Fracture Strength and Principal Stress Fields during Crush Testing of the SiC Layer in TRISO-Coated Fuel Particles

Brian C. Davis^{a,e}, Logan Ward^{b,e}, Darryl P. Butt^c, Brent Fillery^d, Ivar Reimanis^{b,e}

^aDepartment of Mechanical Engineering, Colorado School of Mines, 1500 Illinois St., Golden, CO 80401, USA

^bDepartment of Metallurgical and Materials Engineering, Colorado School of Mines, 1500 Illinois St., Golden, CO 80401, USA

^cMaterials and Science Engineering Department, Boise State University, 1910 University Blvd., Boise, ID 83725, USA

^dRolls-Royce Deutschland Ltd & Co KG, Eschenweg 11, Dahlewitz, 15827 Blankenfelde-Mahlow, Germany

^eColorado Center for Advanced Ceramics, Colorado School of Mines, 1500 Illinois St., Golden, CO 80401, USA

Abstract

Diametrical compression testing is an important technique to evaluate fracture properties of the SiC layer in TRISO-coated nuclear fuel particles. This study was conducted to expand the understanding and improve the methodology of the test. An analytic solution and multiple FEA models are used to determine the development of the principal stress fields in the SiC shell during a crush test. An ideal fracture condition where the diametrical compression test best mimics in-service internal pressurization conditions was discovered. For a small set of empirical data points, results from different analysis methodologies were input to an iterative Weibull equation set to determine characteristic strength (332.9 MPa) and Weibull modulus (3.80). These results correlate well with published research. It is shown that SiC shell asphericity is currently the limiting factor of greatest concern to obtaining repeatable results. Improvements to the FEA are the only apparent method for incorporating asphericity and improving accuracy.

© 2016 Published by Elsevier Ltd.

Keywords:

TRISO, SiC, strength, crush test, FEA, HTGR, FCM

1. Introduction

Tri-isotropic (TRISO) coated fuel particles are at the center of numerous nuclear energy designs and concepts, such as high temperature gas reactors (HTGRs) and fully ceramic microencapsulated (FCM) fuels [1, 2]. The SiC layer in the TRISO coating functions as a spherical pressure vessel, preventing unintended release of fissile materials. Its structural integrity is imperative to the overall safety rationale that supports the use of HTGRs, FCM fuel elements, and other designs which use TRISO coated fuel particles.

The SiC layer is applied using chemical vapor deposition (CVD). Its mechanical properties are dependent on the texture of the base onto which it is applied [3]. The SiC exhibits brittle fracture and the bulk of the fissile material is contained so long as no crack penetrates the full thickness of the shell. Thus, understanding principal stress fields and fracture strengths of this application-specific CVD SiC shell is important.

A typical modern TRISO particle (Figure 1) features a SiC shell which is $\sim 800\mu\text{m}$ in diameter and only $\sim 35\mu\text{m}$ thick, and

therefore mechanical testing is challenging.

Figure 1. A cutaway view of a TRISO-coated particle showing the overall size, options for core materials, and order of TRISO applied layers [4].

Numerous destructive test methods have explored the fracture of SiC isolated from the TRISO assembly [3, 5, 6]. Recent work has focused on diametrical compression of SiC hemispheres [7, 8]. A key limitation of this method is that fracture inducing stress is generated on only a small portion of the shell.

Results are sensitive to local geometry conditions and scaling functions must be used to predict full shell strength. However, this method has been less tedious than internal pressurization and it is suitable for high temperature testing [9, 10].

Figure 2 illustrates the basic principles of the test method. A soft metal, such as brass, is typically used as the crush plate.

Figure 2. Diagram of the diametrical compression crush test. The SiC shell rests freely on the fixed bottom ram. The crush plate is attached to the top ram, which moves at a rate controlled by reaction force. The crush plate is thick enough to ensure that the top ram does not affect the contact pressure distribution between the SiC shell and crush plate.

While each layer of the TRISO particle is ideally spherical, manufacturing processes result in SiC shells which have unique aspheric geometries (Figure 3(a)). In existing analysis methodologies, a test article (Figure 3(b)) is represented as a uniform hemisphere with an outside radius (R) and thickness (t) [7, 8].

(a) Side-view with a reference circular annotation qualitatively showing the unique individual asphericity of a test article.

(b) Bottom-view of the cut and polished hemisphere base with reference annotations for outside radius (R) and shell thickness (t).

Figure 3. Pre-test SEM images of isolated SiC hemispherical shells.

