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# AGR-5/6/7 Final Fuel Characterization

Douglas W. Marshall

May 2018


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**http://www.inl.gov**

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# AGR-5/6/7 ‘Final’ Fuel Characterization

*Douglas Marshall*

*TRISO Fuel Fabrication Technical Lead*

Gas-Cooled Reactor Program Review Meeting
May 8, 2018, at Idaho National Laboratory

ART
ADVANCED REACTOR TECHNOLOGIES

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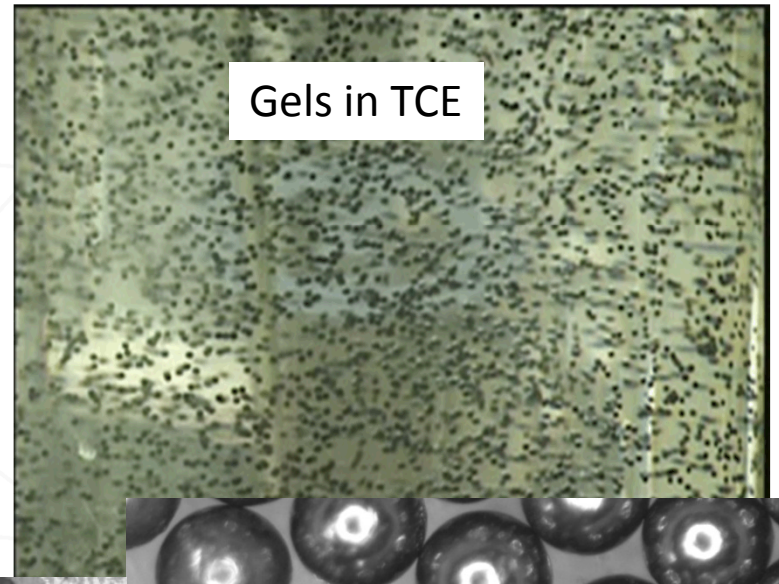
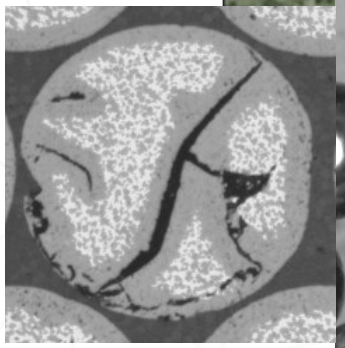
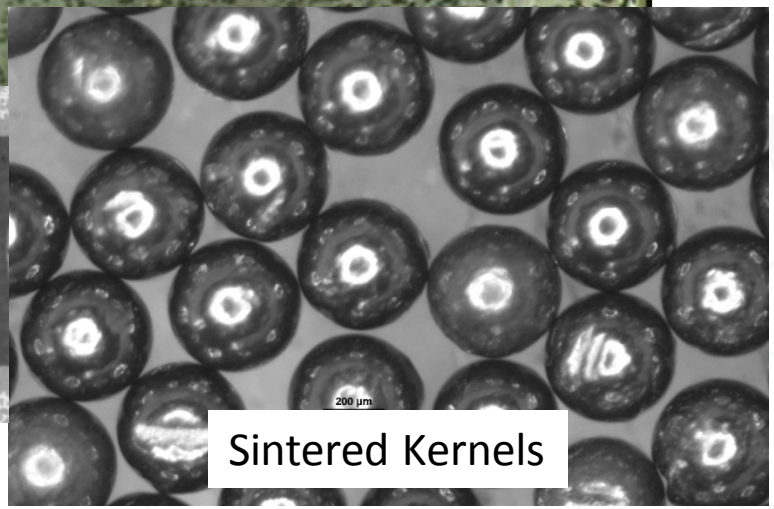
# Historical Overview – Kernels

- Formed and sintered in production-scale equipment since AGR-1
- Internal gelation sol-gel process is used
  - Chilled uranyl nitrate broth with HMTA, urea, and carbon black dribbled into a column of warm TCE
  - Gels are “aged,” washed, dried in a collection pot
  - Dried (or “green”) gels are calcined and sintered in a spouted bed furnace
- Stochastic fissuring potential
- J52L-16-69316 (2013) was too fissured for use
- J52R-16-69317 (2016) had far fewer fissures



