Fission product distribution in irradiated safety-tested and non-safety-tested AGR-2 TRISO particles

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Abstract. Two similarly-irradiated TRISO nuclear fuel particles, one safety-tested and one not-safety-tested, were examined by electron probe microanalysis (EPMA) as part of the advanced gas reactor (AGR) fuel development and qualification programme. The primary objective of this study was to determine the effect of high temperature safety testing on the fission product quantification and distribution. Concentration profiles for fission products were collected along two radii in each particle. Fission product mass balance distribution for both particles was determined by computing the material mass per traverse step-size and multiplying by the weight fraction analysis at that position. Results of these analyses suggest that the non-safety-tested particle contains more fission product mass in the centre 300 µm of the kernel than the safety-tested particle; however, for mobile fission products such as Cs, Xe, Te, Sr, Ba, and Pd, the mass moves out further from the particle centre in the safety-tested particle than the non-safety-tested particle. This mass balance approach to TRISO particle analysis is beneficial at elucidating fission product migration in irradiated and safety-tested particles and will be beneficial in applying to other particles to determine potential irradiation damage effects on the fission product distribution and mobility.

1. Introduction

The AGR-2 experiment is the second of several fuel irradiation experiments planned for the advanced gas reactor (AGR) fuel development and qualification programme. The purpose of the AGR-2 experiment is to build on lessons learned during the preceding AGR-1 experiment. In contrast to the AGR-1 experiment, which used laboratory-scale fuel fabrication equipment and methods, the AGR-2 experiment used prototype industrial-scale equipment and methods for fuel fabrication.

The AGR-2 UCO tristructural isotropic (TRISO)-coated fuel particle consists of a UCO fuel kernel 425 µm in diameter, surrounded by concentric layers of confining materials (Fig. 1). These include a 100 µm thick porous graphite buffer, followed by a 40 µm thick dense graphite inner pyrolytic carbon layer (IPyC). Outside the IPyC is a 35 µm thick SiC layer. Finally, a 40 µm thick dense graphite outer pyrolytic carbon layer (OPyC) overlies the SiC layer [1]. At time of fabrication, the average kernel composition is UC0.392O1.428. The outer portion of the kernel differs from the interior of the kernel in that the kernel periphery (KP) is characterised compositionally by the rapid increase in C concentration and the concomitant rapid decrease in U concentration.

Fission product (FP) distribution in irradiated nuclear fuels has been studied by electron probe microanalysis (EPMA) for several decades. The use of EPMA is advantageous for several reasons. Firstly, the energy resolution of EPMA is typically on the order of 10 eV, compared to ~125 eV for the
modern silicon drift detectors used in scanning electron microscopes [2]. Secondly, the EPMAs used for irradiated specimens are equipped with W-alloy shielding to: 1) reduce detector background noise; 2) reduce the signal to background noise ratio changes that occur when sample-to-detector distance is altered during stage movement; and, 3) preserve the detector life. Finally, the high currents (200 – 300 nA) employed by EPMA in the course of analysing irradiated specimens can improve detection limits by a factor of 10 or more [3].

Numerous earlier EPMA efforts examined FP distribution [4-6], precipitate composition [7], and Cs buffer speciation [8-9]. One previous study examined FP distribution in AGR1 fuel particles [10]. This study expands on these latter efforts by estimating FP product mass in each particle layer.

1.1. Objectives
Several objectives will be achieved by this study. Firstly, the quantitative FP distribution of both irradiated and irradiated- and- safety-tested particles will be characterised. These data are required for modelling fuel behaviour under irradiation conditions, and to date, no such data exists for AGR-2 fuel. Secondly, this study provides the opportunity to separate the effects of irradiation only on FP distribution from those effects resulting from safety-testing similarly irradiated particles. This will lend insight to high temperature effects on FP mobility. Finally, this study demonstrates the utility of using EPMA for such studies. Because of the use of calibration standards and the availability of sophisticated peak deconvolution software, this instrument is essential for quantitative irradiated fuel chemical analysis for the fully coated particle in a time-efficient manner.