The divot left in the soft crush plate (Figure 4) has previously been considered to be perfectly circular (Figure 4(a)), with a radius (r_0) that defines the contact area through which load is applied to the SiC test article [7, 8]. For analyses which allow for asphericity, crush plate divots may be considered ellipses (Figure 4(b)), with their minor radius (r_a) and major radius (r_b) measured from the centroid of the divot area.

Figure 4. Post-test bottom-view SEM images of divots in a brass crush plate created by contact with the SiC shell.

Diametrical compression generates tensile hoop stress on the inner surface of the shell underneath the contact area. This is similar to the tensile stress that internal pressurization generates on the entire inner surface of the SiC shell for a whole TRISO particle during in-service conditions [11]. Some previous researchers have utilized max. principal stress at the shell inner apex (σ_t) together with r_0 to define localized fracture when using Weibull statistical methods to scale up test results to full shell fracture strength (σ_{ff}^F) [7, 8]. Others have employed the more complex scaling function used herein [9]. The end result of either method is an empirically defined Weibull two-parameter statistical distribution for failure probability.

With the intention of improving existing diametrical compression testing and expanding the understanding of test mechanics, there are several questions the present study seeks to investigate:

- How do different calculation methods affect results for max. principal stress at the inner apex of the SiC shell?
- How does the max. principal stress field develop during a diametrical compression test?
- How does shell asphericity affect the principal stress fields?
- What is an appropriate local fracture strength and effective local area to use in a Weibull scaling of the fracture condition?
- What key factor(s) can be identified as limiting accuracy and/or precision?

2. Experimental Test Procedure

The TRISO-coated fuel particles were procured from Babcock and Wilcox (363 North Sam Houston Pkwy E, Houston, TX 77060) with the same lot and sample number. The particles contained surrogate ZrO_2 kernels (i.e. AGR-coated zirconia beads).

Mechanical test specimens were created by mounting the particles in hot wax and then grinding and polishing to the mid-plane using 320, 800, and 1200 grit SiC paper. ZrO_2 cores

which had not fallen out were removed with tweezers. The hemisphere particles were heated to 800°C in air to burn off PyC layers and wax. The resulting SiC hemisphere shells were sonicated in Alconox and DI water, then in isopropyl alcohol, before being air dried. Test articles were immersed in buffered oxide etchant, triple rinsed with DI water, and stored for later measurement and testing. Additionally, test articles were placed in an acetone bath and air dried immediately prior to crush testing.

After preparation, geometric parameters of each individual SiC hemisphere were taken using a scanning electron microscope and ImageJ image processing software. Thickness was determined from the mean of 8 measurements made on the polished surface. The outer radius of the shell was calculated from the cross sectional area. Hemisphere height was used to adjust measurements to the midplane [12], producing R and t , which are used in some analyses to represent each test article as a perfect hemisphere of uniform thickness.

Diametrical compression crush testing was performed in a screw-driven tensile test machine (Figure 2). The top ram had a 3 mm thick polished Brass 260 plate attached to it [7, 8]. A load cell (10 lbf) was located under the bottom ram and crosshead travel was force controlled to 3 N/min. The bottom ram was surrounded with tape to contain test article pieces after fracture. Tests were manually stopped when the audible “pop” of a breaking test article was heard and the real time load cell monitor displayed a drastic reduction in force.

Post-test, divot measurements were taken from the brass crush plate in a manner similar to pre-test SiC shell measurements. Circular divot radius (r_0) was calculated from cross sectional area. Elliptical divot radii (r_a, r_b) were measured from the centroid of the cross sectional area to the edge of the divot.

3. Calculations

Calculations were performed to create a correlation between the mechanical test condition at fracture and a Weibull two-parameter statistical definition of fracture probability for the population of whole SiC shells during in-service conditions. Quantitative results of mechanical testing are the force at fracture (P) and crush plate divot measurements (i.e. r_0, r_a, r_b). The Weibull definition of fracture probability for a ceramic population requires inputs for local fracture strength (σ_{fi}^L) and effective local area (S_{fi}^L) for each test article. Five different types of calculations were performed (one analytic, four numerical simulations) with the purpose of generating those input variables. Weibull statistical analyses were then used to determine two parameters, a shape factor and a scale factor, which define the probability of failure in-service.

3.1. Analytic Solution

A simplified force diagram for the diametrical compression crush test setup is shown in Figure 5.

Figure 5. Analytic solution diagram of the diametrical crush test. It is annotated with the test article geometry (R, t), uniformly applied load (P), radius of load area (r_0), and location of the analytic solution (i.e. inner apex) [11].

