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ORNL-27 (4-00)

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Data Compilation for AGR-2 B&W UO₂ Coated Particle Batch G73H-10-93085B

John D. Hunn
Oak Ridge National Laboratory

Coated particle fuel batch G73H-10-93085B was produced by the Babcock and Wilcox Company (B&W) for use as UO₂ fuel for the Advanced Gas Reactor Fuel Development and Qualification Program's AGR-2 irradiation test. Batch G73H-10-93085B is a single batch of TRISO-coated 500 μm nominal diameter 10% low enrichment uranium oxide kernels (LEU). The TRISO-coatings consist of a ~50% dense carbon buffer layer (100 μm nominal thickness) followed by a dense inner pyrocarbon layer (40 μm nominal thickness) followed by a SiC layer (35 μm nominal thickness) followed by another dense outer pyrocarbon layer (40 μm nominal thickness). Argon was added to the hydrogen fluidization gas during SiC deposition.

The AGR-2 Fuel Specification (INL SPC-923) provides the requirements necessary for acceptance of the fuel manufactured for the AGR-2 irradiation test. The bulk of the kernels and coated particle acceptance testing was performed at B&W and is not contained in this report. B&W samples NP-B8488, NP-B8489, and NP-B8490 (all from batch G73H-10-93085B) were sent to ORNL for characterization and compaction. These samples were composited and renamed LEU10. Samples were then riffled for supplemental characterization. The procedures for the limited characterization and qualification of the particles performed at ORNL are outlined in ORNL product inspection plan AGR-CHAR-PIP-09. The BA_{Fo} equivalent optical anisotropies of the inner and outer pyrocarbon layers are reported on Inspection Report Form IRF-09, with a determination as to whether the particle batch satisfied the specified parameters for this property. The batch was found to satisfy the AGR-2 Fuel Specification SPC-923, Rev. 1 for IPyC and OPyC anisotropy.

Also provided in this data package are data on the true BA_{Fo}, average particle weight, and SiC soot inclusion defect fraction. True BA_{Fo} is calculated as $(1+N)/(1-N)$, where N is the diattenuation. This differs from equivalent BA_{Fo} = $1+3N$, which is the calculation used by the fuel specification to allow comparison to historical measurements. Three SiC inclusions were found in a sample of 3510 particles. This corresponds to a defect fraction of $<2.3\text{E-}3$ at 95% confidence. One of these inclusions was of unusual appearance and was determined to contain molybdenum using energy dispersive x-ray analysis.

Inspection Report Form IRF-09: AGR-2 Coated Particles

Procedure: AGR-CHAR-PIP-09 Rev. 0

Coated particle composite ID: LEU10

Coated particle composite description: AGR-2 B&W UO2 Fuel, from G73H-10-93085B

Property	Measured Data				Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value	INL SPC-923				
IPyC anisotropy (BAFo equivalent)	1.0334	0.0027	10	1.833	mean ≤ 1.045	$B = x + ts/\sqrt{n} \leq 1.045$	1.035	pass	DRF-18
				3.981	dispersion $\leq 0.01 \geq 1.06$	$D = x + ks < 1.06$	1.044	pass	
OPyC anisotropy (BAFo equivalent)	1.0219	0.0012	10	1.833	mean ≤ 1.035	$B = x + ts/\sqrt{n} \leq 1.035$	1.023	pass	DRF-18
				3.981	dispersion $\leq 0.01 \geq 1.06$	$D = x + ks < 1.06$	1.027	pass	

Comments

LEU10 was obtained by combining 3 sublots of G73H-10-93085B (NP-B8488, NP-B8489, and NP-B8490).

John Hum

QC Supervisor

10-19-09

Date

Accept coated particle composite (Yes or No): Yes

J.S. Dwyer for MC Vance

QA Reviewer

10/21/09

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:	M09052602
Sample ID:	LEU10-B01
Sample Description:	AGR-2 B&W U02 Fuel, from G73H-10-93085B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R09052602\

Particle #	Grid Position	Diattenuation			Equivalent BAfo = 1+3N		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0117	0.0027	0.0008	1.0351	0.0081	0.0024
2	4,5	0.0107	0.0022	0.0007	1.0321	0.0066	0.0021
3	4,6	0.0106	0.0025	0.0007	1.0318	0.0075	0.0021
4	5,4	0.0104	0.0027	0.0008	1.0312	0.0081	0.0024
5	5,5	0.0123	0.0024	0.0007	1.0369	0.0072	0.0021
6	5,6	0.0097	0.0024	0.0007	1.0291	0.0072	0.0021
7	6,4	0.0103	0.0028	0.0009	1.0309	0.0084	0.0027
8	6,5	0.0121	0.0022	0.0008	1.0363	0.0066	0.0024
9	6,6	0.0121	0.0022	0.0007	1.0363	0.0066	0.0021
10	7,7	0.0115	0.0023	0.0007	1.0345	0.0069	0.0021
Average		0.0111	0.0024	0.0008	1.0334	0.0073	0.0023

