**Specification**
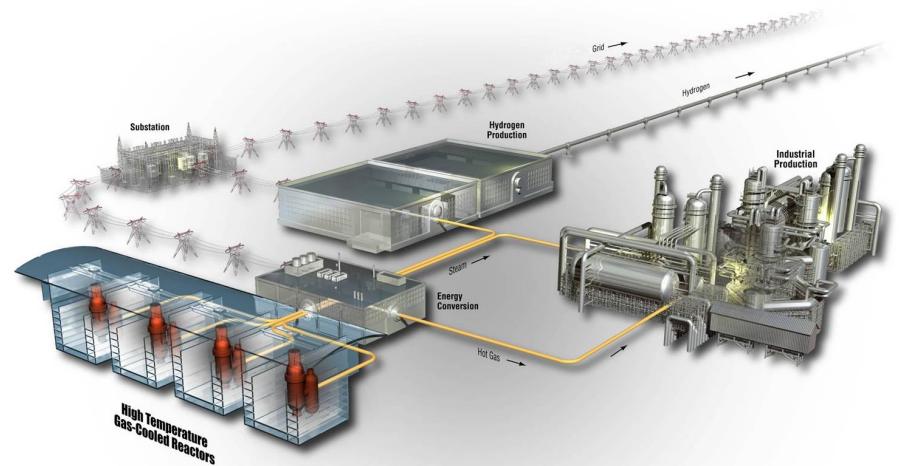
Project No. 23841; 29412

# AGR-5/6/7 Fuel Specification

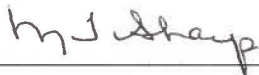The INL is a
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| INL ART TDO Program | Specification (SPC) | eCR Number:  648649 |
|---|---|---|

| SIGNATURES | | | |
|---|---|---|---|
| Signature and Typed or Printed Name | Signature Code | Date (mm/dd/yyyy) | Organization/Discipline |
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P    For Preparer of the document.

A    For Approval: This is for non-owner approvals that may be required as directed by a given program or project. This signature may not be applicable for all uses of this form.

C    For documented review and concurrence.

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# ACRONYMS

| | |
|---|---|
| ADUN | acid-deficient uranyl nitrate |
| AGR | Advanced Gas Reactor |
| ART | Advanced Reactor Technologies |
| ASME | American Society of Mechanical Engineers |
| ATR | Advanced Test Reactor |
| BWXT | BWX Technologies |
| DUF | dispersed uranium fraction |
| EKF | exposed kernel fraction |
| HMCF | heavy-metal contamination fraction (DUF + EKF) |
| HMTA | Hexamethylenetetramine |
| INL | Idaho National Laboratory |
| IPyC | inner pyrolytic carbon |
| LEU | low-enriched uranium |
| LEUCO | low-enriched uranium carbide/oxide |
| NQA | Nuclear Quality Assurance |
| OPyC | outer pyrolytic carbon |
| SiC | silicon carbide |
| TDO | Technology Development Office |
| TRISO | tristructural isotropic |
| UCO | uranium carbide/oxide |

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# PART 1  INTRODUCTION

The Idaho National Laboratory (INL) Advanced Reactor Technologies (ART) Technology Development Office (TDO) program is developing and qualifying fuel for a first-of-a-kind high temperature gas-cooled reactor (HTGR) and associated systems to generate electrical power and produce hydrogen or process heat. One of the critical technology development areas of the program is nuclear fuel development and qualification. Fuel qualification is needed to support licensing, operation, and near-term deployment of an HTGR reactor.[5] Toward this end, the Advanced Gas Reactor (AGR) program includes manufacturing fuels for a series of irradiation experiments in the Advanced Test Reactor (ATR) at INL.[5] The fifth, sixth, and seventh irradiation experiments, called "AGR-5/6/7," will use fuel manufactured entirely in engineering-scale equipment to demonstrate the performance of fuels fabricated in a production environment. This fuel will be composited from multiple low-enriched uranium carbide/oxide (LEUCO) kernel forming runs, multiple tristructural isotropic (TRISO) coating runs, and multiple overcoating runs. The fuel will be composited into lots as kernels, TRISO particles, and compacts.

Previous AGR fuel specifications[1,2,8,9] provide the basis for most of the following fuel specifications, which are specific to the AGR-5/6/7 experiment, and may not be immediately applicable to fuel produced for an HTGR reactor. Fuel specifications will be influenced by the final reactor design.

# PART 2  QUALITY ASSURANCE

During the manufacturing, handling, and shipping of the fuel described in this specification, a documented quality assurance program shall be in effect. The quality assurance program shall conform to the requirements of American Society of Mechanical Engineers (ASME) Nuclear Quality Assurance ASME NQA-1-2008 (with 1a 2009 addenda), Part I and Part II, Subpart 2.7 (as applicable). To achieve compliance with the quality requirements, the fuel fabricator shall utilize trained or qualified personnel (as required), controlled processes, and measurement techniques that are traceable to recognized industry standards.

Where sampling and statistical procedures are used in place of 100% inspection in verifying compliance with acceptance criteria, all statistical procedures shall have a confidence level of 95% for the material characterizations, unless otherwise indicated in the tables of this specification, when testing compliance with specifications. Statistical sampling guidance for meeting the required confidence levels is available in PLN-4352, "Statistical Sampling Plan for AGR-5/6/7 Fuel Materials." Where acceptance criteria are given for both the mean and the distribution of an attribute, statistical procedures shall be applied separately for each criterion.

All data used to confirm compliance with this specification are assumed to be normally distributed. See the Statistical Methods Handbook for Advanced Gas Reactor Fuel Materials[3] for further discussion on this point. During qualification, statistical data used to confirm compliance with this specification shall be tested for normality. The results of normality testing shall be included in the qualification report to INL.

A shift toward a production approach and mindset is being made with the AGR-5/6/7 experiments in that no batch specifications are given except when the data cannot be obtained on composited lots. It is the responsibility of the fabricator to characterize fuel batches (kernel, TRISO particles, or compacts) sufficiently to ensure that the composited lots will comply with the fuel specifications.