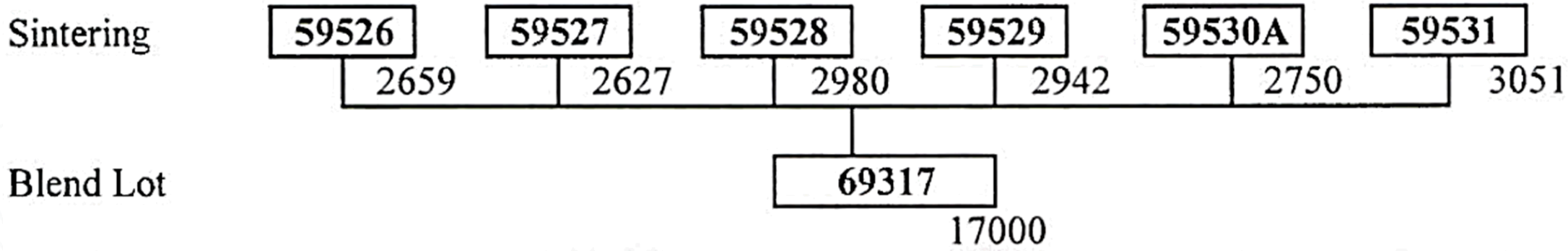



Fissured kernel



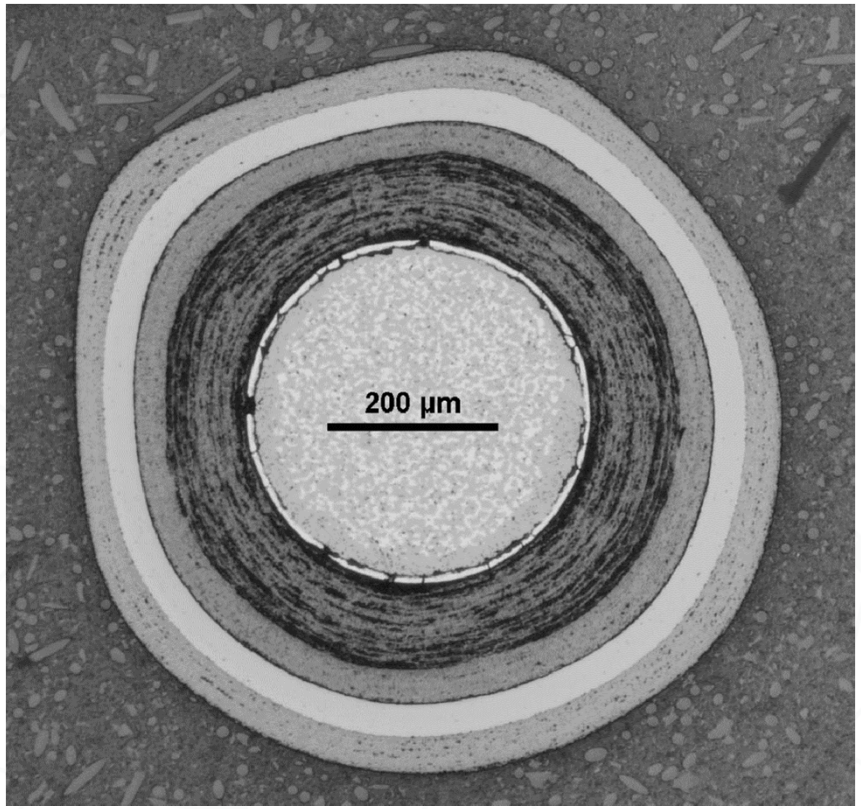
# Historical Overview – Kernels (cont.)

- Composited from 6 kernel sintering batches; yielding 17 kg of certified kernels in J52R-16-69317