2. Methods

2.1. Irradiation and safety testing
TRISO particles in Compacts 2-2-3 and 2-2-2 were fabricated using prototypic-scale equipment and were subjected to irradiation as described by Collin [11]. Table 1 shows the irradiation parameters for both compacts and safety testing parameters for Compact 2-2-2. Following irradiation, Compact 2-2-2 was safety tested using the Oak Ridge National Lab Core Conduction Cooldown Test Facility, which was used to evaluate the release of FPs upon heating. Morris et al. [12] discuss the procedures and equipment for this process. Following the safety test, Compact 2-2-2 was subjected to a leach-burn-leach test. Details of the test can be found in Demkowicz [13]. Following the
leach-burn-leach test, both safety-tested and non-safety-tested particles were subjected to gamma counting to determine how the actual FP inventory of each particle compared to the predicted FP inventory. At the conclusion of the gamma counting, one particle each was selected from Compacts 2-2-3 and 2-2-2 for this electron probe microanalysis (EPMA) study. Each particle has exhibited some loss of FPs as evidenced by a measured to calculated $^{110}\text{mAg}$ ratio of $<1$ (Table 1).

**Table 1.** Shows the irradiation and safety-testing parameters for the two examined AGR-2 particles.

| Parameter                          | AGR2-223-RS34 | AGR2-222-RS19 |
|------------------------------------|---------------|---------------|
| $^{235}\text{U}$ enrichment        | 14.03         | 14.03         |
| % FIMA average burnup              | 10.8          | 12.55         |
| Time-average, volume average temp. | 1161          | 1287          |
| Approximate fast fluence ($\times 10^{25}$, n/m$^2$) | 2.99          | 3.39          |
| Measured to calculated $^{110}\text{mAg}$ ratio | 0.84          | 0.2*          |
| 1,600 °C safety test (hours)       | * measured after safety test | 300 |

2.2. Sample preparation

Each particle was mounted into a metallography mount with Buehler EpoHeat epoxy. The mounts were then polished to mid-plane thickness and $<1$ µm finish using standard methods. Each mount was then coated with 15 nm of aluminium to ensure sample conductivity. Images of the specimens are shown in Figure 1.

2.3. Electron probe microanalysis

Electron probe microanalysis was performed at Idaho National Laboratory’s Irradiated Materials Characterization Facility using a CAMECA SX100R electron probe microanalyser, which is specifically designed for the analysis of highly radioactive specimens. To that end, the instrument’s electronics and detectors are shielded to 3 Ci of $^{137}\text{Cs}$.

Various peak and background counting times were employed depending on the phase type and element concentration. In all cases, the accelerating voltage was 20 kV and the beam size was just focussed. The current employed for each element was 200 nA, except for Si in the SiC layer, which was measured using a current of 20 nA to avoid excessive detector dead time.

The choice of X-ray lines, standards, diffracting crystals, and corrected interferences is shown in Table 2. For some elements, an acceptable standard was not available. In this situation, a “virtual” standard was employed. The intensity for a virtual standard is determined by measuring the intensities of the same X-ray lines on the same crystal and spectrometer for elements that are adjacent on the periodic table to the X-ray line of interest. An X-ray intensity can then be interpolated for the line of interest.

Each X-ray line was measured using a peak and two backgrounds. The count time for each X-ray peak varied with the abundance and fluorescence yield of the element, but in general each peak was counted for 300 to 500 seconds, with a background count time approximately one-third of the peak time. For elements with higher count rates such as U in the kernel and Si in the SiC layer, the count rate was 20 - 30 seconds for the peak and 10 - 15 seconds for each background. Note that standards were not counted in the same manner as the sample points; the concentration of the element in the standard is generally so much higher than it is in the unknown that it is not necessary to count for more than 30 seconds for the peak and 10 seconds for the background. Measuring the standards for the shorter time does not negatively affect quantification. In fact, if standards were measured at the same beam currents and for the same time period as unknowns, there would likely be excessive detector counter
deadtime, which itself would negatively influence quantification. Finally, some standards, such as phosphates, are beam sensitive and are measured using a defocused 20 µm beam. All standards were measured using a 20 nA current, and the dead time for all analyses was set to 3 µs.