Mean of average BAfo per particle:	1.0334
Standard deviation of average BAfo per particle:	0.0027

Comments

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G. E. Jellison
Operator

July 2, 2009
Date:

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

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M09052602
Sample ID:	LEU10-B01
Sample Description:	AGR-2 B&W U02 Fuel, from G73H-10-93085B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R09052602\

Particle #	Grid Position	Diattenuation			Equivalent BAFO = 1+3N		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0073	0.0027	0.0015	1.0219	0.0081	0.0045
2	4,5	0.0070	0.0022	0.0010	1.0210	0.0066	0.0030
3	4,6	0.0065	0.0024	0.0011	1.0195	0.0072	0.0033
4	5,4	0.0070	0.0027	0.0015	1.0210	0.0081	0.0045
5	5,5	0.0075	0.0025	0.0011	1.0225	0.0075	0.0033
6	5,6	0.0072	0.0027	0.0014	1.0216	0.0081	0.0042
7	6,4	0.0073	0.0029	0.0017	1.0219	0.0087	0.0051
8	6,5	0.0078	0.0032	0.0013	1.0234	0.0096	0.0039
9	6,6	0.0078	0.0029	0.0014	1.0234	0.0087	0.0042
10	7,7	0.0076	0.0029	0.0015	1.0228	0.0087	0.0045
Average		0.0073	0.0027	0.0014	1.0219	0.0081	0.0041

Mean of average BAFO per particle:	1.0219
Standard deviation of average BAFO per particle:	0.0012

Comments

G. E. Jellison
Operator

July 2, 2009
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:	M09052602
Sample ID:	LEU10-B01
Sample Description:	AGR-2 B&W U02 Fuel, from G73H-10-93085B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R09052602\

Particle #	Grid Position	Diattenuation			True BAfo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0117	0.0027	0.0008	1.0237	0.0055	0.0016
2	4,5	0.0107	0.0022	0.0007	1.0216	0.0045	0.0014
3	4,6	0.0106	0.0025	0.0007	1.0214	0.0051	0.0014
4	5,4	0.0104	0.0027	0.0008	1.0210	0.0055	0.0016
5	5,5	0.0123	0.0024	0.0007	1.0249	0.0049	0.0014
6	5,6	0.0097	0.0024	0.0007	1.0196	0.0049	0.0014
7	6,4	0.0103	0.0028	0.0009	1.0208	0.0057	0.0018
8	6,5	0.0121	0.0022	0.0008	1.0245	0.0045	0.0016
9	6,6	0.0121	0.0022	0.0007	1.0245	0.0045	0.0014
10	7,7	0.0115	0.0023	0.0007	1.0233	0.0047	0.0014
Average		0.0111	0.0024	0.0008	1.0225	0.0050	0.0015

Mean of average BAfo per particle:	1.0225
Standard deviation of average BAfo per particle:	0.0019

Comments

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<i>G. E. Jellison</i> Operator	<i>July 2 2009</i> Date
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Data Report Form DRF-18B: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - OPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M09052602
Sample ID:	LEU10-B01
Sample Description:	AGR-2 B&W U02 Fuel, from G73H-10-93085B
Folder containing data:	\\mc-agr\AGR\2-MGEM\R09052602\

Particle #	Grid Position	Diattenuation			True BAFO = $(1+N)/(1-N)$		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0073	0.0027	0.0015	1.0147	0.0055	0.0030
2	4,5	0.0070	0.0022	0.0010	1.0141	0.0045	0.0020
3	4,6	0.0065	0.0024	0.0011	1.0131	0.0049	0.0022
4	5,4	0.0070	0.0027	0.0015	1.0141	0.0055	0.0030
5	5,5	0.0075	0.0025	0.0011	1.0151	0.0051	0.0022
6	5,6	0.0072	0.0027	0.0014	1.0145	0.0055	0.0028
7	6,4	0.0073	0.0029	0.0017	1.0147	0.0059	0.0035
8	6,5	0.0078	0.0032	0.0013	1.0157	0.0065	0.0026
9	6,6	0.0078	0.0029	0.0014	1.0157	0.0059	0.0028
10	7,7	0.0076	0.0029	0.0015	1.0153	0.0059	0.0030
Average		0.0073	0.0027	0.0014	1.0147	0.0055	0.0027

Mean of average BAFO per particle:	1.0147
Standard deviation of average BAFO per particle:	0.0008

Comments

G. E. Jellison
Operator

July 22 2009
Date

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

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	LEU10-C00
Particle Lot Description:	AGR-2 B&W U02 Fuel, from G73H-10-93085B
Filename:	\\mnc-agr\AGR\ParticleWeight\W09052201_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	0.1594	0.1571	0.1407	0.1348	0.2127
Number of particles:	109	106	96	93	145
Average weight/particle (g):	1.462E-03	1.482E-03	1.466E-03	1.449E-03	1.467E-03

Mean average weight/particle (g):	1.465E-03
Standard error in mean average weight/particle (g):	5.21E-06