Kernel, TRISO particle, and compact lots will be composed of multiple kernel, TRISO particle, or particle overcoating batches, respectively. Individual batches do not need to pass the lot specification limits, provided that the full off-specification batch is included and that the composited lot will pass the specification limits. Any blending of non-compliant batch material into a composited lot shall be done in accordance with an approved blend plan with the concurrence of INL. Inclusion of small quantities of

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material from off-specification batches into a lot (i.e., "salting") is prohibited to ensure that the composited lot is represented by a normally distributed population and to prevent having excessive material in population tails beyond critical limits.

Where batch-to-batch variability is much greater than the variability within a measured batch characteristic, such that a normally distributed lot population is not assured (with the limited number of batches in the lot), compliance with the lot specification can be demonstrated by verifying that each individual batch comprising the lot is compliant with the lot specification.

Some characterization methods employ very small sample sizes, such as for particle diameters, layer densities, and pyrocarbon diattenuation. In these cases, blended lots can be tested for compliance with the fuel specifications either by stratified sampling of the individual batches to ensure equal representation in the composited aliquot or pooling data from individual batches to form weighted population means and standard deviations.

The minimum precision/significant figures required to demonstrate compliance with specifications are indicated by the precision of the values in this specification. A population will be considered compliant if the analytical value for a property, when rounded to the indicated precision, is compliant.

## PART 3  LOW-ENRICHED URANIUM (LEU) FUEL SPECIFICATIONS

Product specifications are provided in this section for LEUCO kernels, TRISO coated particles, and fuel compacts. Chemical feedstock purity specifications are defined in SPC-1363.[7] The fuel fabricator is responsible for internal processes and procedures to fabricate the fuel to the product specifications.

### 3.01  LEUCO KERNELS

The composited kernel lot(s) shall consist of multiple kernel batches combined and thoroughly mixed to ensure uniformity prior to sampling for acceptance. The mass of a fuel kernel composite is not specified; however, a fuel kernel composite for use in AGR-5/6/7 is expected to contain at least 12 kg (net weight) of uranium carbide/oxide (UCO) kernels. The kernel composite lot specifications are given in Table 1.

Table 1. LEUCO kernel lot specifications.

| Kernel Lot Property | Mean[a] | Critical Region[a] | Critical Fraction |
|---|---|---|---|
| Diameter ($\mu$m) | $425 \pm 10$ | <375<br>>475 | $\leq 0.01$<br>$\leq 0.01$ |
| Envelope density (g/cm$^3$) [b] | $\geq 10.4$ | Not specified | Not specified |
| Uranium fraction (gU/gUCO) | $\geq 0.885$ | Not specified | Not specified |
| $^{235}$U enrichment (g$^{235}$U/gU) | $0.155 \pm 0.001$ | Not specified | Not specified |
| Carbon/uranium (atomic ratio) | $0.40 \pm 0.10$ | Not specified | Not specified |
| Oxygen/uranium (atomic ratio) | $1.50 \pm 0.20$ | Not specified | Not specified |
| (Carbon + oxygen)/uranium (atomic ratio) | $\leq 2.0$ | Not specified | Not specified |

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Table 1. (continued).

| Kernel Lot Property | Mean[a] | Critical Region[a] | Critical Fraction |
|---|---|---|---|
| Individual impurities (ppmw): <br> Li, Na, Al, Cl, Ca, V, Cr, Mn, Fe [c], Co, Ni, Cu, and Zn | ≤100 each | Not specified | Not specified |
| Process impurities (ppmw): <br> P, S | ≤1,500 each | Not specified | Not specified |
| Aspect ratio | Not specified | ≥1.05 | ≤0.10 |
| **Measurement Only** | | | |
| Microstructure [d] | | | |

(a). Specified mean values and fraction in critical regions determined at the 95% confidence level. The ± values represent an allowable range for the mean value and are not standard deviations of the mean.

(b). May be computed from pooled batch data.

(c). Iron contamination in natural acid-deficient uranyl nitrate (ADUN) solution, batch J52K-NU-01403, was reduced during kernel fabrication (kernel batch J52K-NU-59502 R) from 344 ppm Fe/ gU in the ADUN to 85 ppm Fe/ gU in the sintered kernel batch; a reduction factor of 4.

(d). Samples must show evidence of oxidic and carbidic phase interspersion within the kernels (Figure 1).

Figure 1 is a visual standard for a commonly accepted interspersion of oxidic and carbidic phases within a fuel kernel. Note the gray, oxidic "rind" on the perimeter of the kernel sections and the white carbidic phase comingled with an oxidic phase in the core of the kernel. While the kernel population with the finely divided carbidic phase is the more desirable of the two populations shown in Figure 1, both show acceptable evidence of oxidic and carbidic phases being present in the kernels.
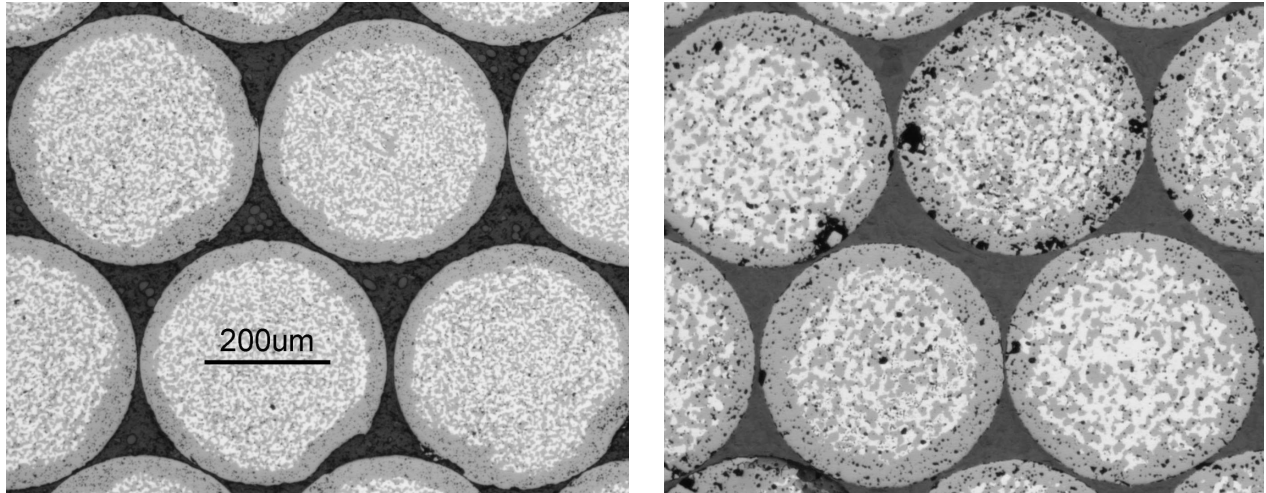


Figure 1. Visual standard of interspersed oxidic and carbidic phases.