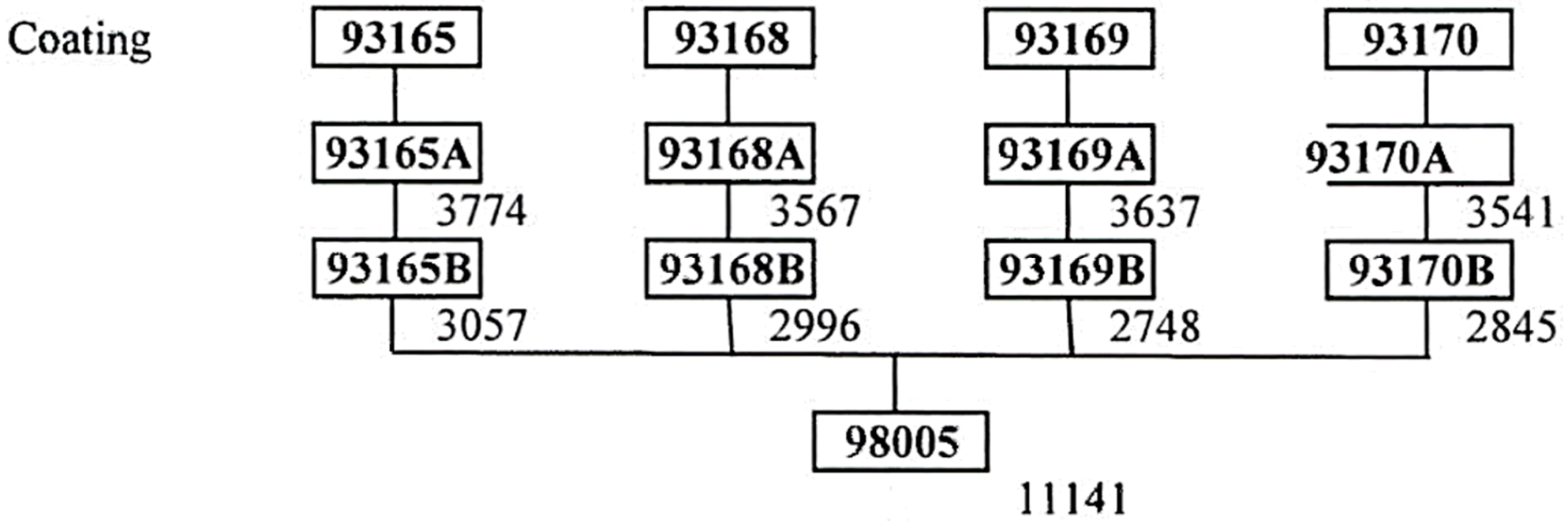
# Historical Overview – TRISO

- Tristructural isotropic (TRISO) fuel particles coated in a spouted bed furnace retort
- AGR-1 and AGR-3/4 coating applied at ORNL in a 2” diameter retort
- AGR-2 and AGR-5/6/7 coated in a small, production-scale (6” diameter) retort
- Four coatings applied in a semi-continuous operation
  - Buffer
  - Inner pyrocarbon
  - Silicon carbide
  - Outer pyrocarbon
- TRISO particle lot J52R-16-98005 was certified for AGR-5/6/7 in May, 2017



# Historical Overview – TRISO (cont.)

- Four TRISO batches were composited, after two sieving operations to reject fractured kernels, into a 11.1 kg TRISO particle lot J52R-16-98005



# Historical Overview – Compacts

- TRISO particles are overcoated with a resin-infused graphite powder and pressed into cylindrical fuel "compacts"
- AGR-1, AGR-2, and AGR-3/4 fuels formed in laboratory-scale equipment
  - Small tilted drum overcoater
  - Pressed one at a time with weighed charges
  - Two furnaces for carbonization and thermal treatment
- AGR-5/6/7 fuel formed in engineering-scale equipment
  - 1-2 kg of TRISO overcoated in an hour in pharmaceutical equipment
  - 4-hole automated press
  - Single furnace for carbonization and thermal treatment