The analytic solution for the thin shell Laplacian PDE was published first by E. Reissner in 1948 [13], and again more recently [7, 8]. For a shell under a load P which is uniformly distributed within an area defined by radius r_0 , the equibiaxial hoop stress at the inner apex, σ_t , is given by [7, 8]:

$$\sigma_t = -C_1 \frac{P \sqrt{1 - \nu^2}}{t^2} + C_2 \frac{P(1 + \nu)}{t^2} \quad (1)$$

$$C_1 = 0.2205 - 0.04\mu - 0.0115\mu^2 \quad (2)$$

$$C_2 = 1.2044e^{-1.2703\mu} \quad (3)$$

$$\mu = r_0 \left[\frac{12(1 - \nu^2)}{R^2 t^2} \right]^{\frac{1}{4}} \quad (4)$$

where ν is Poisson's ratio of the shell. It is important to note that σ_t is not necessarily the peak max. principal stress (σ_{1pk}) in the entire shell. P and r_0 may be determined empirically in order to evaluate σ_t from Equations 1–4. Alternatively, r_0 may be determined by:

$$r_0 = KP^n \quad (5)$$

where K and n are empirical constants determined by FEA (see next section) [12]. By substituting Equation 5 into Equation 4, r_0 is removed as an input to the solution and σ_t as a function of P can be defined. This function describes the changing equibiaxial stress at the inner apex of the shell as force increases during a crush test.

3.2. FEA: 2D Axisymmetric Models

Three different axisymmetric FEA models were created in order to better understand the crush test and its potential solutions. They also bridge the gap between the analytic solution and a computationally intensive 3D model.

The first model (i.e. one-body 2D shell) simulates the test by applying an evenly distributed pressure to a SiC test article composed of second-order 2D axisymmetric shell elements (SAX2). It is most similar to the analytic solution. In the second model (i.e. one-body 2D quad), the test article is composed

of reduced integration second-order 2D axisymmetric quadrilateral elements (CAX8R). Both of these models can be solved with the nonlinear geometry option (NLGEOM) either on or off. (In Abaqus, when NLGEOM is turned on, the solver regularly updates the geometry with the deformed mesh.)

The third model (Figure 6) better represents test conditions, while still being limited to axisymmetric geometry. This model includes both the shell and crush plate. The contact pressure field between the two bodies is dependent on total reaction force and material properties; thus, it is not assumed to be uniform. Due to plastic deformation in the crush plate, the NLGEOM option must be turned on to yield accurate results.

Figure 6. Front view of the two-body 2D FEA model. It consists of a SiC shell simulated with reduced integration second-order 2D axisymmetric quadrilateral elements (CAX8R) and a Brass 260 crush plate simulated with first-order 2D axisymmetric quadrilateral elements (CAX4). Element edge length is $\sim 1\mu\text{m}$ in the area of contact and high stress. The bottom of the shell is constrained from translation in the y-direction. Crush force is generated by translation in the y-direction of the top edge of the crush plate.

While the analytic and one-body FEA solutions use empirical data for r_0 and P , the two-body 2D FEA defines contact by crush plate translation (Figure 7), (l_{da}), given by:

Figure 7. Diagram for calculating crush plate translation (not to scale). It is annotated with two of the input variables, shell outside radius (R) and divot radius (r_0); and the output variable, crush plate translation (l_{da}).

$$l_{da} = R - \sqrt{(R)^2 - (r_0 + C_s)^2} \quad (6)$$

where C_s is a constant related to the elastic behavior of the SiC and Brass 260. It is unique to each test article and is determined by iterating the FEA simulation until the desired divot size is achieved. In all two-body FEA models, P is a result of the simulation. It is taken as the y-direction reaction force acting on the bottom of the SiC shell (Figure 6).

3.3. FEA: 3D Symmetric Model

To incorporate some form of asphericity into the FEA simulations, a 3D symmetric model was created (Figure 8). It uses spline curves to define a test article cross-section in the xy-plane which is revolved -90° about the x-axis. This results in an aspheric test article and a crush plate divot that closely matches the empirical elliptical radii r_a and r_b .

Figure 8. Isometric view of the two-body 3D symmetric FEA model. It consists of a SiC shell and Brass 260 crush plate, both simulated with first-order 3D hexahedral elements (C3D8). Element edge length is $\sim 3\mu\text{m}$ in areas of contact and high stress. Faces on the symmetry boundaries are constrained from translation and rotation out-of-plane. The bottom surface of the shell is constrained from translation in the y-direction. Contact is prescribed by translation in the y-direction of the top surface of the crush plate.

For the purpose of quantifying deviation from the spherical shell loading condition, a dimensionless divot asphericity parameter (r_{asp}) is given by:

$$r_{asp} = \frac{r_b - r_a}{r_0} \quad (7)$$

where r_{asp} is used in comparing results of different analyses.