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## 3.02  TRISO-COATED PARTICLES

The BWX Technologies (BWXT) 6-inch diameter coater uses a nominal UCO kernel charge of 2.0 kg; producing approximately 4.5 million TRISO particles per batch. Although a single coater batch will produce more than enough particles for all test compacts (including archival and analytical samples), use of fuel produced by a single run is not representative of industrial processes where fuel will be manufactured from composited lots. AGR-5/6/7 is to demonstrate fuel fabricated from a composited TRISO particle lot.

### 3.02.01  TRISO COATING PROCESS AND UPGRADING SPECIFICATIONS

All four coatings shall be applied in an uninterrupted coating run to LEUCO kernels to be used for AGR-5/6/7 experiment irradiations. Coating process conditions are specified in Table 2, because the product specifications in Section 3.02.02  may not be sufficient to guarantee product performance. Process specifications in Table 2 are point estimates and not tested to 95% confidence.

Table 2. LEU TRISO particle process specifications.

| Parameter | Buffer [f] | Inner Pyrocarbon (IPyC) | Silicon Carbide (SiC) | Outer Pyrocarbon (OPyC) |
|---|---|---|---|---|
| Argon to hydrogen ratio[a] | — | — | 30:70 | — |
| Control temperature (TC-4)[b] | 1,420°C ± 20°C | 1,310°C ± 10°C | 1565°C ± 30°C | 1,350°C ± 15°C |
| Coating gas fraction (CGF)[c] | 0.60 ± 0.06 | 0.30 ± 0.03 | 0.030 ± 0.005 | 0.32 ± 0.03 |
| Acetylene gas fraction[d] | 1.0 | 0.54 ± 0.02 | — | 0.54 ± 0.02 |
| Coating rate, μm/min[e] | ~20 | ~3.0 | ~0.38 | ~2.7 |

(a)  Nominal ratio on a coating gas (MTS)-free basis
(b)  Temperatures shown are defined as the average temperature during the coating deposition with the exception of the buffer layer. Buffer temperature is defined as the average temperature in the minute prior to initiating acetylene flow.
(c)  Average coating gas fraction for 6-inch diameter coater distributor defined as the ratio of coating gas flow to total gas flow.
(d)  Average acetylene gas fraction (ignoring gas stabilizers) divided by the total coating gas flow (acetylene + propylene).
(e)  Coating rate is required to be reported. Values shown are guidelines.
(f)  Buffer coated fuel kernels should be fluidized for over 20 minutes following buffer deposition to effect some densification/polishing of the buffer surface and minimize the potential for a strong bond between the buffer and IPyC layers. Post-irradiation examination of fuel from the AGR-1 experiment has implicated the strength of the buffer-IPyC bond as a possible contributor to tears in the IPyC, separation of the IPyC and SiC layers, and occasional damage/tearing of the SiC layer. A weak bond between the buffer and IPyC layers prevents stress accumulation in the IPyC resulting from buffer shrinkage and non-uniform debonding. Although the minimum time needed to adequately polish the buffer layer is not known for the 6-inch TRISO coater, it is believed that the time required to cool the bed of buffered particles to the IPyC deposition temperature and to draw a buffer hot sample (totaling 25–30 minutes) is adequate.

TRISO particles shall be size classified by sieving to remove undersized particles (particles with missing, broken, or thin layers) and oversized particles (particles with abnormally thick layers and accretion debris). The fuel fabricator shall select appropriate sieve sizes to achieve the desired separation. Use of an inclined, vibrating table process to sort aspherical and broken particles from the TRISO product is discretionary. A requirement to employ tabling for upgrading TRISO was suspended for the AGR-5/6/7 experiments because it was not shown to be effective in reducing defect levels and the operation resulted in a substantial rejection of compliant product.

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AGR-1 and AGR-2 irradiation experiments have shown that a low-strength bond between the buffer and IPyC layers is desirable as it promotes debonding when the buffer shrinks during irradiation and reduces mechanical strain that could induce cracking or tearing of the IPyC layer. Two indicators of low bond strength are 1) striated growth bands in the buffer layer with low density regions near the IPyC interface and 2) IPyC fragments that readily separate from the buffer layer when preparing a sample for IPyC density analysis. The length of the time interval that the bed fluidizes between the end of buffer deposition and the start of IPyC deposition may also influence bond strength, but insufficient data exist to establish a minimum time interval for any specific coater or kernel charge. Approximately $30 \pm 5$ minutes appears adequate in the 6-inch coater being used for the AGR fuel with a kernel charge $\leq 2.0$kg.