Dixie Barker
Operator

5-22-09
Date

Data Report Form DRF-32: Counting of Particles with SiC Soot Inclusion Defects by Visual Inspection

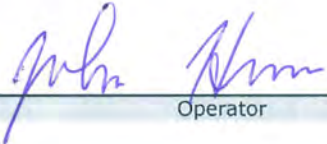
Procedure:	AGR-CHAR-DAM-32 Rev. 0
Operator:	John Hunn/Jason Ramey/Fred Montgomery
Sample ID:	LEU10-E01
Sample Description:	AGR-2 B&W UO2 fuel, from G73H-10-93085B
Folder containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Inclusions\P09061101\
DRF filename:	\\mc-agr\AGR\ImageProcessing\Completed_Inclusions\P09061101_DRF32R0.xls

Mean average weight/particle (g):	1.47E-03
Uncertainty in average weight/particle (g):	5.21E-06
Weight of sample of particles (g):	5.143
Approximate number of particles in sample:	3510
Uncertainty in number of particles in sample:	12

Number of particles with SiC soot inclusion defects:	3
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Comments

3/3510 corresponds to a $<2.3\text{E-}3$ defect fraction at 95% confidence.
Two identified defects appear to be standard carbon soot inclusions, the third was determined by SEM/EDS to be a molybdenum inclusion.


Operator

7-26-2010
Date

Three particles were identified as exhibiting inclusions in the SiC layer. A defect fraction of 3/3510 corresponds to $<2.3\text{E-}3$ at 95% confidence. Figures 1 and 2 show what appear to be carbon soot inclusions. These features are similar to, but do not appear to be polishing artifacts because they follow the curvature of the SiC. More significant is the bright inclusion in the SiC of the particle in Figure 3. A bulge associated with this inclusion indicates that it is not a polishing artifact.

The inclusion in Figure 3 was analyzed by scanning electron microscopy (SEM). Backscattered electron imaging (Figures 4 and 5) shows the SiC grain structure and indicates that the inclusion is a heavy element (higher scattering cross-section). Energy dispersive x-ray spectroscopy (EDS) identified the inclusion as containing molybdenum with possible trace amounts of tungsten and iron (Figure 6). The molybdenum inclusion does not appear as a single lump of material surrounded by SiC, as is often observed for cases of foreign matter trapped in a coating layer. The Mo is intimately dispersed in the SiC, indicating a possible reaction with the Si. Both metallic molybdenum and molybdenum disilicide would have a metallic appearance, as observed in the optical images (the carbide would be black).

Some particles were noted as having abnormal regions in the inner pyrocarbon (IPyC) layer, near the Buffer/IPyC interface. An example of a large anomaly of this type is shown in Figure 7. An abnormal region of typical size is shown in Figure 8. The fraction of these anomalies was not rigorously determined, but based on observed anomalies the estimated population is about 13/1170, which corresponds to $<1.8\text{E-}2$ at 95% confidence.