All FEA models use “General, Static” steps with the Abaqus standard solver and the same material properties. Elasticity data for SiC ($E=450$ GPa, $\nu=0.21$) and Brass 260 ($\nu=0.375$) were taken from the literature [8, 14, 15]. Young’s Modulus and a true stress vs. true plastic strain curve for the Brass 260 crush plate were empirically generated by subjecting a coupon of the material to a separate tensile test [12].

3.4. Weibull Statistics

An article fracturing during a crush test is the result of a tensile principal stress initiating a crack from a microscopic flaw in the SiC and propagating that crack through the shell. The tensile principal stress which initiates and propagates the crack is referred to as the local fracture strength (σ_f^L) because it is only acting on an effective local area (S_E^L) which contains the crack initiation site. The standard ratio for correlating the fracture strengths of different size areas is given by [16]:

$$\left(\frac{\sigma_f^F}{\sigma_f^L} \right) = \left(\frac{S_E^L}{S_E^F} \right)^{1/m} \quad (8)$$

where σ_f^L and S_E^L , and σ_f^F and S_E^F are the fracture strength and effective area, respectively, for the local area and full shell area, respectively; and m is the Weibull modulus.

In the crush test, S_E^L is considered to be dependent on divot size, which means that each individual test article has a unique value for S_E^L . An iterative approach is necessary to allow for the unique S_E^L values of individual test articles. In the first step, full shell fracture strength for the i th test article (σ_{fi}^F) is given by:

$$\sigma_{fi}^F = \sigma_{fi}^L \cdot \left(\frac{S_{Ei}^L}{S_E^F} \right)^{1/m} \quad (9)$$

where the i subscript denotes the i th test article and m must be given an initial estimate value for the first iteration ($m_0=3.5$). S_E^F is the full shell effective area for internal pressurization in-service conditions; it is given by:

$$S_E^F = 2 \cdot \left(4\pi (\bar{R} - \bar{t})^2 \right) \quad (10)$$

where $\bar{R}$ and $\bar{t}$ are average shell outer radius and average thickness, of the test article population, respectively. The coefficient of “2” in Equation 10 denotes that the in-service internal pressurization of a SiC shell induces two equal tensile principal stresses acting on the spherical inner surface (i.e. biaxial tension: $\sigma_1=\sigma_2$) [17]. S_{Ei}^L is the effective local area of the i th test article, which when formulated as a numerical calculation is given by [9]:

$$S_{Ei}^L = \sum_j \left[\left(\frac{\sigma_{1j}}{\sigma_{1pk}} \right)^m + \left(\frac{\sigma_{2j}}{\sigma_{1pk}} \right)^m \right] \cdot S_j \quad (11)$$

where σ_{1j} , σ_{2j} , S_j are max. principal stress, mid. principal stress, and surface area of the element face on the shell inner surface, respectively, for the j th element; and σ_{1pk} is the peak max. principal stress in the entire test article. A conservative failure probability estimator is given by [18]:

$$P_i = \frac{i}{N+1} \quad (12)$$

where P_i is the failure probability for the i th-ranked stress datum and N is the total number of samples. Determination of full shell characteristic strength (σ_0^F) and m may be performed in the standard way using:

$$\ln \left[\ln \left(\frac{1}{1-P} \right) \right] = m \cdot \ln(\sigma_f^F) + \ln \left(\frac{1}{(\sigma_0^F)^m} \right) \quad (13)$$

where the values for m and σ_0^F are adjusted to achieve the minimum residual curve fit. The new curve fit value for m is compared to the previous value and another iteration of Equations 9–13 is performed, if necessary (convergence limit: $\epsilon=0.01$).

At the conclusion of the iterative process the final values for m and σ_0^F are the shape and scale factors, respectively, which define the two-parameter Weibull distribution for the fracture strength probability of a whole SiC shell during in-service conditions.

4. Results & Discussion

Before calculations were performed, a theoretical test article labeled “AV” was generated by averaging values of the population (Table 1). This article functions as a representative which can be used in comparing calculation methods and simulating the mechanical test.

To understand how different calculation methods affect stress results, each method that models a spherical shell was used to determine σ_t at the fracture condition for the “AV” test article. Additionally, multiple calculation methods were used to simulate the mechanical test (without a fracture condition), to understand the development of principal stress fields during a test and the effects of asphericity.

Empirical fracture conditions from 8 test articles were used as inputs to each calculation method and associated values for σ_{fi}^L and S_{Ei}^L were generated. Different combinations of these values were substituted into Weibull statistical calculations and the results were compared with previous research in order to determine the current optimum input values for the Weibull distribution.

4.1. Calculation Method Comparison

Figure 9 shows σ_t for test article “AV” using each calculation method that models a spherical shell.

