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A logical approach for zero-rupture Fully Ceramic Microencapsulated (FCM) fuels via pressure-assisted sintering route

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ABSTRACT

A pathway to Fully Ceramic Microencapsulated (FCM™) fuel pellets showing absence of sintering-derived "rupture" has been demonstrated. In the typical FCM manufacturing process, TRistructral ISOtropic (TRISO) particles show statistically significant rupture, caused by contact of particles during the axial shrinkage of fuel pellet that accompanies pressure-assisted sintering. To solve this, template SiC powder discs were fabricated to host planes of TRISO particles, and the disks were stacked to form a cylindrical "green" pellet. After sintering, up to ~34% packing fraction of particles (Vp) was physically feasible without contact between planes. Sintering was shown to reduce the axial displacement between planes of TRISO particles, and X-ray Computed Tomography (XCT) showed planes separated by a displacement of ~100 μm. XCT, optical microscopy and SEM showed the very limited radial displacement of particles. However, the relative density of the FCM pellet was limited to ~95%. The current results support the zero-rupture concept as viable, but perturbations to TRISO arrangements and limited matrix density require further investigation to improve FCM fuel uniformity and microstructure.

1. Introduction

Fully Ceramic Microencapsulated (FCM™) nuclear fuels consist of TRistructural ISOtropic fuel particles embedded in a dense ceramic matrix. A TRISO particle contains a fissionable microsphere (e.g. UO_2) coated to form concentric layers of buffer pyrolytic carbon (PyC), inner PyC (IPyC), SiC, and outer PyC (OPyC). The SiC layer functions as the pressure vessel and the primary fission product (FP) release barrier. Consolidation of TRISO within a fully-dense SiC matrix provides an additional FP release barrier, as well as other diverse benefits. Proposed as a transformational technology for fission reactors [1,2], the SiC matrix is sintered using a small amount of alumina and yttria as sintering additives [3]. This type of SiC matrix is often classified as a nano-infiltration and transient eutectic-phase (NITE) SiC, a special category of processing routes for SiC. The NITE SiC matrix is able to form a radiation-resistant form of SiC of a near-theoretical density at relatively low applied pressures and temperatures. Such mild sintering conditions prevents deformation to the SiC layer and minimizes consumption of OPyC. TRISO fission product retention is typically considered viable to at least 1800 °C [4,5] and successful irradiations of the NITE SiC matrix and its variants have been conducted [6,7]. Therefore, FCM is proposed as a robust form of Accident Tolerant Fuel (ATF) fuel for a Light Water Reactor (LWR) core and other advanced reactor systems [8,9].

Fabrication of FCM has been conducted in laboratory-scale investigations, but the technology has not progressed to industrial deployment. Briefly, particles are overcoated with NITE SiC mixture, and uniaxially pressed into a "green" pellet. This green pellet is thus a random packing of TRISO particles. During sintering, uniaxial shrinkage occurs, resulting in both reduced displacement between particles and re-arrangement of the particles. Prior work cited volume fractions of particles (here after V_p) as ~30% [16], 18–41% [7], 20–24%, and 37% [17,18]. The absence of rupture is reported in selected fuel pellets. However, TRISO particles have shown to be ruptured [7,10–13] after sintering, and thus require additional down-selection. The morphologies are not widely disseminated [11–13] but released data consistently shows fragments of two or more particles in the sintered microstructure. It is hypothesized that re-arrangement and convergence of particles from the original particle positions leads to "particle-particle" interactions [7], hereby referred to as "rupture". As Vp increases, the

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starting displacement between particles decreases, and so particles begin in closer proximity in the greenbody. Consequently, an increase in rupture probability would be expected from both shrinkage and re-arrangement.

