.1 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Volume
1.800	169.84	179.17	174.51
1.850	282.22	290.47	286.35
1.900	390.81	396.74	393.78
1.950	490.13	497.06	493.60
2.000	581.24	587.94	584.59

Linear Fit			
slope	StDev	intercept	StDev
4.86E-04	3.02E-06	1.71E+00	1.25E-03



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	230.54	1.8242	26	430.17	1.9212	51		
2	231.47	1.8246	27	431.63	1.9219	52		
3	233.79	1.8258	28	437.17	1.9246	53		
4	237.88	1.8278	29	439.30	1.9256	54		
5	240.52	1.8290	30			55		
6	243.03	1.8303	31			56		
7	386.33	1.8999	32			57		
8	390.72	1.9020	33			58		
9	393.27	1.9033	34			59		
10	395.16	1.9042	35			60		
11	395.86	1.9045	36			61		
12	397.29	1.9052	37			62		
13	397.29	1.9052	38			63		
14	399.86	1.9065	39			64		
15	400.84	1.9069	40			65		
16	400.84	1.9069	41			66		
17	401.91	1.9075	42			67		
18	406.15	1.9095	43			68		
19	406.37	1.9096	44			69		
20	409.30	1.9110	45			70		
21	409.30	1.9110	46			71		
22	420.50	1.9165	47			72		
23	421.41	1.9169	48			73		
24	422.12	1.9173	49			74		
25	425.54	1.9189	50			75		

Average density of PyC fragments:	1.8937
Standard deviation in density of PyC fragments:	0.0354
certainty in calculated density of PyC fragments:	0.0018

Operator

Date

Figure 23: Data report for measurement of OPyC density of ZrX800-30T.

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

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	G.E. Jellison
Mount ID:	M16070601
Sample ID:	ZrX800-30T
Sample Description:	800 micron kernel Brian Jolley
Folder containing data:	\\mc-agr\AGR\2-MGEM\Ryymmddnn\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0074	0.0031	0.0020	1.0149	0.0063	0.0041
2	0.0068	0.0031	0.0019	1.0137	0.0063	0.0039
3	0.0073	0.0027	0.0019	1.0147	0.0055	0.0039
4	0.0071	0.0023	0.0019	1.0143	0.0047	0.0039
5	0.0067	0.0027	0.0020	1.0135	0.0055	0.0041
6	0.0088	0.0037	0.0021	1.0178	0.0075	0.0043
7	0.0075	0.0026	0.0020	1.0151	0.0053	0.0041
Average	0.0074	0.0029	0.0020	1.0149	0.0059	0.0040
St. Dev.	0.0007	0.0005	0.0001	0.0014	0.0009	0.0002

Comments

Operator	Date

Figure 24: Data report for measurement of IPyC anisotropy of ZrX800-30T.

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

Procedure:	AGR-CHAR-DAM-18 Rev. 3
Operator:	G.E. Jellison
Mount ID:	M16070601
Sample ID:	ZrX800-30T
Sample Description:	800 micron kernel Brian Jolley
Folder containing data:	\\mc-agr\AGR\2-MGEM\Ryymmddnn\

Particle #	Diattenuation			OPTAF = (1+N)/(1-N)		
	Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	0.0078	0.0031	0.0019	1.0157	0.0063	0.0039
2	0.0074	0.0031	0.0019	1.0149	0.0063	0.0039
3	0.0070	0.0029	0.0019	1.0141	0.0059	0.0039
4	0.0072	0.0028	0.0019	1.0145	0.0057	0.0039
5	0.0073	0.0030	0.0020	1.0147	0.0060	0.0041
6	0.0088	0.0035	0.0021	1.0178	0.0070	0.0043
7	0.0077	0.0030	0.0019	1.0155	0.0061	0.0039
Average	0.0076	0.0031	0.0019	1.0153	0.0062	0.0039
St. Dev.	0.0006	0.0002	0.0001	0.0012	0.0004	0.0002

Comments

Operator		Date

Figure 25: Data report for measurement of OPyC anisotropy of ZrX800-30T.