Table 2. Shows the X-ray line, standard used, diffracting crystal, and corrected interferences for each measured element.

| Element | X-ray line | Standard | Diffracting crystal | Interferences corrected |
|---------|------------|----------|---------------------|------------------------|
| U       | Mα         | U        | QTZ                 | Pd, Ru                 |
| Zr      | Lα         | Zr       | PET                 |                         |
| La      | Lα         | LaP₂O₅₁₄  | LiF                 | Cs                     |
| Ce      | Lα         | CeP₂O₅₁₄  | LiF                 | Ba                     |
| Nd      | Lα         | NdP₂O₅₁₄  | LiF                 |                         |
| Pr      | Lα         | PrP₂O₅₁₄  | LiF                 | La                     |
| Sm      | Lα         | SmP₂O₅₁₄  | LiF                 | Ce                     |
| Eu      | Lα         | EuP₂O₅₁₄  | LiF                 | Pr, Nd                 |
| Ba      | Lα         | barite    | QTZ                 | Pr                     |
| Cs      | Lα         | pollucite | PET                 | U, Ce, Te              |
| Xe      | Lα         | virtual   | PET                 | Te, La                 |
| Mo      | Lα         | Mo       | PET                 |                         |
| Ru      | Lα         | Ru       | PET                 | Ba                     |
| Ag      | Lα         | Ag₂Te    | QTZ                 | Cd, U                  |
| Pd      | Lα         | Pd       | QTZ                 | Ru, U                  |
| Te      | Lα         | Ag₂Te    | PET                 |                         |
| I       | Lα         | TlBrI    | PET                 | Cd, Ba                 |
| Cd      | Lα         | Cd       | QTZ                 |                         |
| C       | Ka         | SiC      | PC1                 | U                      |
| O       | Ka         | ThO₂     | PC1                 |                         |
| Si      | Ka         | SiC      | TAP                 | U, Pu                  |
| Sr      | Lα         | SrTiO₅   | TAP                 | Si                     |

Quantitative radial traverse data was collected using PROBE FOR EPMA software. The traverse paths measured on each particle are shown in Figure 1. For each particle, one radius was measured in a region in which there was a gap between the buffer and IPyC, and a second radius was measured in a region that lacked a gap, or the gap was small. The distance between points varied from 2 - 5 µm depending on the region of the particle being measured. All X-ray collection programs used the Pouchou and Pichoir (PAP) matrix correction algorithm [14] and the mass-absorption coefficients used were those of Farthing and Walker [15].

2.4. Fission product mass balance calculations

The first approach to evaluating differences between safety-tested and non-safety-tested particles was to estimate the mass of select FPs in the central kernel. This was accomplished by applying the method described in Section 2.5 to the first 150 µm of each radius (from the particle centre extending outward 150 µm. This provides an estimate of 300 µm of the approximate 425 µm kernel diameter. The 300 µm distance was chosen because that portion of the kernel is more homogeneous (based on qualitative micro-porosity distribution) than portions beyond 300 µm. Thus, FP distribution homogeneity in this region is an assumption for this study. The method described in section 2.5 was then applied to the entirety of both radii.
2.5. Calculation method
Quantitative weight percent EPMA data from the radial traverse measurements in each particle was converted to an elemental mass in each particle, subdivided according to the particle layer (i.e., kernel, buffer, IPyC, etc.). The procedure and assumptions employed were as follows:
1. The weight percent of the measured element at a given location is assumed to be homogeneous for a slice of the particle equivalent to a hemispheric slice of the measured traverse step-size width.
2. The volume of the hemisphere slice of the measured traverse step-size width is calculated in cubic micrometres.
3. The material density is known (the buffer densifies during irradiation and is assumed to double for this calculation).
4. The mass of the hemispheric slice is computed from volume and density.
5. The weight percent measured at that point is changed to weight fraction and is multiplied by the mass of the hemisphere slice to arrive at a mass in grams of the element, per one hemispheric slice.
6. The procedure is repeated for each hemisphere slice for the first measured radial traverse.
7. The procedure is then repeated for the other radial traverse to account for the inhomogeneity that results from the existence of thermal gradients across the particle.
8. The total mass of the element in the particle is calculated by adding all the slices from both hemispheres together.