Max. Principal Stress vs. Calculation Method

Figure 9. Chart of σ_t vs. calculation method for the test article “AV”. The value for $P(16.47N)$ in the first three calculation methods (i.e. one-body) is the reaction force result from the two-body 2D FEA. This prevents differences in P from interfering with the comparison.

Comparing the analytic solution to the one-body 2D shell FEA, the analytic σ_t is higher if NLGEOM is off. This is due to differences in the representation of R . While the one-body 2D shell FEA appropriately models R as the outside radius, the analytic solution makes no considerations for shell thickness on a particular side of R . The analytic solution is thus limited to effectively solving for a shell with a larger R . The analytic σ_t is higher because σ_t is positively correlated with R [12].

From both one-body FEA models, σ_t is always higher with NLGEOM on. This is because the SiC shell elastically deforms during the test, flexing into a shape with a larger R in the contact

area. The NLGEOM option simulates the test more accurately and due to the positive correlation between σ_t and R , produces a higher result. The key difference between the two-body 2D FEA and the other analyses is the inclusion of the crush plate. This method produces a non-uniform load on the SiC shell; however, the results show that σ_t is not significantly affected at this r_0 and P .

The two-body 2D FEA model was also used to test the effects of friction between the plate and shell. An additional simulation using a friction coefficient ($\mu=0.30$) resulted in a similar P while reducing σ_t by $\sim 10\%$. This is likely a direct result of change in the contact force direction. Specifically, a vertical force on the shell generates tensile stress on the inner surface through bending and a horizontal force generates a compressive stress on the outer surface with bending contributing significantly less to the tensile stress on the inner surface. Therefore, shifting of the force vector to a more horizontal angle reduces σ_t . The coarse divots in Figure 4 suggest that friction is present during the test. However, further frictional analyses must be reserved for future research because there is currently no empirical data that properly quantifies μ in this application.

4.2. Mechanical Test Simulation

The calculation methods employed herein are primarily used to post-process mechanical test data at the test article fracture condition. However, these methods can also be used to simulate the mechanical test itself. (It should be noted that these simulations do not define a fracture condition and therefore cannot replace actual mechanical testing.)

Figure 10 shows the progression of σ_t and σ_{1pk} during a crush test, where results from the analytic solution and two-body 2D FEA are plotted.

Figure 10. Plot of σ_t and σ_{1pk} vs. P for test article “AV” with two calculation methods: analytic & two-body 2D FEA. The analytic solution is continuous due to Equation 5 ($K=18.38\mu\text{m}/\text{N}$ and $n=0.484$). The FEA solution must be solved discretely; however, results are produced at $\delta P \sim 0.15\text{N}$ intervals. The curves are terminated at $P=80\text{N}$, beyond expected test article cases.

Both analysis methods indicate an initial rise to a peak σ_t , followed by a decline. The difference in σ_t magnitudes at $P=16.47\text{N}$ is consistent with Figure 9. The analytic solution and FEA simulation correlate best in the beginning of the test, when elastic deformation of the SiC shell is minimal and surface pressure affects a smaller area. For $P < 35\text{N}$, σ_{1pk} is located at the inner

apex and is equal to σ_t ; therefore, σ_t can be used to represent σ_{1pk}^L (Equation 9). At $P \sim 35\text{N}$ (which correlates to $r_0 \sim 105\mu\text{m}$), the two curves diverge, the difference between σ_{1pk} and σ_t increases, and σ_t is not a valid representation of σ_{1pk}^L .

To show how the stress field on the inner surface of the SiC shell develops as a crush test progresses, the max. principal stress (σ_1) along the path shown in Figure 11(a) was plotted for 16 progressive divot sizes (Figure 12). The different symbols/colors represent different regimes of behavior, as elucidated below.

Figure 11. Two-Body FEA models which are annotated with a red line that denotes the principal stress results path.

Figure 12. Plot of σ_1 vs. x -position on the Figure 11(a) path. The legend shows the ascending r_0 values at which results are evaluated. Curve line-type and color separates them into three test regimes (i.e. blue dash lines with open circle symbols are the small divot regime, red dash lines with asterisk symbols are the large divot regime, and black dash lines with no symbols are the transition regime, with the solid black line representing the divot size that generates the largest σ_{1pk} during the entire test). These results are from the same FEA as Figure 10. The plot area is terminated at $x=120\mu\text{m}$ for simplicity.