FCM is fundamentally flawed if there are ruptured particles – the goal of FCM is obviously to maintain the TRISO particle layers, so the SiC matrix can act as an additional FP barrier and thermal conductivity medium. Maximizing particle density without rupture and mitigating the physical contact are key requirements. The displacement between particles must be controlled to prevent physical contact. A proposed "zero-rupture" fuel design leads to both maximized and consistent TRISO packing, and the industrial scalability of FCM fuel. The approach hypothesizes that eliminating the radial convergence is needed to prevent rupture. The manuscript describes how planes of particles with a fixed in-plane arrangement are stacked with an interplanar SiC matrix. The fuel pellet is successfully sintered, reveals consistent displacement between planes, and thus a pathway to eliminate rupture.

2. Experimental work

High-purity (>99%) SiC with a d_{50} size ~80 nm (Nanomakers, France, Lot# M15_99_035_009) was blended with oxides (US Research Nanomaterials Inc.) with respective d_{50} size of ~80 nm for Al_2O_3 (US3008) and ~45 nm for Y_2O_3 (US3551). The mass ratio was $94SiC\text{-}3.9Al_2O_3\text{-}2.1Y_2O_3$, hereby referred to as NITE SiC. The powder was formed into green bodies, which were templates for both trigonal and random-in-plane packing. Fig. 1(a) shows the template for a close-packing arrangement, where each TRISO particle is hosted by a cavity in the "green" pellet (unfired ceramic). The repeating sequence is either ABAB or ABCA (HCP or FCC stacking). A top and bottom NITE SiC disk completes the green fuel pellet. In Fig. 1(b) the template is random-in-plane ($A_1A_2A_3$). The repeating unit for random in-plane requires a cavity and lid, is substantially simpler in execution, but has a lower theoretical Vp.

Fig. 1 shows the 2D cross-section of the disks. When stacked in a graphite die, these form a cylindrical green-body pellet ready for sintering. In early studies, both ZrO_2 and HTGR-type TRISO were used. Ratios were maintained for LWR geometry; HTGR-type TRISO particles (~0.7 mm) had a nominal LWR geometry (12.7 mm), therefore ~1 mm ZrO_2 particles used 20 mm diameter pellets to preserve aspect ratios. The sintering parameters are described elsewhere [14]. After sintering, the cylinder was measured via dimensional inspection with digital micrometer (Model 293-185-30 IP65 QuantuMike Mitutoyo Corporation, Japan) and gas pycnometer (AccuPyc II 1340, Micromeritics Inc., Norcross, GA, USA). Bulk and Archimedes densities were respectively obtained from weight divided by the obtained volumes. MicroXCT™ (now Carl Zeiss X-ray Microscopy, Inc., CA, USA) was conducted under previously reported parameters [7]. The volume fraction of particles (Vp) was calculated from initial and final volume of particles (assuming no volume change) and the bulk volume of the pellet. Cut cross-sections were mounted and polished to 1 μm finish, and SEM (EVO MA15, Carl Zeiss Microscopy LLC, NY, USA) was conducted at the Joint Institute for Advanced Materials (JIAM).

3. Results

The early results show the simulation of close-packing and random-in-plane packing with particle fuels. The XCT scans in Fig. 2(a) show the progression of images beginning with the (i) top layer of the ZrO_2 particles (bright circles) in a NITE SiC matrix (dark contrast). 2D trigonal packing is demonstrated, although deviation in the pattern seen toward the rim. Fig. 2(a)(ii) shows an inset of the next layer super-imposed on (i) demonstrating the transition between planes. As (ii) layer emerges from the trigonal interstice of (i), it shows two planes with local tetrahedral configurations. A tetrahedron is the basic polygon of close-packing. This configuration has been successfully used to boost packing of ZrO_2 in surrogate inert matrix fuels [15].

Fig. 2(b) shows random-in-plane packing of ZrO_2 particles. Due to constraint of the perform, the packing density of "random-in-plane" is higher than random packing due to regions of trigonal packing. As a technology demonstrator, the random-in-plane concept was assembled and sintered with a designed ratio of ~34 vol% TRISO. Fig. 3(a) shows an XCT image of the cross-section and (b) shows a 3D reconstruction. TRISO positions can be determined by the bright contrast of ZrO_2 within the surrogate TRISO. The fuel kernels are typically half the diameter and a quarter of the total TRISO volume. Therefore, consistent and uniform spacing between TRISO planes is evident from the unoccupied region above and below each plane of particles, and obviously no ZrO_2 center exists within a "TRISO diameter" of another. Analysis shows the separation between adjacent planes (see Supplementary A1 for rotations). Therefore, current results support this technology as a pathway to sintering zero-rupture TRISO fuels.