3. Results

3.1. Morphology
Both studied particles are characterised by a nearly complete delamination of the buffer from the IPyC. In addition, the KP region in both particles exhibits variable thickness, ranging from approximately 30 - 100 µm thick. Finally, the KP is quite distinct from the kernel central region in that the KP is characterised by a large density of small-diameter pores (~ < 1 µm in diameter), in contrast with the centre of the kernel, which is typified by fewer, much larger pores (~1 - 50 µm in diameter).

3.2. Fission product distribution
3.2.1. Cs, Xe, I. Figures 2a and 2b show the Cs compositional profiles for both particles. Several observations can be made. Firstly, for both particles, the compositional profile on the gap side is similar to that of the non-gap side. Both show elevated elemental concentrations in the buffer and IPyC as compared to the kernel. For both particles, the elemental concentration begins to rise in the kernel perimeter. In addition, the elemental concentration profile is continuous as it crosses the gap suggesting that the gap formed after the element had migrated from the buffer to the IPyC.

Figure 2. Quantitative radial traverses for Cs for both examined particles. The non-gap side of the particle is shown in blue while the gap side is shown in orange.
There are differences in Cs concentration profiles between safety-tested and non-safety-tested particles. The peak Cs concentration in the buffer of safety-tested particle is on the order of 50 - 100 % greater than in non-safety-tested particle (AGR2-222-RS19 versus AGR2-223-RS34). In addition, elemental kernel concentrations are generally greater in the non-safety-tested particles compared to that of safety-tested particles. Xe and I profiles are similar to Cs (data not shown).

3.2.2. Sr, Ba, Te, Eu. Figures 3a and 3b show the Sr profile for both studied particles. In contrast to Cs, Xe, and I, the Sr concentration profile is asymmetrical across particle AGR2-222-RS19. Specifically, Sr is approximately four times greater in concentration in the KP on the gap side of the particle as compared to the non-gap side. For both particles, the elemental concentration increases in concentration in the KP and reaches its maximum near the KP-buffer boundary. Concentrations then decline precipitously in the buffer. Contrasting the safety-tested particle, concentration profiles on particle AGR2-223-RS34 are more symmetrically distributed. Ba, Te, and Eu profiles are similar to Sr (data not shown).

3.2.3. SiC-IPyC interface. The SiC-IPyC interface for both particles is shown in Figs. 4a to 4d. With both particles, the Pd and Ag concentration is higher on the non-gap side than the side with the gap. Both particles also show Pd penetration through most of the SiC layer, but little Ag penetration. This may be because the Ag has not penetrated into the SiC layer of the particles or may be because the quantity in the SiC layer is below the detection limit. Although the Ag concentration at the interface is similar for both particles, the Pd concentration is approximately three times higher in the safety-tested particle than the non-safety-tested particle.

3.2.4. Fission product mass balance calculations. The results of the FP mass balance calculation described in section 2.5 for the inner 300 µm kernel are shown in Table 3. The estimated FP mass per particle is shown in Table 4. For Cs, Xe, Te, Ba, Mo, Ru, and Pd, the non-safety-tested particle, RS34 contained a greater FP mass in the inner most 300 µm of the kernel than did the safety-tested particle, RS19. In contrast, the estimated FP mass in the whole particle is greater for particle RS19 than it is for RS34 for all tested elements except Pd and Ru.
Figure 4. a-b) Pd concentration profile, and c-d) profile of the Ag concentration, across the IPyC-SiC boundary. The gap side of the particle is indicated by the orange symbol and the non-gap side is indicated by the blue symbol.