For divot sizes up to $80\mu\text{m}$ (i.e. blue open circles in Figure 12), σ_{1pk} occurs only at the inner apex ($x=0$). At larger divot sizes, σ_{1pk} shifts to a toroid shape. Figures 10 & 12 present consistent evidence of a perpetually changing max. principal stress field with three regimes:

1. Small Divot Regime ($r_0 < \sim 80\mu\text{m}$): σ_{1pk} is increasing in magnitude and located at the inner apex

2. Transition Regime ($\sim 80\mu\text{m} < r_0 < \sim 105\mu\text{m}$): σ_{1pk} is still located at the inner apex; however, it decreases in magnitude as it increases the area it affects
3. Large Divot Regime ($r_0 > \sim 105\mu\text{m}$): σ_{1pk} is located in a toroid shaped region which decreases in magnitude as it moves further away from the inner apex

The crush test is designed to mimic internal pressurization by ideally producing biaxial tension on the inner surface of the SiC shell. This occurs from the beginning of the test through the transition regime. However, if divots are too small, measurement error in r_0 , r_a , and r_b are prohibitively large (e.g. annealed nickel crush plate [12]). The Brass 260 crush plate achieves optimal r_0 values at room temperature, fracturing test articles near the end of the small divot regime ($r_0 \sim 70\mu\text{m}$).

The general behavior of the principal stress fields (Figures 10–12) is a result of test geometry (i.e. elastically deforming hemispherical shell in contact with a plastically deforming flat plate). When shell dimensions are held constant, the magnitudes of the principal stress fields are a result of the material strength of the crush plate; specifically, the yield strength (YS_{min}) and the subsequent plastic strain curve which terminates at tensile strength (TS_{min}). As it is important to perform mechanical tests which produce fracture inducing σ_{1pk} values near the end of the small divot regime, the two-body 2D FEA model can be used to select appropriate crush plate materials.

It is important to note that the present mechanical test simulation results are dependent on Brass 260 mechanical properties at room temperature ($YS_{min} \sim 225\text{MPa}$, $TS_{min} \sim 545\text{MPa}$). Future high temperature testing needs to repeat the mechanical test simulation process on candidate crush plate materials in order to find one that will fracture test articles near the end of the small divot regime. This includes generating Young's modulus and true stress vs. true plastic strain data at test temperatures.

Figure 13 shows tensile principal stresses (σ_1 , σ_2) on the two edge path in Figure 11(b) for an aspheric shell ($r_a = 61.1\mu\text{m}$, $r_b = 82.4\mu\text{m}$).

Figure 13. A plot of σ_1 and σ_2 vs. position. These results are from the two-body 3D FEA “AV” test article simulation at $r_a = 61.1\mu\text{m}$, $r_b = 82.4\mu\text{m}$, and $P = 16.22\text{N}$. As a free surface, the inner surface of the SiC shell is in plane stress, resulting in one principal stress component always equal to zero. The min. principal stress (σ_3), which is not plotted here, is either zero or in compression throughout the test.

Shell asphericity results in principal stress profiles which are not symmetric and the peak stresses below the divot are not equibiaxial. Thus, considering the stress fields of σ_1 and σ_2 independently per Equation 11 represents an improvement in accuracy.

4.3. Comparison to Empirical Data

Figure 14 shows the correlation between force and divot size for 9 SiC hemispheres crush tested using a Brass 260 crush plate.

Figure 14. A plot of r_0 vs. P with power law curve fits. (This data is analogous to performing a hardness test on the crush plate where the indenter is the SiC shell.) The first dataset is the empirically generated mechanical test results of the 9 test articles. The second set consists of r_0 vs. P values from the two-body 2D FEA mechanical test simulation used in Figures 10–12. The third set is produced by the two-body 3D FEA mechanical test simulation, where plotted r_0 values match the elliptical area created by r_a and r_b .

It is seen that the two-body 2D FEA divot sizes are consistently smaller than the empirical test, possibly because it simulates the Brass 260 as a stiffer material than it actually is. Altering the Brass 260 material properties in the FEA model to simulate it as a softer material would shift the 2D FEA curve. However, due to the variability in the empirical data, that shifted curve would still be a poor fit (as shown by the empirical power law curve fit).

To further investigate the variability in the empirical data points, the ratio of the FEA reaction force to the empirically measured force was calculated (Table 1). This force ratio does not correlate to r_{asp} , suggesting that divot radii values do not capture a contributing factor to variability in the empirical data points. Figure 14 shows that multiple empirical data points have an $r_0 \sim 70\mu\text{m}$ for different values of P . SiC shell asphericity in the direction of divot depth is the likely cause, because it allows divots with similar radii to have different volumes. In Figure 14, the 3D FEA results match the 2D FEA power law curve because although the 3D FEA introduces asphericity, that limited model of asphericity results in divots of similar volume. Thus, a more complex representation of asphericity in a FEA model will be necessary to produce r_0 vs. P results that match empirical data. The best way to implement this is to use actual shell geometry in a non-symmetric 3D FEA.