Obviously, this does not exclude other damage (such as manufacturing defects, CVD SiC decomposition, etc) which affect fuel performance. Rather, this approach shows a method of FCM fabrication eliminating a major hurdle to industrial fabrication. The logic needs to be verified and validated by further work, but it is apparent this fabrication method will prevent touching particles before and after sintering. Fig. 3(a) highlights remaining challenges that currently limit the packing. These are respectively labelled A, B and C and are discussed later. Fig. 3(a) also shows Z-contrast in XCT images, indicating absence of material i.e. porosity of the NITE SiC phase (confirmed by Fig. 5) within each plane of particles. Previous FCM designs using random packing did not show inhomogeneity in

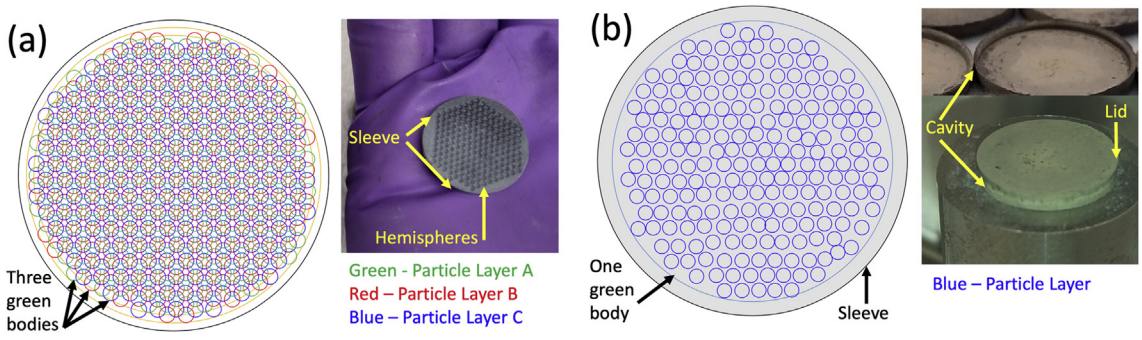

Fig. 1. Greenbody templates for the base repeating unit of **(a)** trigonal in-plane (for HCP/FCC) and **(b)** random in-plane arrangements.

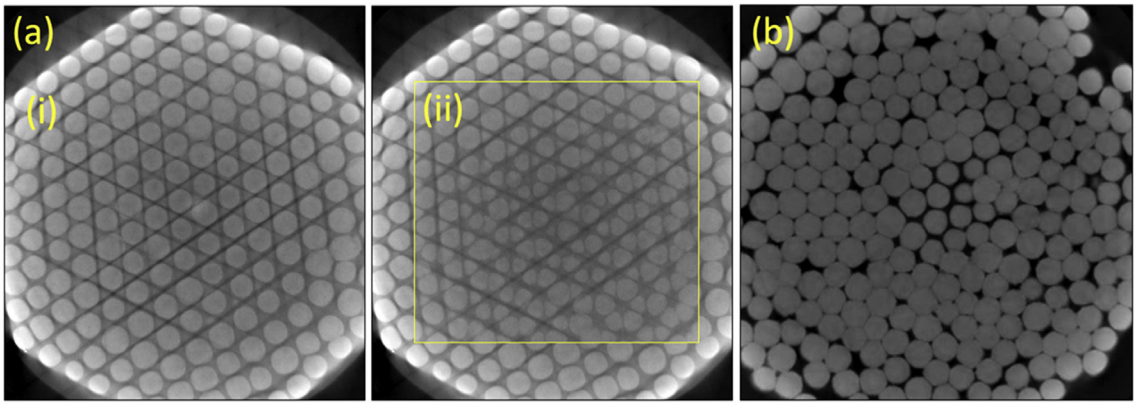

Fig. 2. XCT scans from the top of the pellet demonstrating ZrO_2 particles arranged in (a) (i) trigonal in-plane with close packing. The inset (ii) shows that the next layer emerges from the apparent trigonal interstice, indicative of tetrahedral arrangement and thus representing the ability to produce close-packing. In (b) random in-plane arrangement is demonstrated. Regions of trigonal packing are observed due to deformation during sintering.