### 3.02.02  LEU TRISO PARTICLE SPECIFICATIONS

Table 3. LEU TRISO particle specifications.

| TRISO Particle Property | Mean[a] | Critical Region | Critical Fraction | Notes |
|---|---|---|---|---|
| **Lot Variable Properties** | | | | |
| Buffer density (g/cm$^3$) | $1.05 \pm 0.10$ | Not specified | Not specified | Point estimate, b |
| IPyC density (g/cm$^3$) | $1.90 \pm 0.05$ | $\leq 1.80$<br>$\geq 2.00$ | $\leq 0.01$<br>$\leq 0.01$ | b, c |
| Buffer thickness (μm) | $100 \pm 15$ | $\leq 58$ | $\leq 0.01$ | d |
| IPyC thickness (μm) | $40 \pm 4$ | $\leq 30$<br>$\geq 52$ | $\leq 0.01$<br>$\leq 0.01$ | d |
| SiC thickness (μm) | $35 \pm 3$ | $\leq 28$ | $\leq 0.01$ | d |
| OPyC thickness (μm) | $40 \pm 4$ | $\leq 20$ | $\leq 0.01$ | d |
| SiC density (g/cm$^3$) | $\geq 3.19$ | $\leq 3.17$ | $\leq 0.01$ | c |
| OPyC density (g/cm$^3$) | $1.90 \pm 0.05$ | $\leq 1.80$<br>$\geq 2.00$ | $\leq 0.01$<br>$\leq 0.01$ | c |
| IPyC diattenuation | $\leq 0.0170$ | $\geq 0.0242$ | $\leq 0.01$ | e |
| OPyC diattenuation | $\leq 0.0122$ | $\geq 0.0242$ | $\leq 0.01$ | e |
| SiC aspect ratio (faceting) | Not specified | $\geq 1.14$ | $\leq 0.01$ | f |
| **Lot Attribute Properties** | | | | |
| Defective IPyC coating fraction | $\leq 1.0 \times 10^{-4}$ | Not specified | Not specified | g |
| Defective OPyC defect fraction | $\leq 3.0 \times 10^{-4}$ | Not specified | Not specified | h |
| **Lot Measurement Only** | | | | |
| Pre-burn exposed uranium fraction (kernel equiv./particle count) | — Not specified — | | | i |
| Post-burn exposed uranium fraction (kernel equiv./particle count) | — Not specified — | | | j |
| SiC microstructure | Grains size < visual standard | | | k |
| SiC soot inclusions | — Not specified — | | | l |
| Abnormally shaped particles | — Not specified — | | | m |

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Table 3. (continued).

| Pyrocarbon surface connected porosity (ml/m²) | — Not specified — | |
|---|---|---|

(a)  The ± values represent an allowable range for the mean value and are not standard deviations of the mean.

(b)  Calculated from pooled batch data.

(c)  Specified values for density correspond to those measured by the sink/float method. If another method is used, a correlation to sink/float densities must be developed.

(d)  Metric determined using the ORNL "LAYERS" program/method.[10, 11, 12]

(e)  Diattenuation can be converted to an equivalent polarimeter $BAF_o$ by the formula: $BAF_o = 1.010 * (1 + N) / (1 - N)$. Using either stratified sampling of batches or pooled coater batch data to demonstrate compliance is recommended.

(f)  Aspect ratio is defined as the ratio of maximum to minimum diameters of the coated particle and is to be measured on SiC-coated particles following burn back of the OPyC layer.

(g)  Uranium dispersion, determined by x-ray analysis is used to quantify IPyC defects in a particle population that has been heat treated to 1800°C for ≥1 hr. The visual standard for acceptable and unacceptable fuel dispersion is given in Figure 3. This analysis was historically performed on TRISO liberated from compacts by deconsolidation without additional heat treatment.

(h)  Nondestructive visual examination for a broken or missing OPyC layer (exposed SiC layer).

(i)  This property is measured by the pre-burn leach in a leach-burn-leach sequence with measured kernel equivalents rounded to the nearest integer on individual samples.

(j)  This property is measured by the post-burn leach in a leach-burn-leach sequence with measured kernel equivalents rounded to the nearest integer on individual samples.

(k)  Specification will be met if the average SiC grain size is judged smaller than the average grain size shown in the visual standards (Figure 2). Using stratified sampling of batches or pooled coater batch data to demonstrate compliance is recommended.

(l)  Particles shall be examined for soot inclusions between the IPyC and SiC layers using mounted and sectioned particles.

(m)  Particles with a single, aspherical (partial) kernel forming "dimpled" particles (i.e., having a concave surface) and particles with compound kernels and kernel fragments forming capsular, triangular, or other complex shapes. Particles with a single, aspherical kernel should be counted separately from those having compound kernels. Refer to Figures 4 and 5.
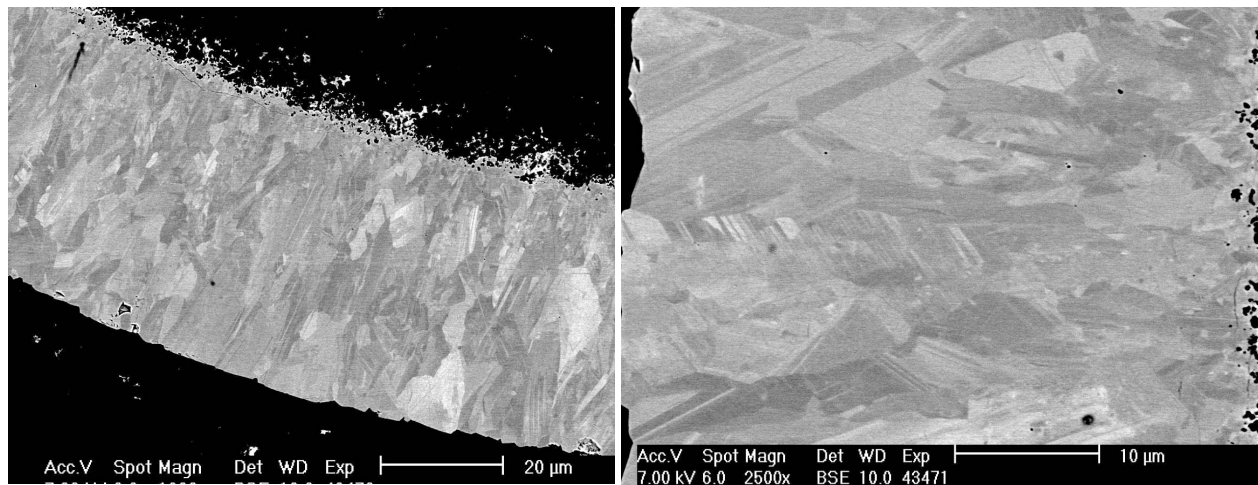


Figure 2. SiC with excessively large grain sizes.