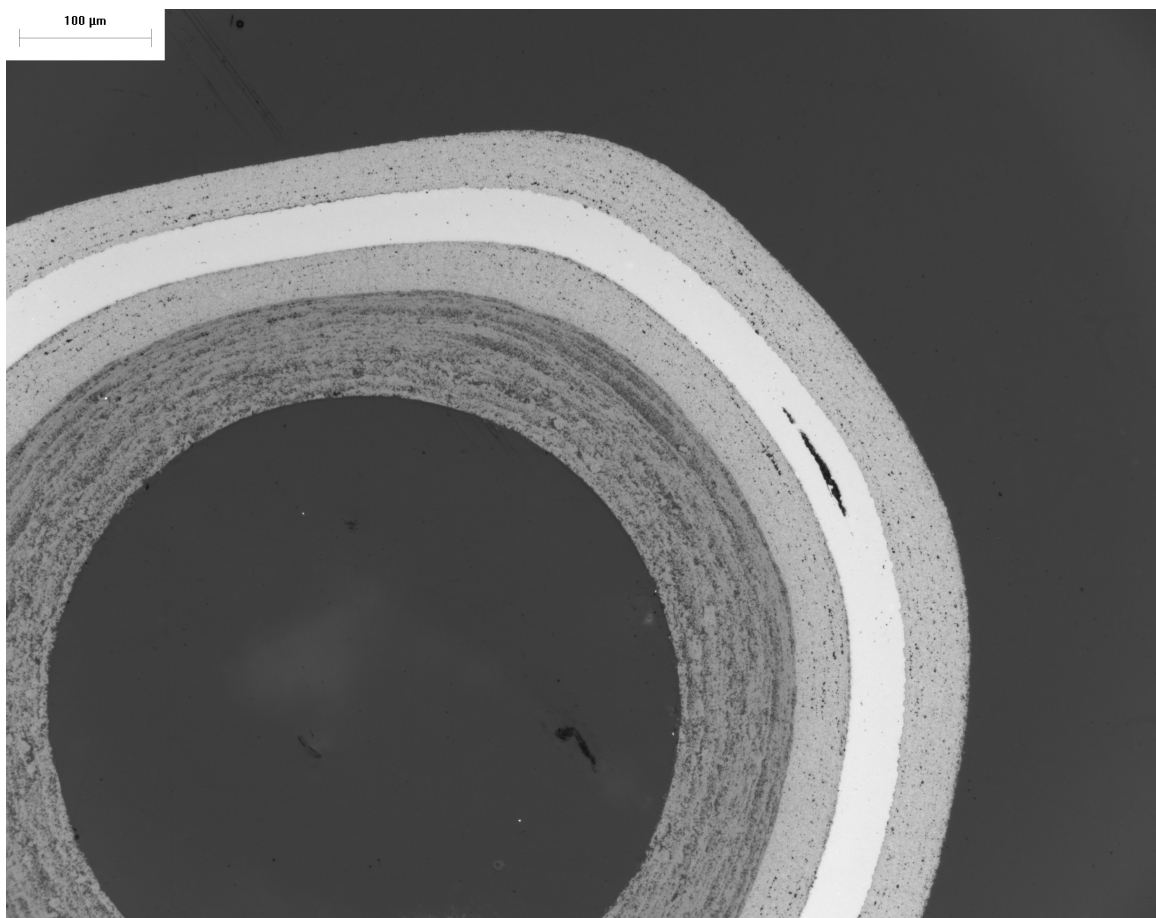


Figure 1. Image of particle from mount M09052704, frame 040 showing possible soot inclusion in SiC.

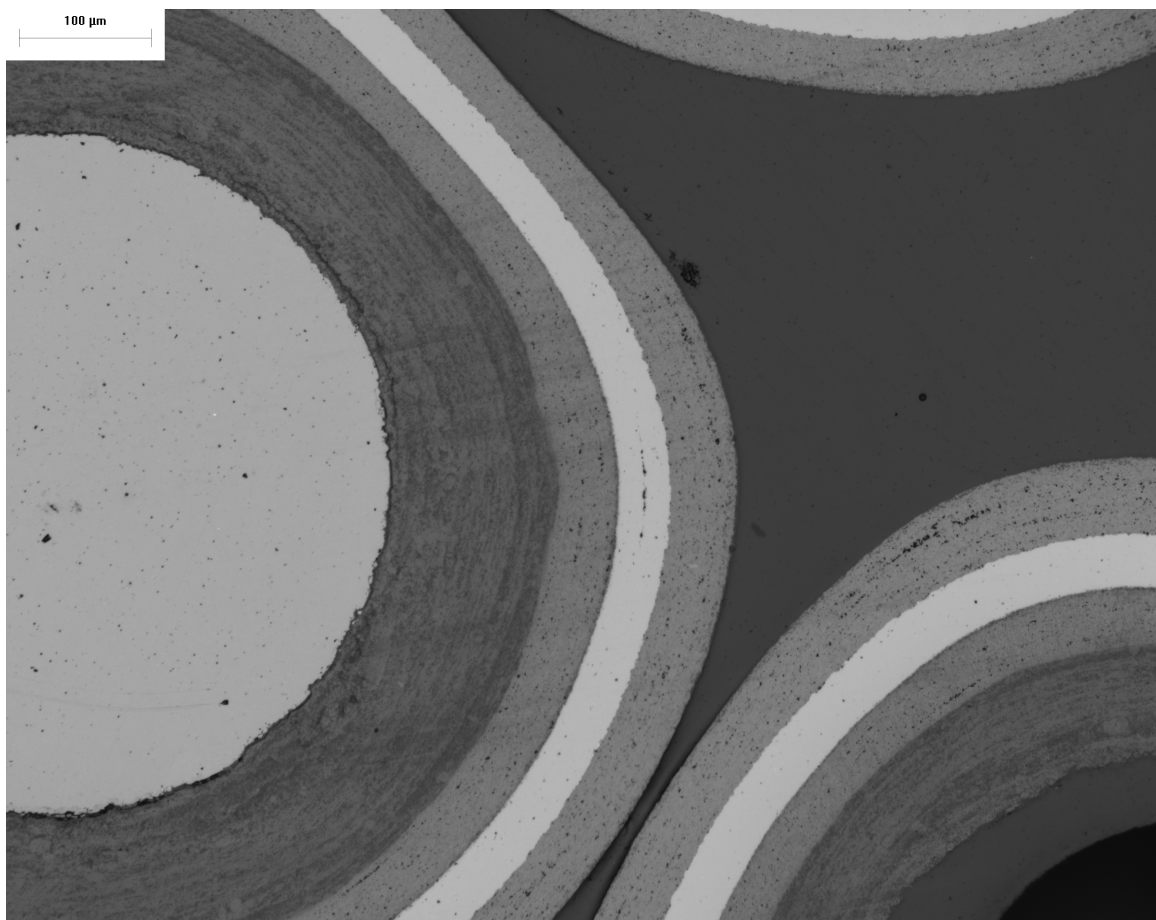


Figure 2. Image of particle from mount M09052701, frame 083 showing possible soot inclusion in SiC.