Table 3. Estimate of the mass of select FPs mass (in gram) located in the inner 300 µm of the kernel diametral sphere. The larger mass is in bold font.

| Particle | Cs     | Xe     | Sr     | Te     | Ba     | Mo     | Nd     | Ru     | Pd     |
|----------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| RS34     | 5.1E-08| 1.9E-08| 6.9E-08| 1.8E-07| 9.6E-08| 1.7E-06| 1.5E-06| 8.7E-07| 1.1E-07|
| RS19     | 3.0E-08| 7.3E-09| 2.1E-07| 1.4E-07| 8.7E-08| 1.3E-06| 2.0E-06| 4.6E-07| 5.5E-08|

Table 4. Estimate of the mass of select FPs mass (in gram) present in each particle. The larger mass is in bold font.

| Particle | Cs     | Xe     | Sr     | Te     | Ba     | Mo     | Nd     | Ru     | Pd     |
|----------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| RS34     | 2.7E-06| 3.4E-07| 1.5E-06| 1.1E-06| 2.2E-06| 4.6E-06| 5.0E-06| 3.1E-06| 6.1E-06|
| RS19     | 3.3E-06| 1.1E-06| 2.0E-06| 2.4E-06| 3.7E-06| 5.5E-06| 7.0E-06| 3.0E-06| 1.2E-06|

4. Discussion

4.1. Particle morphology and fission product distribution

For both particles AGR2-222-RS19 and AGR2-223-RS34, two radii were examined, one on the side of the particle where the buffer remained bonded to the IPyC, and one on the side where the buffer became delaminated from the IPyC, creating a substantial gap between the buffer and IPyC. This morphology is quite common among irradiated AGR1 particles (AGR1 particles preceded the AGR2 particles in the AGR sets of experiments) and is thought to be the result of the inward buffer densification [16]. Assuming that this hypothesis is correct (as opposed to the gap having formed during sample preparation), the FP distribution evidence suggests that the gap formed after Cs, I and Xe had migrated into the IPyC, or that the gap did not alter the transport properties of these elements. Figure 2 shows that Cs has crossed the gap and appears to maintain the same concentration profile as the other side of the particle that has no gap.

While most FPs have similar concentration profiles on both sides of the particle (gap side versus non-gap side), the exceptions include Sr, Te, Ba, and to a lesser extent, Eu. Sr, Ba and Te concentrations are up to four times higher in the KP of the non-gap side of safety-tested particle than on the side with the gap (Figures 3a and 3b). However, this difference cannot be due to the gap as the gap is beyond the
buffer, outside of this concentration spike. For non-safety-tested particle AGR2-223-RS34, the concentration asymmetry is not present; however, this asymmetry has been observed in other non-safety-tested particles (data not shown). Perhaps concentration gradient differences based on incipient asymmetry are enhanced through the safety-testing process.

Several researchers describe the effects of temperature gradients across TRISO particles. Tiegs [5] and Minato et al. [7] both describe the transport of Pd and rare earth elements down the temperature gradient. Grubmeier et al. [17] noted the diffusion of rare earth and platinum group elements to the cooler side of the particle but stated that they were not necessarily co-located. Kleykamp [4] described the presence of an “azimuthal temperature gradient” across the particle that influenced FP transport and precipitation.

Figures 4a to 4d show that on the non-gap side of both particles, Pd and Ag are on the order of 3 – 4 times higher in concentration than it is on the gap side of the particle. Figure 1 (a-b, boxes 1 and 2) shows visible precipitates at the SiC-IPyC interface, whereas no such obvious precipitates are present at the SiC-IPyC interface where the buffer has been separated from the IPyC. Given that the precipitates are located where the buffer-IPyC remains intact, this suggests that this is the cooler side of the particle and that delamination occurs preferentially where the particle is hotter.

Kleykamp [4] showed that for TRISO with UO₂ kernels, Te and Pd migrated to the cold side of the particle. As Te, Sr, Ba, and Eu all exhibit similar behaviour, and Sr, Ba, and Eu are predicted to exist as carbides [18], this suggests that transport properties for these species are similar and that they accumulate on the colder side of the particle.

4.2. Fission product distribution in safety-tested versus non-safety-tested particles

Section 3 provided data concerning differing concentration gradients in the safety-tested versus non-safety-tested particles as a two-dimensional representation of the FP behaviour as a concentration traverse across a plane. By employing the method describe in Section 2.5, it is possible to approximate FP masses in different particle layers across three dimensions, thus arriving both an estimated FP inventory for the whole particle, and the mass inventory for each particle layer. This allows a more thorough comparison of FP migration between the two particle treatments.