An important input to the Weibull scaling function is σ_{fi}^L , which is plotted against P in Figure 15.

Table 1. Discrete Empirical Input and Solution Results Data.

	Test Article ID Number								AV
	1	2	3	4	5	6	7	8	
Geometry Inputs (μm)									
Shell Outside Radius (R)	397	378	402	413	397	384	398	408	397
Shell Thickness (t)	38.0	38.1	37.6	37.3	37.0	37.3	37.2	36.7	37.4
Plate Divot Radius (r_0)	68.0	69.8	70.0	81.8	71.9	62.2	69.7	70.4	70.5
Plate Divot Minor Radius (r_a)	60.1	57.7	59.0	79.0	65.3	53.9	62.9	59.6	62.2
Plate Divot Major Radius (r_b)	75.8	83.1	83.1	85.4	79.6	73.9	77.7	82.9	80.2
Divot Radius Asphericity Constant (r_{asp})	0.230	0.364	0.346	0.079	0.199	0.322	0.213	0.330	0.256
Max. Principal Stress Results (MPa)									
Analytic Solution (σ_t)	2230	1274	1727	2120	2124	1414	1827	2113	2473
FEA One-Body 2D Shell (NLGEOM=off)	2093	1202	1631	2068	2021	1315	1727	2004	2341
FEA One-Body 2D Shell (NLGEOM=on)	2273	1265	1749	2370	2227	1374	1862	2196	2599
FEA One-Body 2D Quad (NLGEOM=off)	2378	1374	1844	2312	2279	1501	1951	2250	2647
FEA One-Body 2D Quad (NLGEOM=on)	2535	1429	1948	2586	2462	1551	2071	2421	2874
FEA Two-Body 2D (σ_{1pk})	2772	2707	2865	3046	2919	2673	2871	2979	2871
FEA Two-Body 3D (σ_{1pk})	2887	3109	3277	2984	3038	2886	2946	3308	3132
Reaction Force Results (N)									
Empirical Data (P)	13.68	8.67	10.96	18.37	14.21	7.44	11.49	13.05	–
FEA One-Body 2D Quad (NLGEOM=on)	13.51	8.61	10.85	18.12	14.03	7.38	11.37	12.89	15.82
FEA Two-Body 2D	15.12	16.00	16.12	21.77	16.85	12.81	15.75	16.11	16.47
FEA Two-Body 3D	14.48	15.99	16.68	21.92	16.87	12.98	16.19	16.54	16.22
Force Ratio (Two-Body 2D FEA to Empirical)	1.11	1.85	1.47	1.19	1.19	1.72	1.37	1.23	–

Figure 15. A plot of σ_{fi}^L vs. P for 8 of the test articles (one was removed as an outlier). There are four datasets, one for each solution method. The legend shows the calculation result that is taken as σ_{fi}^L for each method. The analytic solution and one-body 2D quad FEA both use r_0 and P as independent input values. In the two-body 2D and 3D FEA, r_0 and P are dependent variables in the solution.

The results in Figure 15 correlate with the curves in Figure 10 (i.e. rise and fall of stress as force increases). The analytic solution and one-body 2D quad FEA results also correlate with the findings in Figure 9. However, both two-body FEAs have inaccurately high values for P , as would be expected from the results in Figure 14. Due to the positive correlation between P and σ_{1pk} , the two-body FEAs result in erroneously larger σ_{1pk} values.

Another notable characteristic in Figure 15 is the difference

in variability between two groups of solutions: those that use r_0 and P as independent input variables (analytic and one-body FEAs) and those which produce r_0 and P as solution dependent results (two-body FEAs). As previously stated, the two-body 2D FEA does not incorporate asphericity and the 3D FEA uses a limited representation of asphericity. While both the analytic solution and one-body 2D FEAs feature a spherical SiC shell, they incorporate the effects of real shell asphericity by way of empirically generated independent values for r_0 and P . This is what creates the higher, and more realistic, variability in those solutions.

Variability in σ_{fi}^L is crucial because it is negatively correlated to m , which in turn is positively correlated to σ_{fi}^F (i.e. low variability generates less reduction in fracture strength when scaling to larger areas, resulting in calculated full shell fracture strengths that are erroneously high).

Figure 16 shows results for three methods of performing the Weibull curve fit on the 8 test articles.

Figure 16. A log-log plot of the Weibull curve fit for three methods: the first, where both σ_{fi}^L and S_{Ei} are from the two-body 3D FEA solution; the second, where both σ_{fi}^L and S_{Ei} are from the one-body 2D quad FEA solution; and the third, where σ_{fi}^L is from the one-body 2D quad FEA solution and S_{Ei} is from the two-body 3D FEA solution.