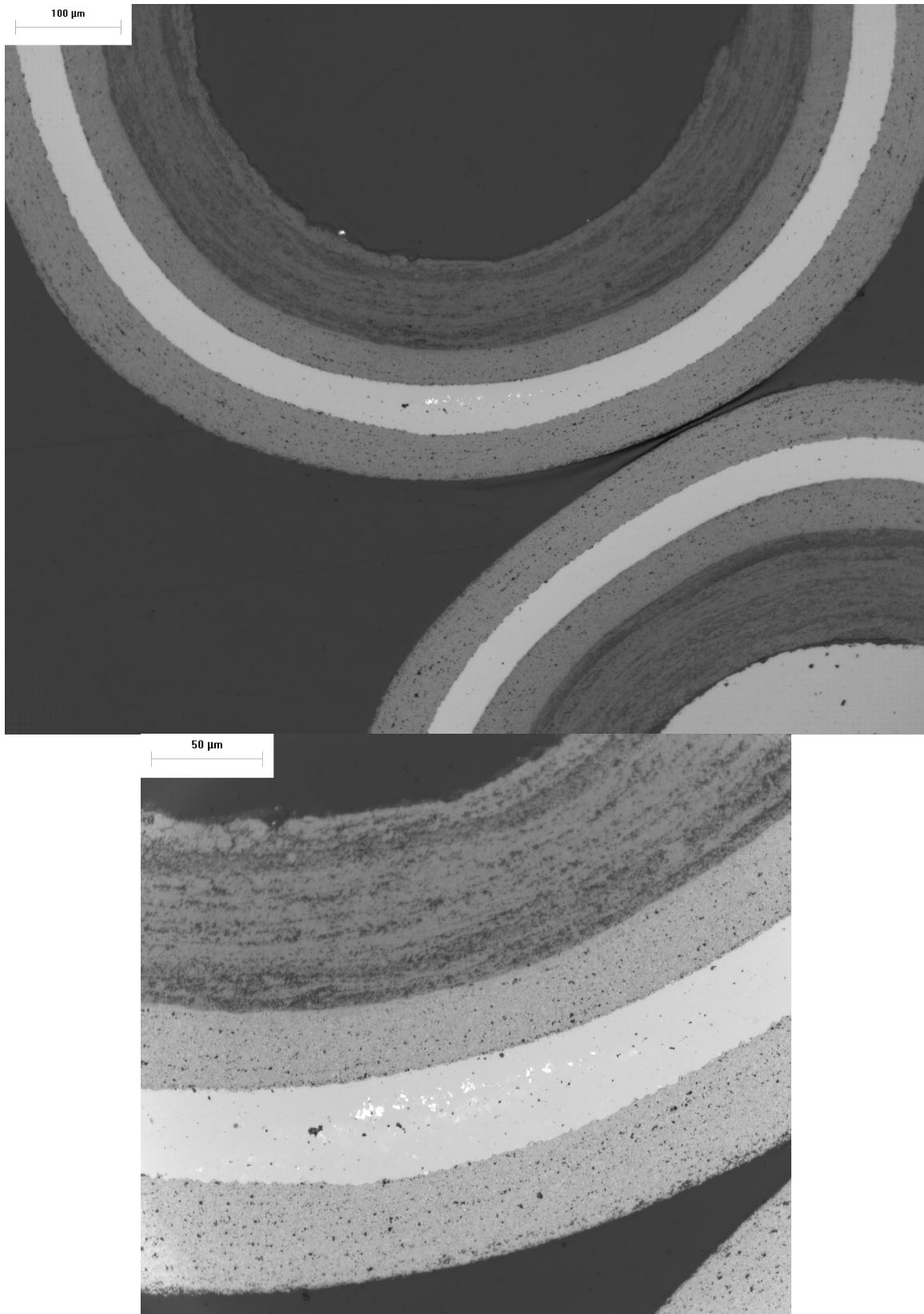


Figure 3. Images of particle in mount M09052701, frame 052 showing Mo inclusion in SiC layer.