Figure 3 has examples of TRISO particle X-ray radiographs showing an acceptable particle (a) and two others (b, c) showing evidence that the IPyC layers were defective. Figure 3(b) shows relatively minor impoundment of uranium at the buffer-IPyC interface whereas Figure 3(c) shows extensive

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uranium dispersion throughout the buffer. Defective IPyC layers allow infiltration of coating gases, specifically chlorine compounds, into the buffer and cause a chemical attack on the kernel. Uranium and uranyl chlorides volatilize at high temperature and migrate into the buffer layer. The IPyC defect manifests readily after heat treating the particles for 1 hour at 1800°C; as is done during compact heat treatment.
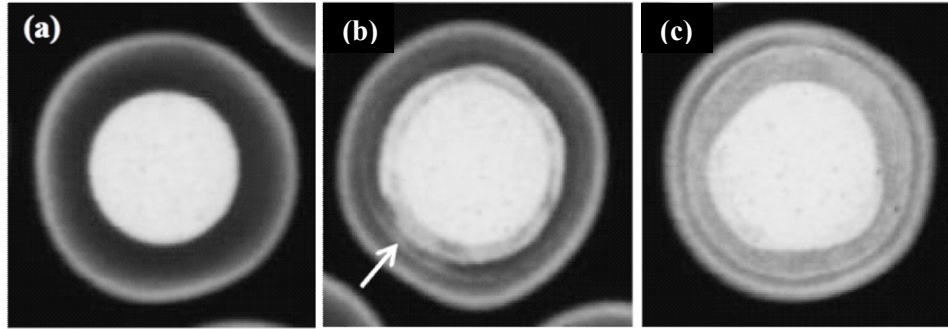


Figure 3. Visual IPyC defect/ uranium dispersion standards for acceptable dispersion (a) and unacceptable dispersion (b and c).

Soot inclusions at the IPyC – SiC interface may compromise the strength and in-pile performance of the SiC, leading to elevated release of fission products out of the TRISO particle. Soot inclusion data are obtained by visual examination of mounted and sectioned particles. An alternative method of using low-resolution x-ray images, as used for uranium dispersion, to identify deformed SiC layers may be used for information only. Significant inclusions manifest as unusually thin/ low-density SiC layers and aspherical SiC shells (i.e., shells with an underlying protuberance).

Abnormally shaped particles often are the result of frangible kernels. Kernels with missing sectors or surface spalling form dimpled TRISO coated particles (Figure 4). Kernel fragments can fuse with whole kernels to produce compound kernels that coat to form particles with capsular or more complex shapes (Figure 5).



Figure 4. Visual examples of dimpled particles.

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Figure 5. Visual example of TRISO-coated particles with compound kernels.

## 3.03  MATRIX SPECIFICATIONS

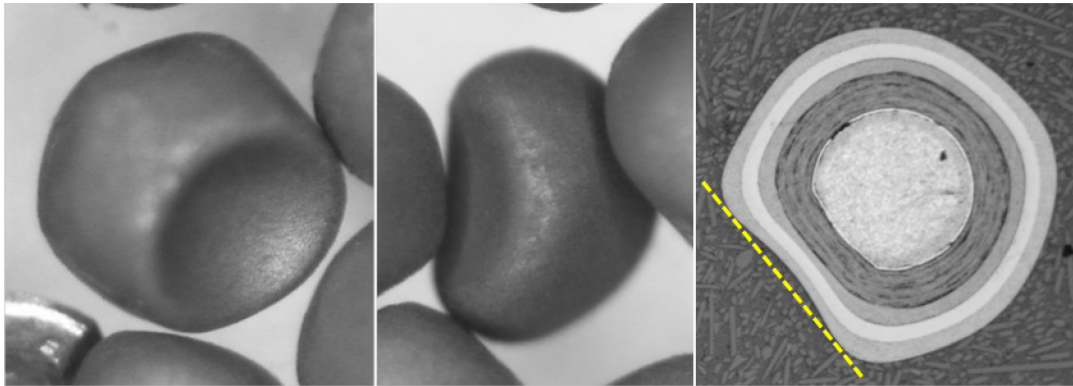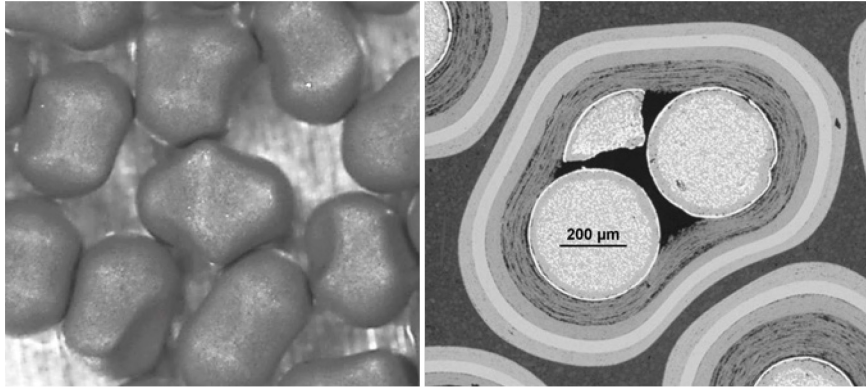The compact matrix shall consist of the following materials and nominal weight fractions:

| | |
|---|---|
| Asbury Carbons No. 3482 natural flake graphite | 64 wt% |
| SGL Carbon No. KRB2000 synthetic graphite | 16 wt% |
| Durite D_SD-1708 novolac phenolic resin | 19 wt% |
| Hexamethylenetetramine (HMTA) | 1 wt% |

The blended powders are to be jet-milled to a final average particle size ($d_p$) in the range: $7~\mu m \leq d_p \leq 15~\mu m$.

The resin shall be kept below 35°C during milling and storage of the resultant resinated graphite matrix product.

## 3.04  LEU COMPACT PRODUCT SPECIFICATIONS

UCO fuel compacts for AGR-5/6/7 irradiation experiments shall meet the specifications in Table 4. The AGR-5/6/7 experiments will have five capsules; each containing multiple fuel stacks of compacts for a total compact count approaching 200 compacts in the test train. Approximately 400 compacts should be shipped to INL for the AGR-5/6/7 experiments to allow for spares and archival samples.