Particle Overcoater

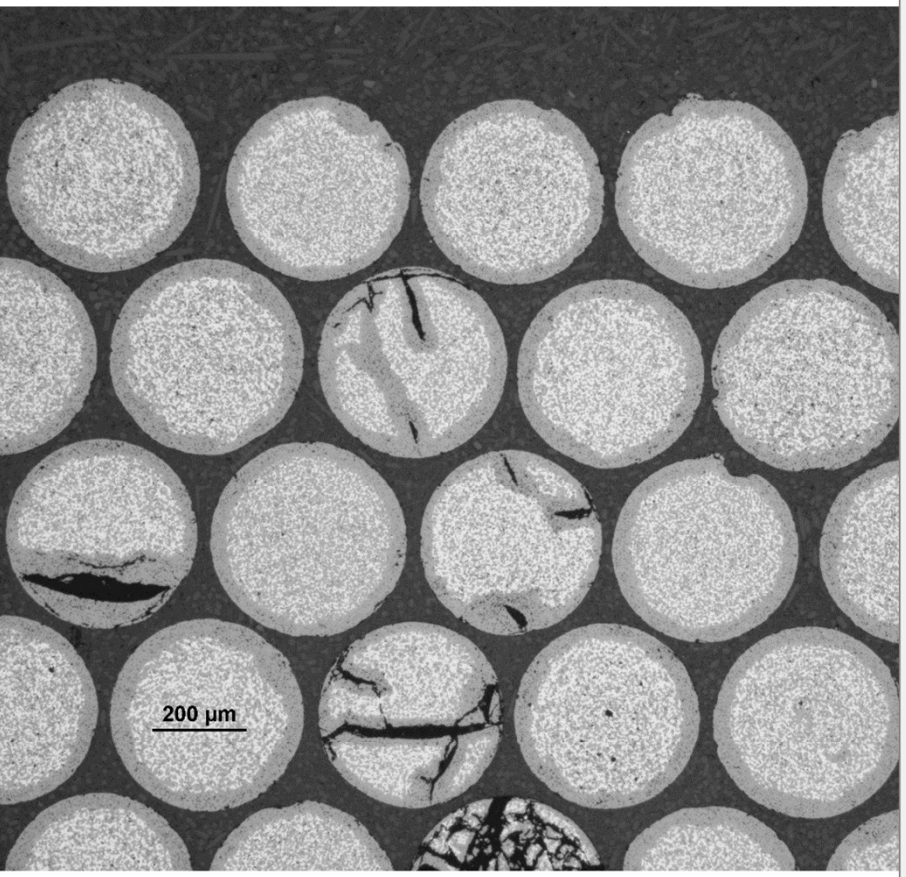
Compacting Press

# Kernel Lot Characterization

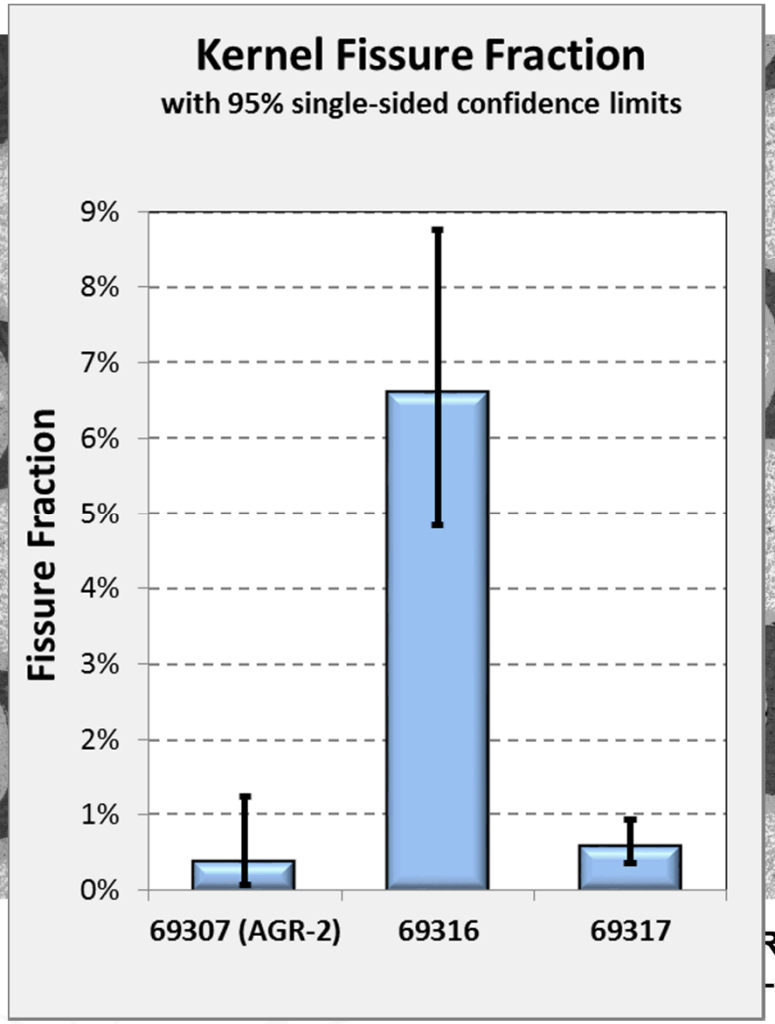
| J52R-16-69317 | Specification | Mean | Status* |
|---|---|---|---|
| Diameter (μm) | 425 ± 10 | 425.8 | **Pass** |
| Envelope density (g/cm$^3$) | ≥10.4 | 11.05 | **Pass** |
| Uranium fraction (gU/gUCO) | ≥0.885 | 0.897 | **Pass** |
| $^{235}$U enrichment (g$^{235}$U/gU) | 0.155 ± 0.001 | 0.1548 | **Pass** |
| C:U (atomic ratio) | 0.40 ± 0.10 | 0.37 | **Pass** |
| O:U (atomic ratio) | 1.50 ± 0.20 | 1.441 | **Pass** |
| (C+O)/U (atomic ratio) | ≤2.0 | 1.811 | **Pass** |
| Individual impurities (ppmw):<br>Li, Na, Al, Cl, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, and Zn | ≤100 each | 5-25 | **Pass** |
| Process impurities (ppmw): P, S | ≤1,500 each | (P) 5 | **Pass** |
| Aspect ratio | ≤ 1.05 @ .95/.90 | 1.012 | **Pass** |

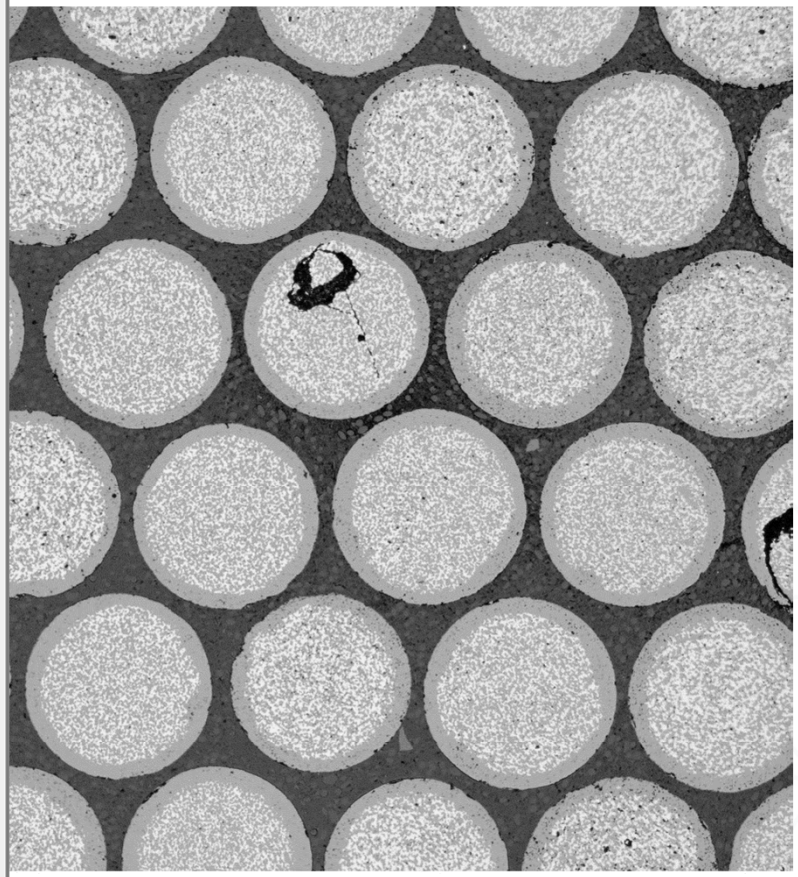
* Pass ≡ meets all specifications on the mean and dispersion critical limits.