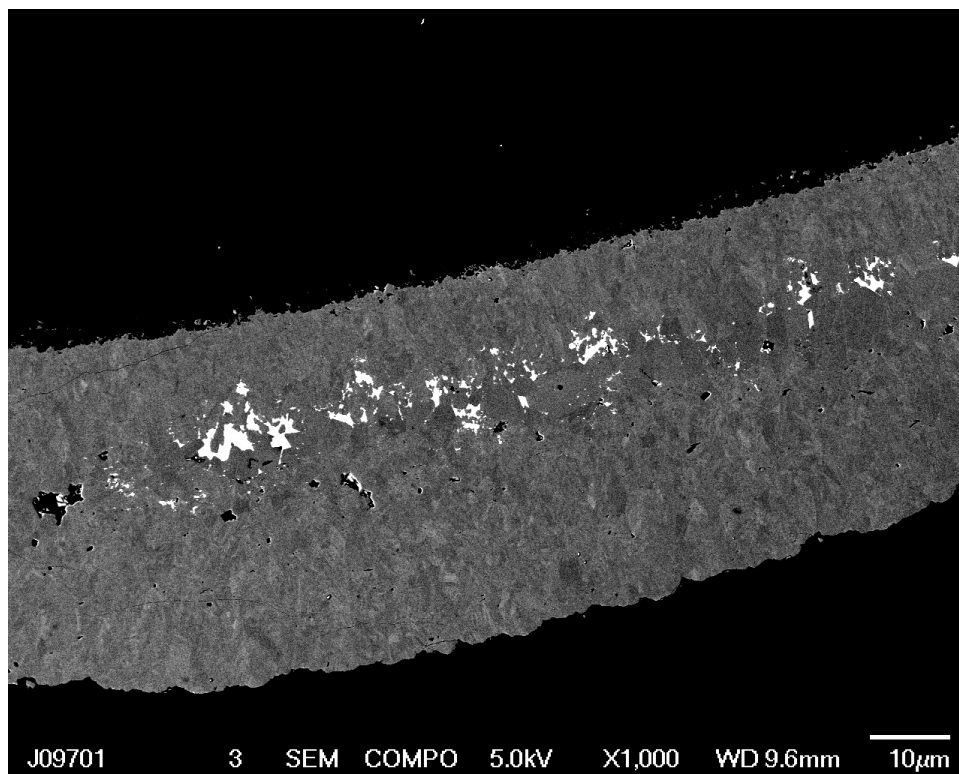


Figure 4. Backscattered electron image of Mo inclusion in SiC layer of particle in Figure 3.

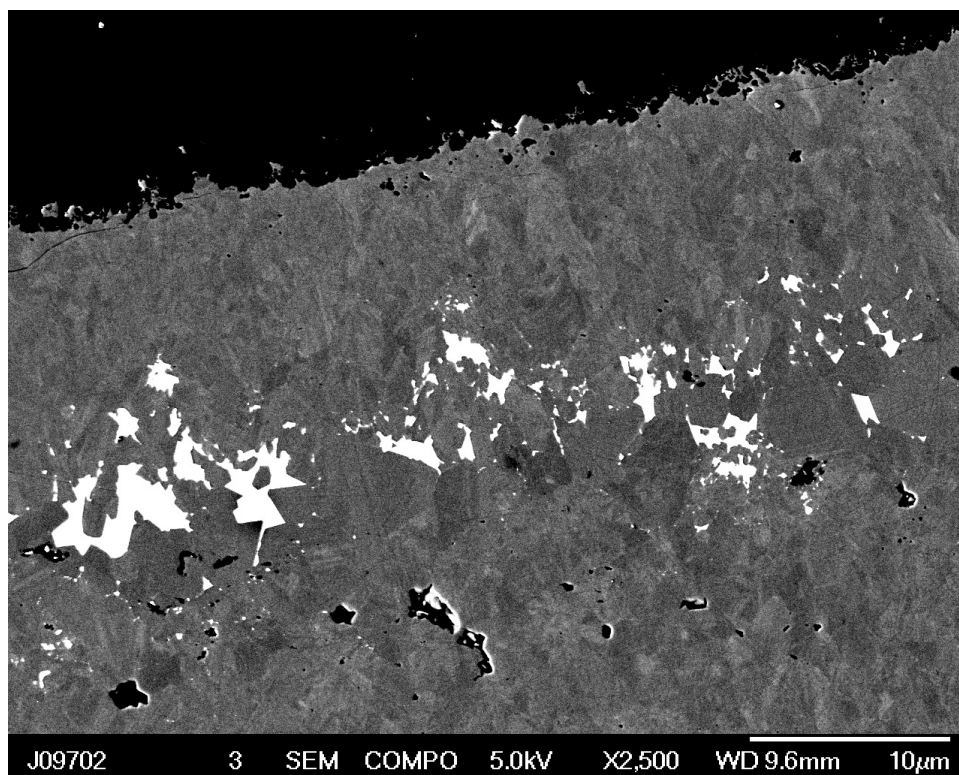


Figure 5. Backscattered electron image of Mo inclusion in SiC layer of particle in Figure 3.