To ensure gravimetric and dimensional stability of the matrix within the compacts, the compacts shall be heat treated, following resin carbonization, for at least 1 hour at a furnace temperature between 1650°C and 1850°C.

The heavy-metal contamination fraction (HMCF)[1,2,8,9] was bifurcated into a dispersed uranium fraction (DUF) and the exposed kernel fraction (EKF) for the first time for the AGR-5/6/7 experiments. The DUF is a metric of uranium that is present outside of a fission gas retentive layer; primarily as contamination on or within the OPyC layer. Surface uranium contamination is quantified in the pre-burn DUF and interior contamination is quantified by the post-burn DUF. The EKF is a metric of particles that have sustained mechanical damage after coating and have substantially cracked or broken coating layers. The DUF is quantified by computing the mean quantity of leachable uranium (from pre- and post-burn leaches), relative to the total amount of uranium in each deconsolidated compact clutch, that exhibited no evidence of exposed kernels (i.e., «1 kernel equivalent recovered per clutch). The EKF is quantified using pre-burn uranium leach data on clutches of deconsolidated compacts in excess of the pre-burn DUF.

The SiC defect fraction is determined from leachable quantities of uranium in the post-burn leaches that exhibit approximately one or more kernel uranium equivalents in excess of the post-burn DUF. A

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detailed description of the method for quantifying the EKF, DUF, and SiC defect fraction is given in Addendum 5.01.

Table 4. UCO heat-treated compact specifications.

| Property | Mean[a] | Critical Limits | Critical Fraction | Notes |
|---|---|---|---|---|
| Variable Properties | | | | |
| Mean uranium loading (gU/compact)<br>    Nominally 40% packing fraction<br>    Nominally 25% packing fraction | $1.36 \pm 0.10$<br>$0.90 \pm 0.08$ | Not specified | Not specified | b |
| Diameter (mm) | Not specified | $\leq 12.20$<br>$\geq 12.44$ | 0<br>0 | c, d |
| Length (mm) | Not specified | $\leq 24.40$<br>$\geq 25.30$ | 0<br>0 | c, d |
| Matrix density (g/cm$^3$) | $\geq 1.65$ | Not specified | Not specified | |
| Iron<br>($\mu$g Fe outside of SiC per compact) | $\leq 25$ | $\geq 100$ | $\leq 0.01$ | |
| Transition metals ($\mu$g)<br>Cr, Mn, Co, and Ni outside SiC per compact | $\leq 50$ each | $\geq 200$ total | $\leq 0.01$ | |
| Calcium ($\mu$g)<br> Ca outside SiC per compact | $\leq 50$ | Not specified | Not specified | |
| Aluminum<br>($\mu$g Al outside SiC per compact) | $\leq 50$ | Not specified | Not specified | |
| Titanium + Vanadium<br>($\mu$g (Ti + V) outside SiC per compact) | $\leq 240$ | Not specified | Not specified | |
| Dispersed uranium fraction (DUF)<br>(g·$U_{leached}$/g·$U_{sample}$) | $\leq 1.0 \times 10^{-5}$ | Not specified | Not specified | e |
| Attribute Properties | | | | |
| Exposed kernel fraction (EKF)<br>(kernel equiv./particle count) | $\leq 5.0 \times 10^{-5}$ | Not specified | Not specified | f |
| Defective SiC coating fraction<br>(kernel equiv./particle count) | $\leq 1.0 \times 10^{-4}$ | Not specified | Not specified | g |
| Defective OPyC coating fraction | $\leq 0.01$ | Not specified | Not specified | h |

(a)   The ± values represent an allowable range for the mean value and are not standard deviations of the mean.

(b)   The actual packing fraction is a function of the TRISO particle and compact dimensions. The values given for compact packing fractions are not specifications, but for convenience in differentiating among the compact lots.

(c)   VHTR compact dimensions are expected to have nominal compact lengths of 0.97 in. (half-height) or 1.94 in. (full-height) and a nominal diameter of 0.49 in. (12.45 mm) with critical limits of 0.487 in. and 0.497 in. (12.37 and 12.62 mm). The AGR experiments use a smaller compact diameter because of geometrical constraints in test train design.

(d)   The compact diameter shall be measured near the top, middle and bottom of each compact. Orthogonal replicate measurements of the diameter are not required. A single length measurement near the axis of each compact will suffice.

(e)   Dispersed uranium is quantified by pooling pre-burn and post-burn leach data where less than 0.5 kernel equivalents were recovered during the leaches.

(f)   The exposed kernel fraction is the quantity of uranium detected in the pre-burn leach of deconsolidated compacts (decremented by the value of the average dispersed uranium from the pre-burn leach) and converted to the nearest integer value of kernel equivalents.

(g)   Leachable uranium not associated with the DUF or EKF. Determined by the quantity of uranium leached during the post-burn leach (decremented by the value of the average dispersed uranium from the post-burn leach) and expressed as

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Table 4. (continued).

| | kernel equivalents rounded to the nearest integer on individual samples. |
|---|---|
| (h) | For defective OPyC coating fraction, count as defects any particles with exposed SiC due to inadequate OPyC deposition or spalled OPyC layers. This attribute is quantified using particles collected following compact deconsolidation. |

## PART 4  METHODS ADDENDUM

### 4.01  COMPACT DEFECT ANALYSIS

The heavy-metal contamination fraction (HMCF) for compacts is subdivided into three fractions: an exposed kernel fraction (EKF), a dispersed uranium fraction (DUF), and a SiC defect fraction. The EKF represents uranium in kernels that are exposed to acid leachant because of traumatic damage to the coating layers (i.e., broken or cracked layers). The DUF represents dispersed uranium present outside of the fission gas retentive layers and is attributed to uranium smudge picked up on the surface of the SiC, during OPyC deposition, and on the outer surface during subsequent fluidization and handling. The SiC defect is associated with defective SiC as it is manifested after the carbonaceous TRISO layers are removed by controlled oxidation in a furnace.