# Kernel Lot Composition and Characterization




J52L-16-69316 Analysis #3



R-16-69317 Analysis Sample #3
- Polished Cross Section of Kernels

# TRISO Lot Composition and Characterization

| J52R-16-98005 | Specification | Mean | Status* |
|---|---|---|---|
| Buffer thickness (μm) | 100 ± 15 | 100.4 | **Pass** |
| IPyC thickness (μm) | 40 ± 4 | 39.24 | **Pass** |
| SiC thickness (μm) | 35 ± 3 | 36.15 | **Pass** |
| OPyC thickness (μm) | 40 ± 4 | 35.03 | **Low mean** |
| Buffer density (g/cm$^3$) | 1.05 ± 0.10 | 1.031 | **Pass** |
| IPyC density (g/cm$^3$) | 1.90 ± 0.05 | 1.897 | **Pass** |
| SiC density (g/cm$^3$) | ≥ 3.19 | 3.195 | **Pass** |
| OPyC density (g/cm$^3$) | 1.90 ± 0.05 | 1.897 | **Pass** |

* Pass ≡ meets all specifications on the mean and dispersion critical limits.

# TRISO Lot Composition and Characterization

| J52R-16-98005 | Specification | Mean | Status* |
|---|---|---|---|
| IPyC diattenuation | ≤ 0.0170 | 0.0153 | **Pass** |
| OPyC diattenuation | ≤ 0.0122 | 0.0102 | **Pass** |
| SiC aspect ratio (faceting) | ≤ 1.14 @ .95/.99 | 1.053 | **Pass** |
| Defective IPyC coating fraction | $\leq 1.0 \times 10^{-4}$ | 4.5E-5 | **Pass** |
| Defective OPyC defect fraction | $\leq 3.0 \times 10^{-4}$ | 0/35k | **Pass** |
| Dispersed U Fraction | (≤ 1.0E-5)** | 1.04E-5 | **High** |
| Exposed Kernel Fraction | (≤ 5.0E-5)** | 9.40E-6 | **Acceptable** |
| SiC Defect Fraction | (≤ 1.0E-4)** | 1.89E-5 | **Acceptable** |

* Pass ≡ meets all specifications on the mean and dispersion critical limits.

** Compact specification isn't relevant to TRISO particles.

# Compact Characterization

- Four batches of overcoated TRISO were separately pressed and divided across four furnace runs for thermal treatment

| Pressing ↓\ Furnace → | A | B | C | D |
|---|---|---|---|---|
| **14154** (39.4% PF) | 108 | 108 | 108 | 140 |
| **14155** (39.3% PF) | 108 | 108 | 108 | 160 |
| **14156** (26.1% PF) | 108 | 108 | 108 | 36 |
| **14157** (25.9% PF) | 108 | 108 | 108 | 0 |

- A-series compacts were used in AGR-5/6/7 while the C and D-series were used for destructive characterization
- Pressure sensor failure interrupted furnace run B between carbonization and heat-treatment cycles. The other three runs used a continuous or “combined” cycle from carbonization through thermal treatment

# Compact Characterization

| Property | Specification | 14154A | 14155A | 14156A | 14157A |
|---|---|---|---|---|---|
| Mean U loading (gU/compact)<br>Nominally 40% PF<br>Nominally 25% PF | <br>1.36 ± 0.10<br>0.90 ± 0.08 | <br>1.428<br>--- | <br>1.388<br>--- | <br>---<br>0.923 | <br>---<br>0.914 |
| Diameter (mm) | 12.20 ≤ D ≤ 12.44 | 12.293 | 12.291 | 12.237 | 12.260 |
| Length (mm) | 24.40 ≤ L ≤ 25.30 | 25.094 | 24.692 | 24.996 | 24.752 |
| Matrix density ($g/cm^3$) | ≥ 1.65 | 1.73 | 1.73 | 1.76 | 1.74 |

# Compact Characterization

| Property | Spec | 14154C | 14155C | 14156C | 14157C |
|---|---|---|---|---|---|
| Iron (μg) | ≤ 25 | < 5 | | | |
| Transition metals (μg)<br>Cr, Mn, Co, and Ni | ≤ 50 each | Cr <25, all others <10 | | | |
| Calcium (μg) | ≤ 50 | <25 | | | |
| Aluminum | ≤ 50 | <25 | | | |
| Titanium + Vanadium | ≤ 240 | <20 | | | |