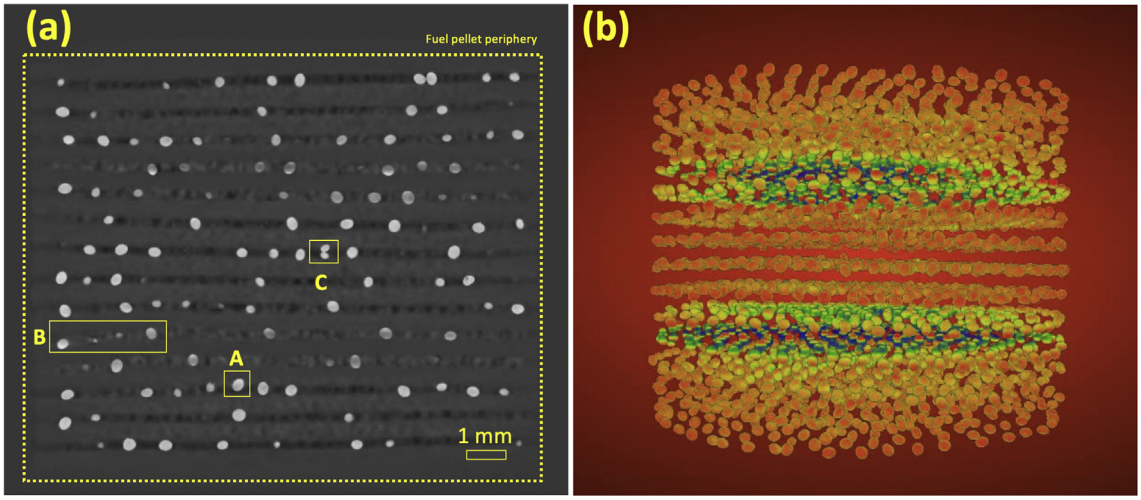

Fig. 3. An example of fuel pellets capable of up to ~34 vol% TRISO particles with (a) Spatial "xy" XCT from the radial edge demonstrating planes of particles (random-in-plane) in stacks without contact between planes. A, B and C represent phenomenon detrimental to reaching the packing limit of FCM fuels. A 3D XCT reconstruction of the fuel pellet (b) shows the spatial position of the surrogate ZrO_2 kernels (Supplementary A1 shows alternative rotations).

matrix density [10]. Therefore, despite identical matrix materials, it is apparent that the planar arrangement, a key feature of the zero-rupture technology, significantly alters the densification process of NITE SiC.

Fig. 4 shows the densification of SiC, FCM and the SiC component of the FCM. The dotted line is the heating profile as a function of time. The solid curve shows the density of the FCM. It increases from 1.84 g/cc to ~2.83 g/cc as sintering proceeds. The SiC densification is shown both without TRISO particles (i.e. a SiC cylinder) and with TRISO particles in an FCM. Without particles (dashed line), the NITE SiC pellet begins at ~1.65 g/cc, and as the temperature reaches 1825 °C, values of 3.05 g/cc at 5 min and ~3.16 g/cc at 10 min are seen, consistent with previous data [11,14] (Compiled data on NITE SiC parametrics are located in Supplementary A2).