Even though the layers are compromised on the damaged particles, the release of radiocesium is far less than for dispersed uranium under normal operating conditions. It is estimated that dispersed uranium releases 5–10 times more radiocesium than a comparable amount of uranium from an exposed kernel.

The method of analyzing compact clutches (≤5 compacts/clutch) by deconsolidation-leach-burn-leach enables the various defect fractions to be quantified.

Pooled variances in the properties of fuel kernels (density, wt% U, diameter, etc.) from AGR-2 fuel kernel data give a standard deviation on the uranium content of a fuel kernel of approximately10%. Any deconsolidated and leached compact clutches having, in aggregate, ≤0.5 kernel equivalents of uranium in the pre-burn and post-burn leachates are considered to be free of exposed kernels and the source of uranium is treated as dispersed. Clutches with ≥0.5 kernel equivalents of uranium are considered to contain damaged particles with exposed kernels (pre-burn) or defective SiC see Figure 6.

The DUF is a variable property. The data from all deconsolidated compact clutches having no apparent damaged particles are pooled to calculate an estimated mean, standard deviation, and 95% confidence level for the DUF. It is further assumed that all compact clutches, including those with exposed kernels, have DUFs represented by the estimated mean.

$\overline{U}_k = average\ gU/kernel$

$$\mathrm{DUF} = \frac{gU\ leached}{gU_{clutch}} \approx \frac{gU\ leached/\overline{U}_k}{clutch\ TRISO\ count}$$

$$\overline{DUF}_{pre-burn} = \sum_{i=1}^{n} DUF_{pre-burn_i}/n$$

$$\overline{DUF}_{post-burn} = \sum_{i=1}^{n} DUF_{post-burn_i}/n$$

Where:

n = number of clutches

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The EKF and SiC defect fraction are attribute properties that are computed from the total uranium leached in the pre-burn and post-burn leaches, respectively, decremented by the associated DUF, and converted into the equivalent leached kernel fraction.

$$Pre\text{-}burn\ kernel\ equivalents = \frac{gU\ leached\ in\ pre\text{-}burn\ leaches}{\bar{U}_k}$$

$$Post\text{-}burn\ kernel\ equivalents = \frac{gU\ leached\ in\ post\text{-}burn\ leaches}{\bar{U}_k}$$

$$Pre\text{-}burn\ exposed\ kernel\ count$$
$$= Round\left(\sum_{i=1}^{n} Pre\text{-}burn\ kernel\ equivalents_i - \frac{n \cdot \overline{DUF}_{pre-burn}}{\bar{U}_k} \cdot g\bar{U}_{clutch}\right)$$

$$Post\text{-}burn\ leached\ kernel\ count$$
$$= Round\left(\sum_{i=1}^{n} Post\text{-}burn\ kernel\ equivalents_i - \frac{n \cdot \overline{DUF}_{post-burn}}{\bar{U}_k} \cdot g\bar{U}_{clutch}\right)$$

$$EKF_{Point\ Est.} = \frac{Pre\text{-}burn\ exposed\ kernel\ count}{\sum_{i=1}^{n} clutch\ TRISO\ count_i}$$

$$SiC\ Defect_{Point\ Est.} = \frac{Post\text{-}burn\ leached\ kernel\ count}{\sum_{i=1}^{n} clutch\ TRISO\ count_i}$$

$$\overline{DUF}_{Point\ Est.} = \overline{DUF}_{pre-burn} + \overline{DUF}_{pre-burn}$$

The TRISO particle batch/lot is to be analyzed for exposed uranium prior to use in forming compacts to provide a measure of confidence that the resulting compacts will likely pass these three defect specifications. This is accomplished with a leach-burn-leach analysis of the TRISO. It is recognized that the pre-burn leach does not recover all of the dispersed uranium inside the OPyC during the pre-burn leach and that the dispersed uranium cannot be reasonably segregated from the point-source uranium associated with either exposed kernels or defective SiC. Nonetheless, the pre-burn exposed uranium fraction is an estimate of the compact exposed kernel fraction and the post-burn exposed uranium fraction is an estimate of the compact SiC defect fraction. The relationship between TRISO and compact defect fractions is shown in Figure 7.

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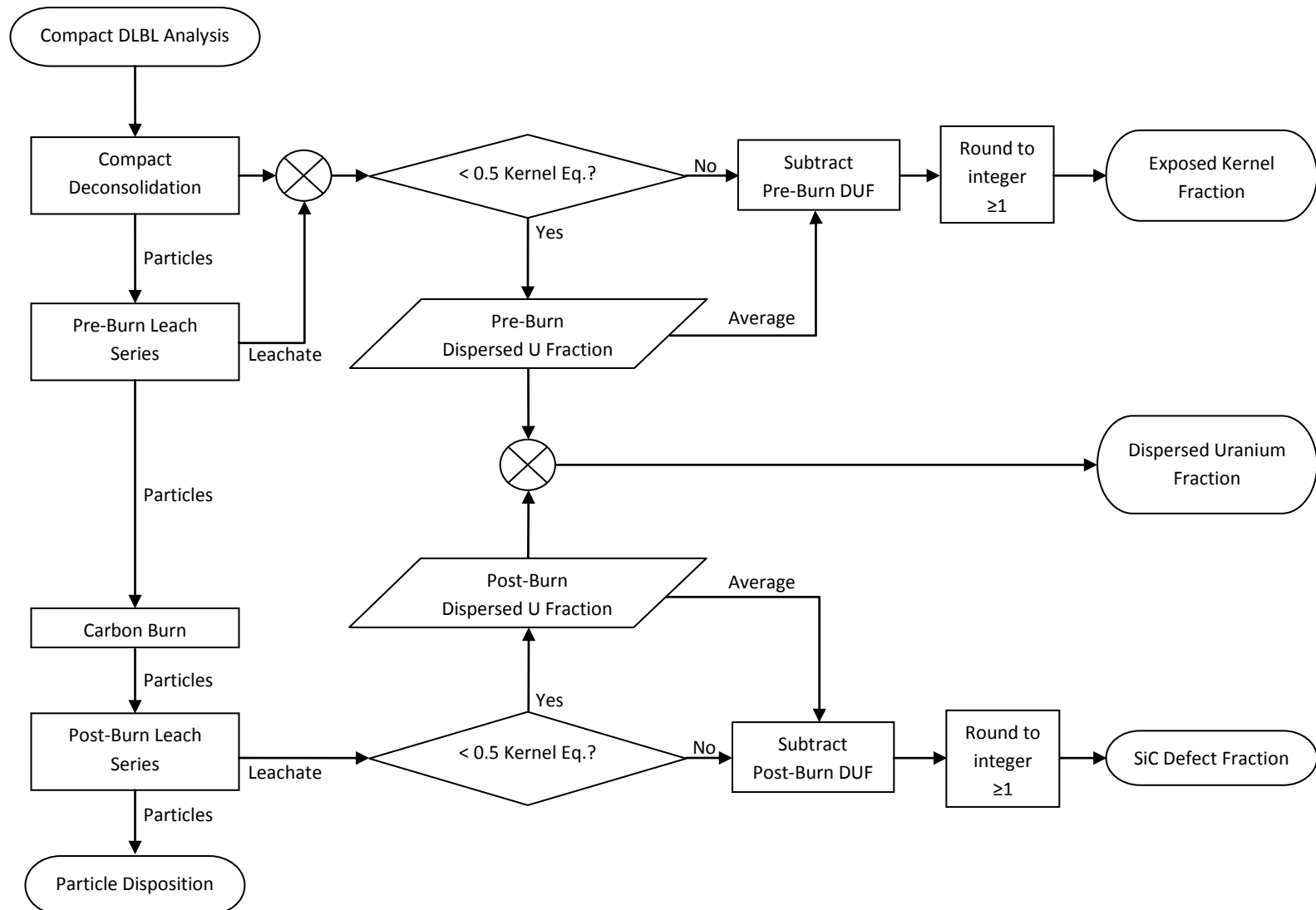
Figure 6. Compact defect fraction analysis.