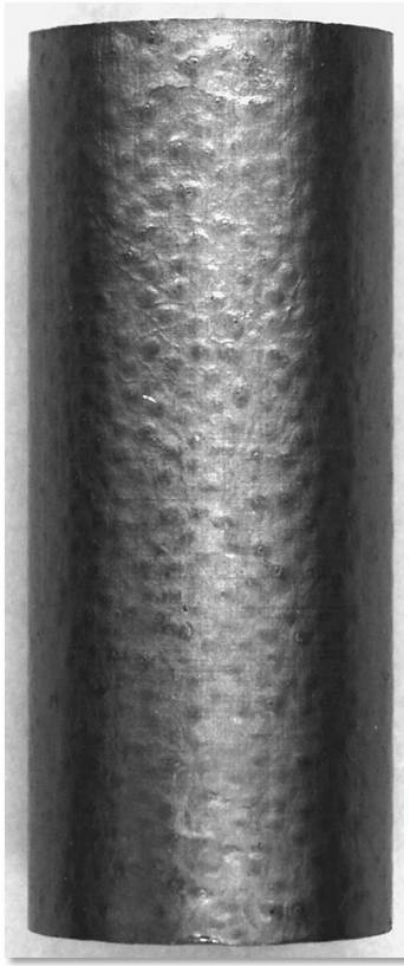
# BWXT-NOG Compact Characterization

| | | 40% PF Compacts | 25% PF Compacts |
|---|---|---|---|
| **Property** | **Spec** | **14154C and 14155C** | **14156C and 14157C** |
| Defective OPyC | ≤ 0.01 | 0/4200 (7.1E-4 @ 95%) | 0/4200 (7.1E-4 @ 95%) |
| Dispersed U Fraction | ≤ 1E-5 | 3.18E-5 (3.80E-5 @ 95%) | 2.66E-5 (2.95E-5 @ 95%) |
| Exposed Kernel Fraction | ≤ 5E-5 | 6.57E-5 (9.28E-5 @ 95%) | 7.39E-6 (1.48E-5 @ 95%) |
| SiC Defect Fraction | ≤ 1E-4 | 6.96E-5 (9.66E-5 @ 95%) | 9.24E-5 (1.22E-4 @ 95%) |

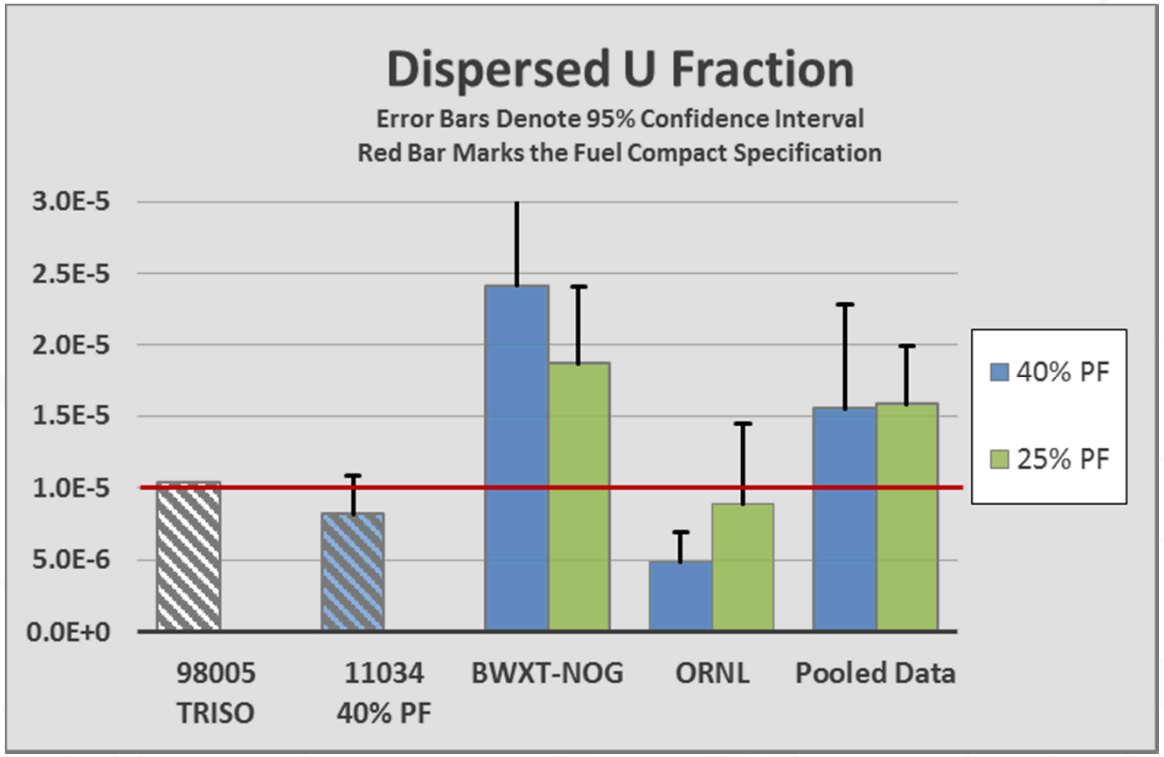
Unexpected results:
The DUF increased to 3× the TRISO lot
The SDF increased 4× - 5×
Inconsistencies in analytical outcomes made some results suspect