Since the final density of the SiC matrix is known, the theoretical FCM density is about ~2.95 g/cc (from 34% Vp (at 2.64 g/cc) and assuming 66% volume SiC matrix). This indicates the FCM only achieved a relative density of 95%. This implies that arranged particles physically inhibit the densification. The dotted-dash line shows that when NITE SiC is sintered with TRISO particles in an FCM, the calculated matrix density both begins and ends at a lower value. The challenges of achieving dense matrix in this FCM design

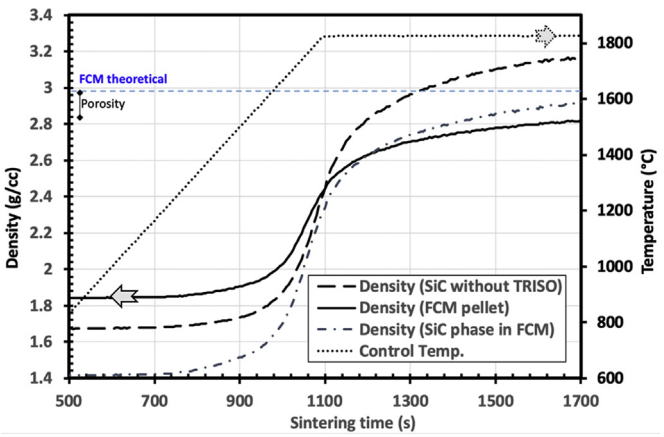

Fig. 4. Density as a function of sintering time to 1825 °C for up to 10 min. The FCM (NITE SiC matrix with TRISO particles), and a reference NITE SiC matrix densification permit the calculation of the matrix density when hosting TRISO particles. The matrix cannot deform and consolidate freely with abiding TRISO particles.

is expected because sintering involves both radial and axial shrinkage components. In previous FCM designs, the free flow of SiC matrix (and subsequent re-arrangement of particles) may have permitted higher matrix densities.

Both XCT data and the above assumptions suggested that the matrix between TRISO particles cannot densify. Cross-sections were cut to confirm particle distribution and interparticle porosity. Microscopy shows either porous matrix where particles touch, or absence of matrix between particles. The absence of matrix is logically explained by the absence of NITE SiC powder during forming. The consolidation mechanism of porous matrix particles was investigated in detail. Fig. 5(a) shows that above a particle there is a small periphery of porous matrix, and pore shapes are normal to the consolidation direction. Fig. 5(b) shows the SiC matrix between the particles. Randomly oriented pores between particles are observed. The matrix also appears to be less sintered than the regions above the TRISO or elsewhere. Fig. 5(c) and (d) show the porosity between particles (digitally removed), showing increasing porosity of the matrix in the interstice.

The matrix shrinkage is locally isotropic andboundaries are enforced by non-shrinking TRISO particles. Therefore, sintering results in porosity between in-plane particles as matrix densifies without boundary shrinkage. Logically, the maximum density of the matrix is only achieved if particles touch during green forming. In summary, the results show that packing of TRISO in SiC by planes of particles is a suitable countermeasure for sintering-derived rupture. Constraining the flow of SiC matrix with TRISO particles only permits axial shrinkage with severe radial constraints. This eliminates re-arrangement during sintering and inhibits matrix densification due to non-shrinking boundaries that cannot move.

4. Discussion

There are several near-term needs for the zero-rupture FCM design. First, improvement in the understanding of powder rheology and green forming is needed to improve deposition and compaction of matrix between particles. Currently, the properties of this type of FCM should be conservative. Fuel performance modeling should use the matrix values (e.g. Weibull strength or thermal conductivity) aligning with “lower density (0.86–0.90)” data per Snead et al. [7] The varying compaction of the preforms appear to affect the densification of SiC sintering. While sintering is macroscopically uniaxial due to confinement of the cylindrical die, the shrinkage of uniaxially pressed cylinders is initially isotropic [16]. Therefore, the particles exert constraint on radial shrinkage. The matrix between TRISO particles can be analogous to pressureless sintering of packed powder within a crucible (i.e. matrix densifies and shrinks from the inner interfaces of OPyC). The relatively random pore orientation in matrix between particles supports this theory. In contrast, flattened pores above particles show the effect of the applied pressure. As TRISO Vp increases, it is logical that a higher proportion of matrix exists in the gaps that have constrained boundaries. Without improvement in powder rheology and consistent particle contact during forming, the maximum attainable matrix density decreases as a function of Vp.