Label A: j09694c inclusion

Label B: j09694b matrix

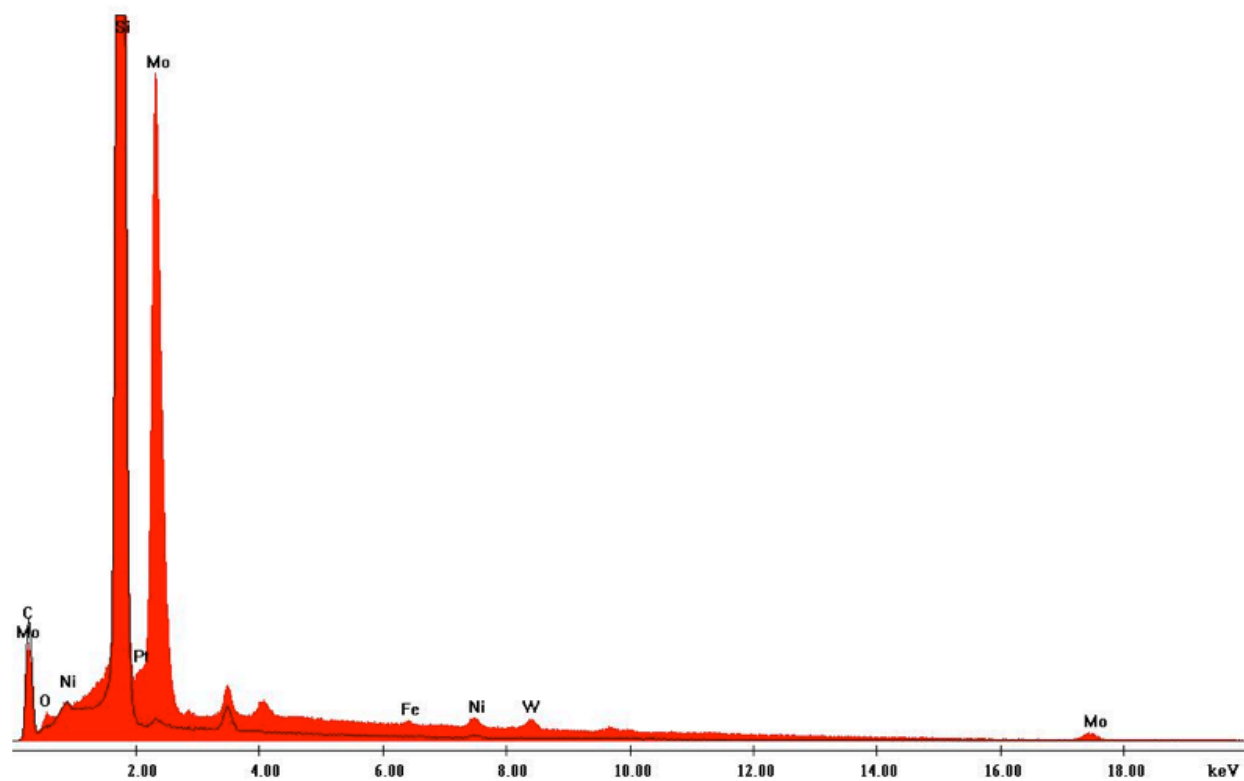


Figure 6. EDS spectra of Mo inclusion in SiC layer. Black line shows background spectra of clean SiC.