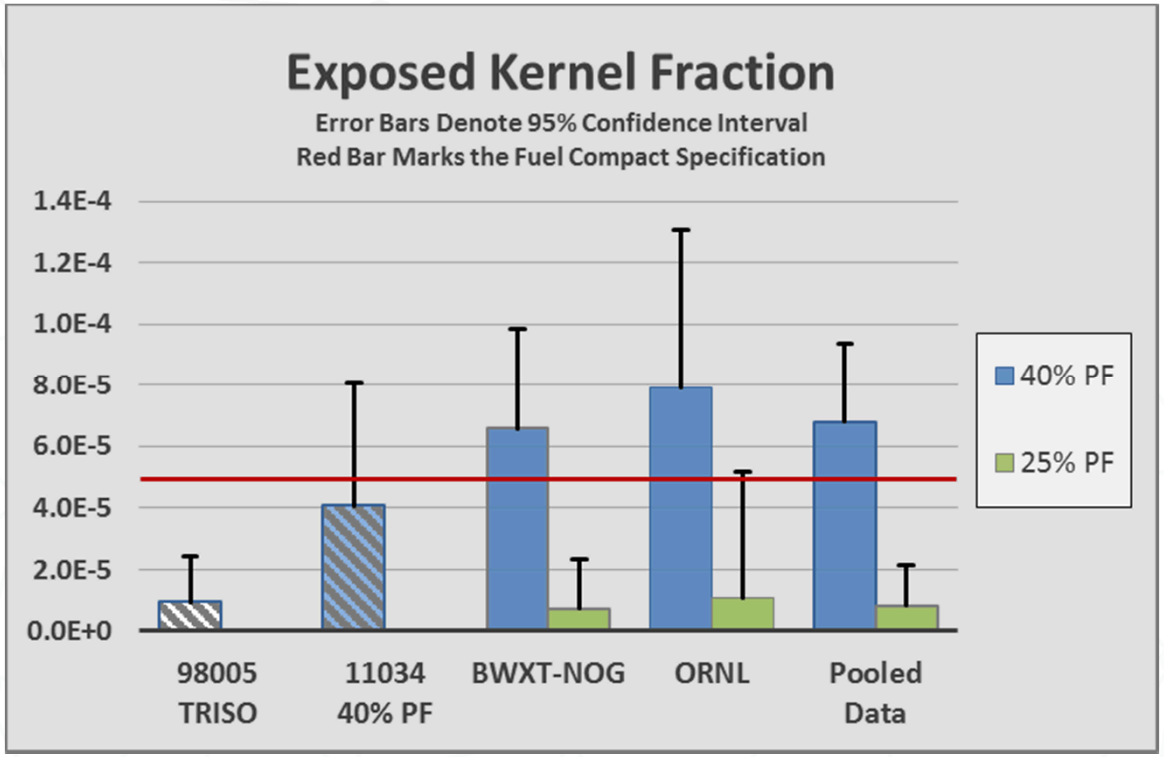
# Compact Characterization

- Confirmatory analysis was requested by ORNL
  - J52R-16-11034 overcoated TRISO (40% PF)
  - 40 compacts from J52R-16-14154C (40% PF) compacts
  - 29 compacts from J52R-16-14156C and 11 compacts from J52R-16-14156D (25% PF)
- ORNL observed unusual analytical results
- Deconsolidate is a thick slurry and difficult to clarify the supernate
- The populations analyzed by ORNL are insufficient to reject any data
- Additional analyses are needed to improve statistics