The pathways to FCM fabrication that potentially increase Vp can be understood on reflection of the historical development. The early attempts in 2011 adopted HGTR manufacturing, replacing carbon precursors with NITE SiC. It is theorized that since shrinkage during sintering is isotropic, any in-plane particles that were randomly packed by overcoating would have a radial component of

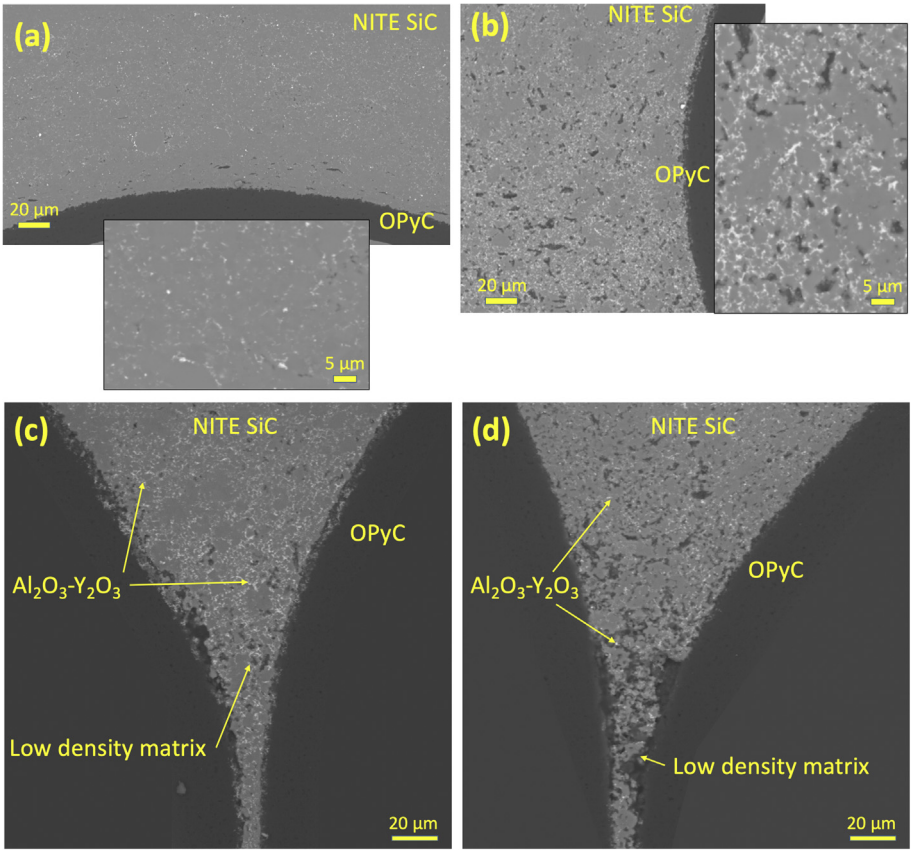

Fig. 5. High resolution SEM images of the NITE SiC matrix viewed near TRISO particles (a) above the plane, in the axial direction of the pellet and (b) in plane, in the radial direction of the pellet, and (c) and (d) shows the progression of desirable matrix infiltration into the cleavage between particles.

convergence. Sintering "makes room" in the particle plane due to shrinkage, and permits net migration of TRISO particles above and below. To complete sintering without mechanical contact, the particle must specifically travel along a vector normal to an interstice, or fill a TRISO "vacancy" in the next layer. The absence of radial shrinkage between particles thus prevents gross particle migration to the adjacent layer.

However, there are FCM fabrication methods that may be able to exclude sintering-derived rupture. First, in HTGR pellets or pebbles, presumably the modulus of the graphite precursor is low and consolidation occurs with minimal shrinkage [17]; for a metal ("M3") matrix, damage to particles was already reported [11]. SiC consolidated by sintering was not expected to be as forgiving as either of the above. However, it is possible that a suitable matrix or process parameters could provide the viscoelastic behavior to mitigate stresses on converging particles. Another suitable fabrication method is where unchanged arrangements of particles are observed. For example, this could be achieved if the greenbody density is relatively close to the sintered density [18]. Similiarly, matrix consolidation without shrinkage (e.g. CVI) cannot exhibit sintering-derived rupture.