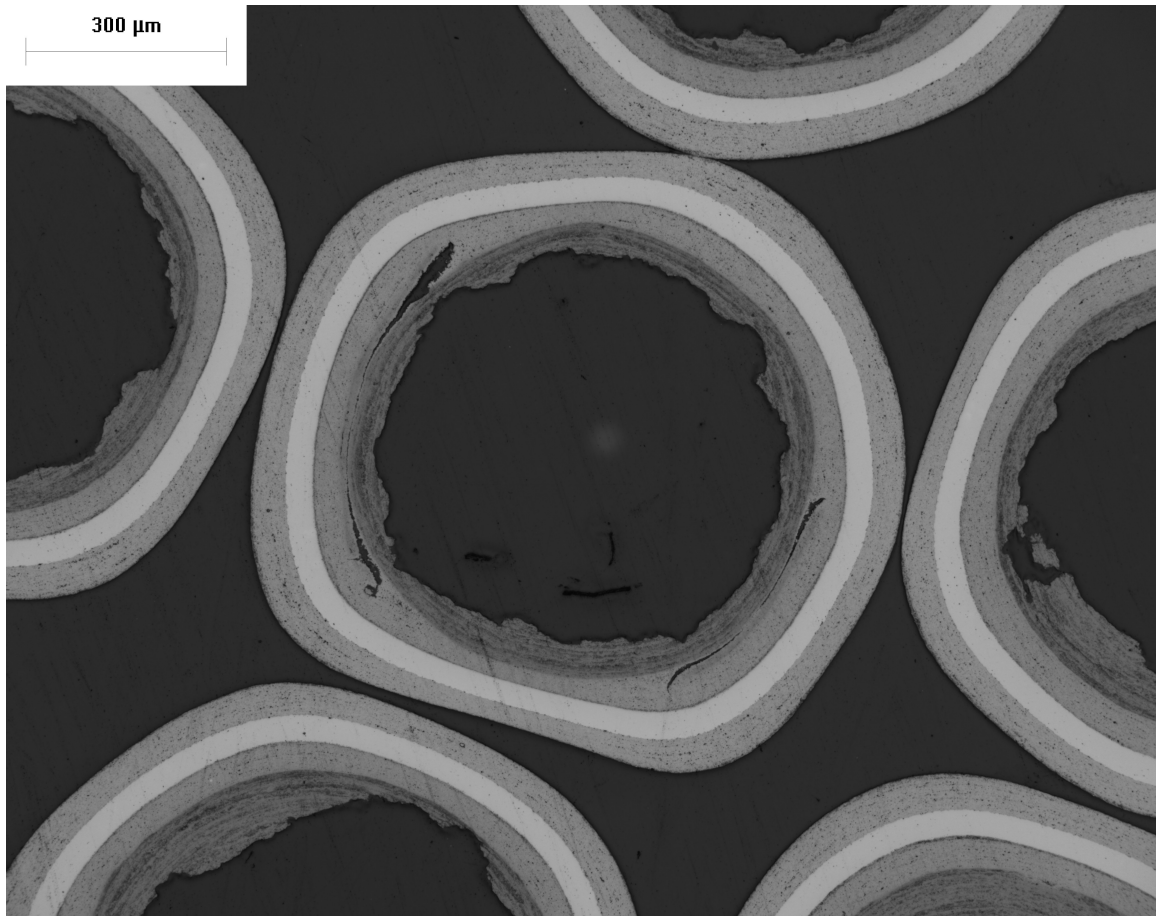


Figure 7. Image of particle from mount M09052702, frame 039 showing abnormal region at Buffer/IPyC interface.

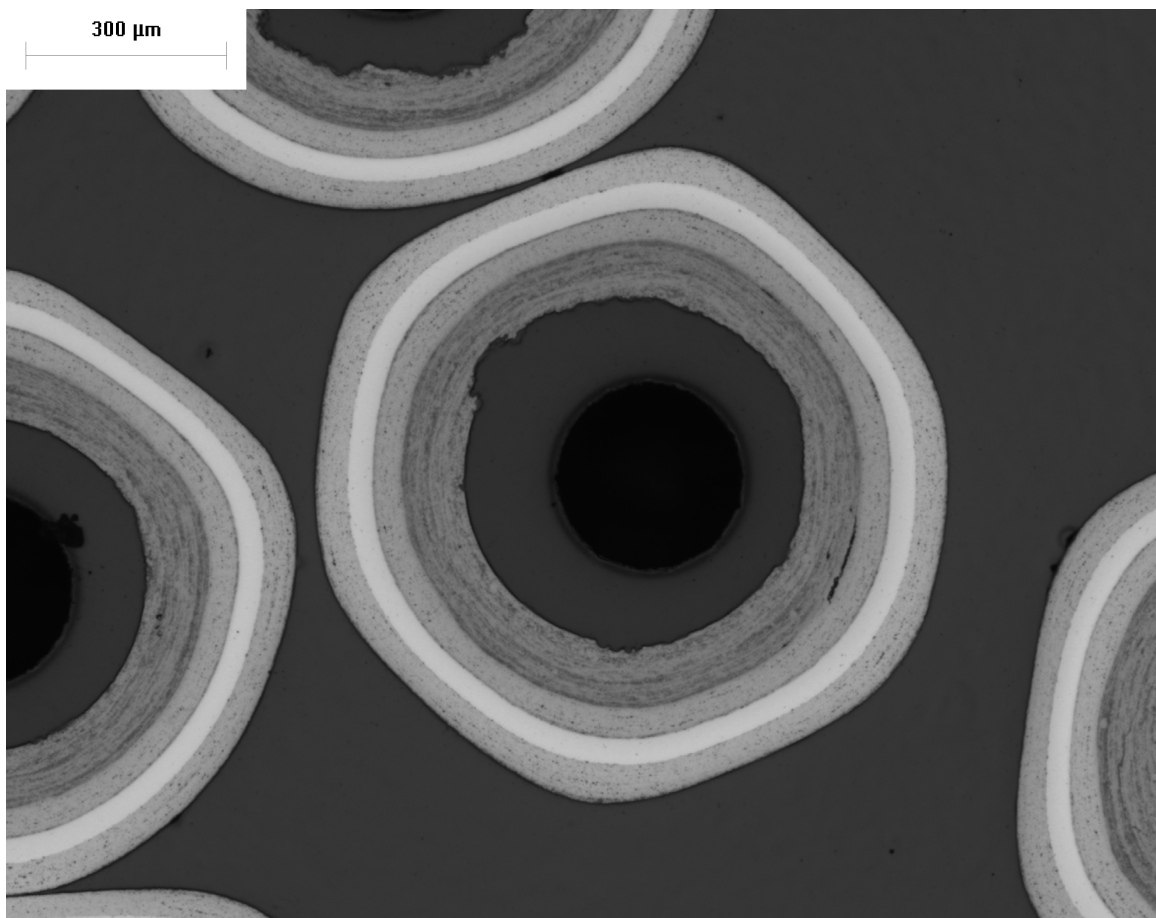


Figure 8. Image of particle from mount M09052701, frame 065 showing abnormal region at Buffer/IPyC interface.