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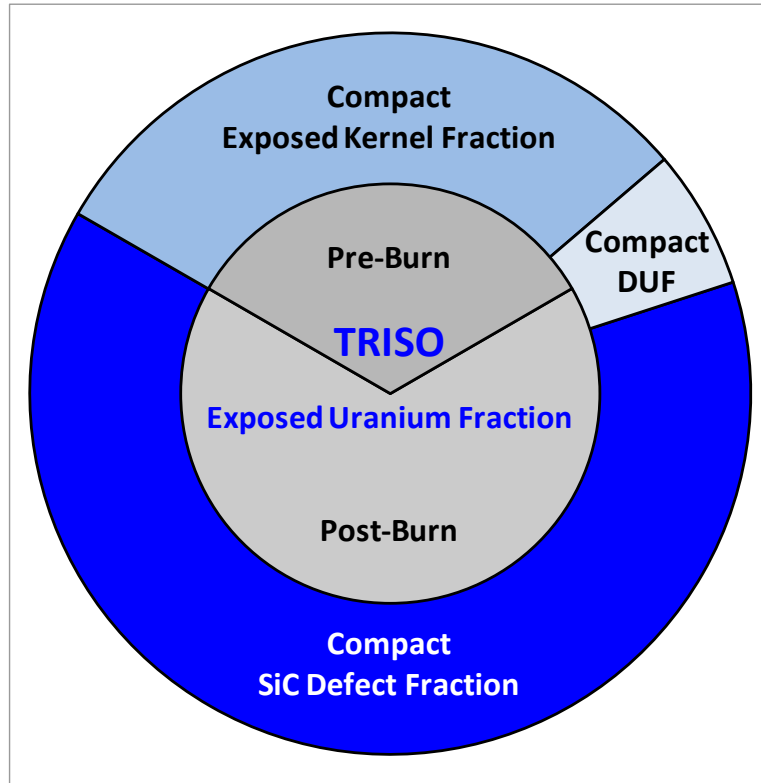
Figure 7. TRISO and compact defect fractions.

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## 4.02  METHOD FOR MATRIX DENSITY DETERMINATION

The method for estimating the matrix density of a compact shall be done using the equations below. Mean kernel and TRISO lot properties will need to be tracked for this calculation. Use the individual mass, TRISO particle count, and physical dimensions of each compact used for this determination and not average compact batch values.

$$\rho_m = \frac{M_C - M_{TRISO}}{V_C - V_{TRISO}}$$

$$M_{TRISO} = C_{TRISO} \cdot AWP_{TRISO}$$

$$V_C = \frac{\pi}{4} \bar{d}_C^2 \cdot l_C$$

$$V_{TRISO} = C_{TRISO} \cdot \frac{\pi}{6} \bar{d}_{TRISO}^3$$

$$C_{TRISO} = ROUND\left(\frac{U_C}{U_k}\right)$$

$$\rho_m = \frac{M_C - C_{TRISO} \cdot AWP_{TRISO}}{\frac{\pi}{4} \bar{d}_C^2 \cdot l_C - C_{TRISO} \cdot \frac{\pi}{6} \bar{d}_{TRISO}^3}$$

If the compact packing fraction is known, then the matrix density can be determined by the following relationship:

$$\rho_m = \frac{\rho_c - \bar{\rho}_p \cdot PF}{(1 - PF)}$$

where:

| | |
|---|---|
| $AWP_{TRISO}$ | average weight (mass) of a TRISO particle |
| $C_{TRISO}$ | compact TRISO particle count |
| $d_C$ | compact average diameter |
| $d_{TRISO}$ | TRISO particle diameter |
| $l_C$ | compact average length |
| $M_C$ | compact mass |
| $M_{TRISO}$ | mass of all TRISO particles in the compact |
| $PF$ | volumetric packing fraction of TRISO coated particles in the compact |
| $U_C$ | compact uranium loading |
| $U_k$ | kernel-equivalent uranium |
| $V_C$ | compact volume |
| $V_{TRISO}$ | volume of all TRISO particles in the compact |
| $\rho_c$ | compact envelope density |
| $\rho_m$ | matrix density |
| $\rho_p$ | TRISO coated particle density |

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