An inverse relationship between particle size and pellet diameter dictates the obtainable Vp values. For either greenbody assembly, the relationship can be immediately assessed by the "areal density" of packed circles within a larger circle. The theoretical limits of random packing (~48%) [17], random with perturbation (rhombohedral or double-nested (~60–64%)) [19] and close-packing (~74%) are known to those familiar with the field. However, maximum Vp is reduced by the requirement of an unoccupied particle-free periphery and loss of packing density without infinite dimensions. In LWR pellet geometry, Terrani et al. calculated that the unoccupied pellet wall subtracts ~8% Vp [10]. Despite regions of close packing observed in the center of random-in-plane packing, the absence of infinite lattice explains why the packing limit is only about ~40 vol% . Therefore, only an incremental improvement in Vp is realized. The emphasis of this work is a methodology able to consistently achieve statistical predictability of rupture required for industrial fabrication. The previous approach likely exhibits an increasing continuum of fuel rupture as a function of Vp. In this new approach, no ruptures should exist below a threshold Vp. For example, this LWR technology demonstrator has a maximum ~40 vol% before particle layers touch. Above this value, "100% rupture" should occur.

Both XCT and microscopy reveal further engineering needed to reach the threshold Vp. The denoted A, B and C perturbations in Fig. 3(a) likely lead to TRISO mechanical interactions below the threshold Vp. A, B and C appear to be a mixture of random and systematic events. Type A appears to be a "random" out-of-plane migration event (see Supplementary A3), and may be caused by green body inhomogeneity. Type B appears to be systematic, and is hypothesized to be caused by green forming pressure distribution or inhomogenous densification of the periphery. Early investigations show that inhomogenous densification is caused by rapid heating [14]. The rapid heating is typical of Pulsed Electric Current Sintering, which makes a commercially viable mass-production technique possible [1] using parallel sintering, or serial "flash" sintering [20]. However, it relies on the resistivity of Joule heating of the graphite die and punch, which conduct current through the outside of the greenbody. Type B is labelled as "systematic" is because it shows adjacent planes maintaining separation, consistent with a temperature-dependent densification. Finally, Type C explicitly demonstrates the effect of asphericity in fabricated particle fuels, which reduce the predictability of displacement between particle planes. Therefore, a comprehensive understanding of A, B and C are required to approach threshold Vp limits, and progress to more demanding close-packing fuels.

5. Conclusions

A pathway to zero-rupture FCM fuel has been demonstrated. Preforms hosting a radially enforced plane of TRISO particles are stacked to form a cylindrical pellet and sintered. The shrinkage between planes of TRISO particles is limited by the mass of ceramic in the preform. A technology demonstrator for LWR fuel was synthesized, showing a volume packing fraction (Vp) of up to 34% in the bulk volume of the pellet. No particle-to-particle contact was observed in optical microscopy or XCT. The absence of sintering-derived rupture is attributed to restriction of the radial and axial shrinkage between TRISO planes. Maintaining the radial formation of the particles in-plane prevents gross particle migration theorized to cause TRISO rupture. Challenges remain in green forming and consolidation of the matrix between particles. The prior rupture problem may have been due to re-arrangment and convergence of particles; constraint from the planar arrangement now appears to inhibit densification of the matrix. Secondly, the demonstrator showed significant room for improved Vp due to non-uniform particle planes. This included "random" migration events, systematic perturbations during sintering, and feedstock quality of the particle fuel. These are significant hurdles to reaching the superlative packing fractions proposed in close-packed arrangements.

CRediT authorship contribution statement

Caen Ang: Writing - original draft, Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Writing - original draft, Writing - review & editing. **Lance Snead:** Formal analysis, Funding acquisition, Project administration, Resources, Validation, Writing - review & editing. **Yutai Kato:** Conceptualization, Formal analysis, Investigation, Methodology, Project administration, Supervision, Visualization, Writing - review & editing.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jnucmat.2020.151987.

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