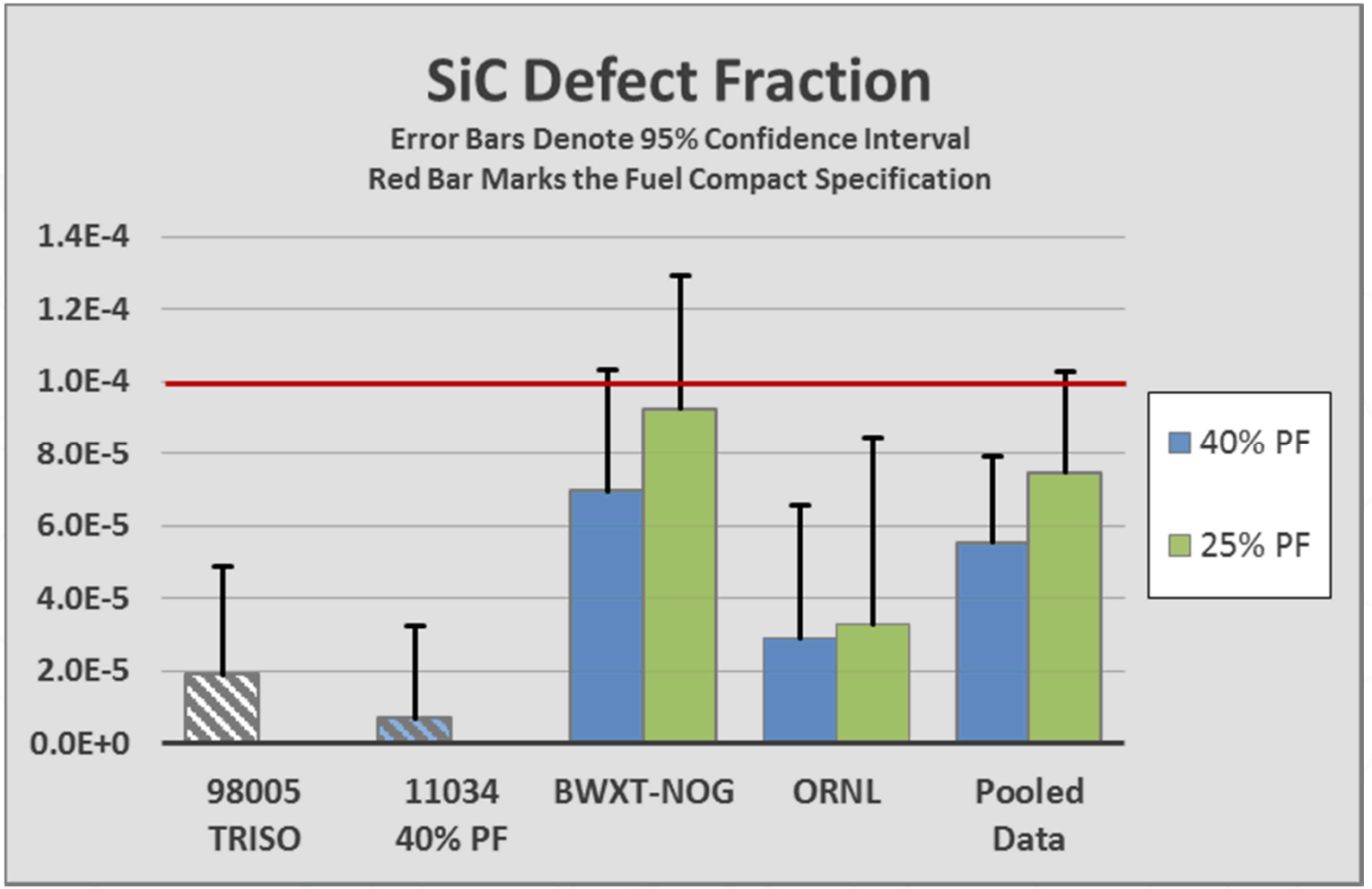


# Compact Characterization



# Compact Characterization



# Summary

- Kernels
  - 17 kg of LEUCO kernels were certified
  - All product specifications were met
  - Fissure fraction is comparable to AGR-2 UCO fuel
- TRISO Particles
  - 11.1 kg of LEUCO TRISO particles were certified
  - All product specifications were met except for OPyC thickness
  - Mean DUF was high, but EKF and SDF were ~1/5$^{th}$ of the compact specification
- Fuel Compacts
  - Fuel compacts of both packing fractions meet all specifications except for DLBL defects
    - DUF is above the specification for both PFs
    - EKF for 25% PF compacts meet specification
    - EKF for 40% PF compacts fail the specification; mostly from damage in the overcoater
    - SDF for both PFs should meet specs with more data
  - ORNL should be tasked to analyze more samples of all compact batches

ADVANCED REACTOR TECHNOLOGIES

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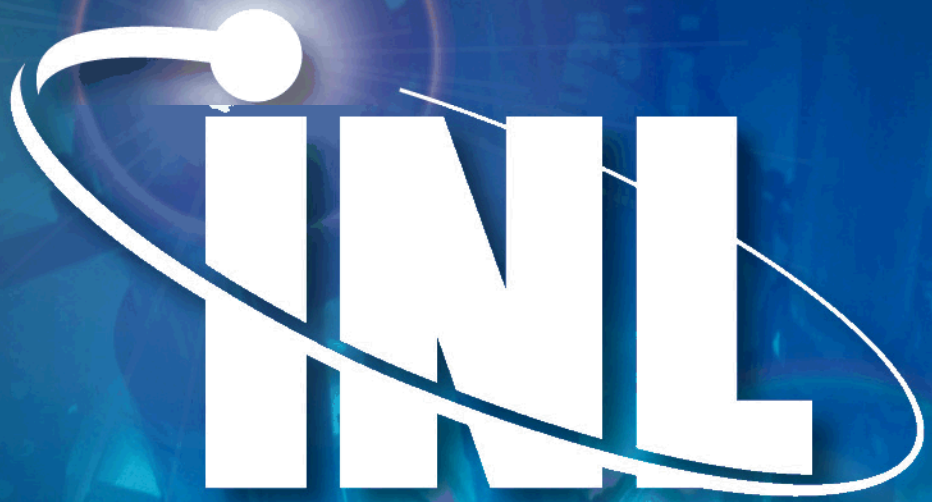


Idaho National Laboratory

# Compact Characterization