Tables 3 and 4 show that although AGR2-223-RS34 has a higher FP mass for Cs, Xe, Ba, Te, Mo, Ru, Pd in the 300 µm kernel centre, AGR2-222-RS19 has overall higher masses of Cs, Xe, Sr, Te, Ba, Mo, and Nd in the whole particle. However, the distribution of some of these elements amongst the particle layers is different in the high-temperature safety-tested particle compared to the non-safety-tested particle.

Figures 5a and 5c show a larger quantity of Cs and Xe in the KP, buffer, and IPyC of AGR2-222-RS19 compared to what is contained in these layers in AGR2-223-RS34 (Figs. 5b and 5d), while AGR2-223-RS34 contains more Cs and Xe in the inner kernel than what is observed in AGR2-222-RS19.

Figures 6a and 6b show that for Sr, most of the mass is located in the KP for both particles. Figures 7a and 7b show distinctively different behaviours between the safety-tested particle and the non-safety-tested particle. In the safety-tested particle, Nd is most abundant in the KP, while in the non-safety-tested particle, most of the mass remains in the inner kernel. Particle AGR2-222-RS19 experienced slightly higher burn up, so it would have produced more nuclide mass, but the data in Fig. 7 suggests that for this element, the safety-test promotes diffusion from the kernel to the more peripheral KP region.

Similarly, Figs. 8a and 8b show that Pd behaves quite differently in the safety-tested particle in that much of the mass has moved to the IPyC and SiC, whereas in particle AGR2-223-RS34, most Pd resides in the KP. Palladium vapour pressure increases from 0.006 Pa at 1,161 °C (the approximate time average volume average irradiation temperature) to 7.4 Pa at 1,600 °C, the temperature of the safety test. This increased vapour pressure will likely result in greater diffusion, thus explaining this observation.
Figure 5. Cs and Xe total elemental mass estimate (in gram) and distribution within particles AGR2-222-RS19 and AGR2-223-RS34.

Figure 6. Sr total elemental mass estimate (in gram) and distribution within particles AGR2-222-RS19 and AGR2-223-RS34.

Figure 7. Nd total elemental mass estimate (in gram) and distribution within particles AGR2-222-RS19 and AGR2-223-RS34.
4.3. Sample size
Due to the high cost of analysing radioactive samples, only two radii on one plane were measured. Observations and resulting conclusions are based on the data obtained from these radial traverses and the assumption of hemispheric homogeneity. However, these particles could be much more heterogeneous than assumed, which could alter the data and subsequent conclusions.

5. Conclusions
- Sr, Te, Ba, and Eu are up to four times higher in concentration on the non-gap side of safety-tested particle AGR2-222-RS19 compared to the gap side. Asymmetry is absent in the non-safety-tested particle AGR2-223-RS34.
- Pd and Ag are up to four times higher in concentration on the non-gap side of the SiC-IPyC boundary of both the safety-tested and non-safety-tested particles, suggesting that the gap forms on the hotter side of the particle.
- Although the safety-tested particle contains a greater mass of FPs Cs, Xe, Sr, Te, Ba, Mo, and Nd than the non-safety-tested particle, the non-safety-tested particle retains more FP mass of Cs, Xe, Ba, Te, Mo, Ru, and Pd in the central kernel compared to the safety-tested particle.
- The FP transport as determined by three-dimensional mass calculations shows that for mobile FPs such as Cs, Xe, Te, Sr, Ba, and Pd, the mass moves out further from the particle centre in the safety-tested particle than the non-safety-tested particle. Palladium is much more abundant in the IPyC and SiC layers of the safety-tested particle than the non-safety-tested particle.
- Computing the FP mass by determining the electron interaction volume and extrapolating the measured concentration layer by layer appears to offer a method to more carefully explore FP transport and mass balance.
- It will be beneficial to re-evaluate this method using additional radial line scan data and mass balance calculations to better evaluate sample heterogeneity along the central plane. In addition, it would be beneficial to compare these results with results obtained from FP production modelling (e.g., ORIGEN) for each particle to evaluate how this FP mass as estimated by EPMA measurements compares to that calculated by the FP model.

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