The resulting values for the two-parameter Weibull curve fit for the first method are $m=17.33$ and $\sigma_0^F=1877\text{MPa}$. Considering the known limitations in the 3D FEA representation of asphericity, the previously stated inaccuracies in two-body FEA values for P and σ_{1pk} , and the lack of correlation with previously published TRISO data, it is certain that this method is currently not suitable for determining σ_0^F for the population [14]. In future research, this method is expected to yield the most accurate results if the actual geometry of individual SiC shells can be modeled.

The second method results in $m=3.67$ and $\sigma_0^F=517.6\text{MPa}$. While the one-body 2D quad FEA used in this method does not directly incorporate asphericity, it does incorporate some of the effects of true shell asphericity by using independent empirical values for r_0 and P as inputs to the FEA model. The resulting higher variability in σ_{fi}^L values (Figure 15) results in a shape factor, m , which is similar to previously published TRISO data [14]. However, the limitations of the one-body model include assumptions of perfect spherical geometry and uniformly distributed surface pressure, bringing into question the accuracy of σ_0^F .

The third method utilizes the one-body 2D quad FEA for determining σ_{fi}^L and the two-body 3D FEA for calculating S_{Ei} with Equation 11. This allows for use of the more complex principal stress profiles generated by an aspheric shell (Figure 13) while also incorporating the higher variability σ_{fi}^L values generated by a one-body solution. Because the shape of the principal stress profiles are primarily dependent on r_0 and values for σ_1 and σ_2 are normalized by σ_{1pk} in Equation 11, S_{Ei} values from the 3D FEA can be used together with σ_{fi}^L values from the one-body 2D quad FEA. This method results in Weibull distribution parameters of $m=3.80$ and $\sigma_0^F=332.9\text{MPa}$. Of the three methods, these results are most consistent with previous TRISO crush test research [14].

5. Conclusions

5.1. SiC Shell Geometry

Aspheric shell geometry in the localized contact area has a significant influence on the resulting principal stress fields:

1. Elastic deformation of the SiC shell (simulated with NL-GEOM) causes the local shell radius to increase, thus creating asphericity during the test which increases σ_1 and σ_2 on the inner surface of the shell, regardless of outer surface pressure condition (Figure 9).
2. Asphericity in the initial shell geometry can result in a different local shell radius in comparison to a spherical approximation of the shell geometry (Figure 3). This different local radius affects σ_1 and σ_2 in a manner similar to the aforementioned elastic deformation.
3. Asphericity present in test articles can result in different volumes of crush plate material being displaced, even when post-test results for divot radii are similar. This affects the magnitude of force acting on the SiC shell during the test and results in values for r_0 and P which are dependent on an individual test article's unique geometry.

It has been shown herein that 1 and 2 listed above affect results for σ_{1pk} and the solution is less accurate when they are not accounted for (Table 1). It has also been shown that simplistic artificial models of asphericity are not sufficient to generate valid results for fracture strength magnitude or population variability. Thus, it cannot be assumed that solutions which approximate SiC geometry as a spherical shell, or a predictably aspheric shell, produce accurate results for determining characteristic strength.

In response to this information, two improvements are recommended:

1. Use a method such as x-ray tomography to better represent individual test article geometry.
2. Compare work-energy performed during empirical tests and strain-energy totals in FEA to verify simulation accuracy.

Previous diametrical compression tests have produced test data fitting a Weibull distribution [7, 8]. These improvements seek to extend the usefulness of this test method by accounting for geometry randomness (presumably normally distributed), resulting in more accurate Weibull distribution parameters.

5.2. Frictional Contact

It has been shown that σ_{1pk} is dependent on the friction coefficient between the plate and shell. In moving forward, future tests should include an empirically derived friction coefficient. This can be determined by using a SiC test article and crush plate sample in an empirical friction coefficient test. A good candidate for this test is the ASTM G133 linearly reciprocating ball on flat sliding wear test [19, 20].

5.3. Principal Stress Fields

The principal stress fields on the inner surface of the SiC shell change drastically during a crush test. These changes introduce challenges in determining local fracture strength. This is exemplified in three ways:

1. As force increases, the pressurized area increases. The interdependency of these variables affects the stress field so that σ_{1pk} changes over time. Thus, sampling σ_{1pk} at fracture does not inherently consider all of the previous σ_{1pk} values the SiC shell has survived (Figure 10).
2. As the pressurized area increases, the shape of the principal stress field drastically changes (Figure 12). The importance of this change is exemplified by the need to divide the development of the stress field into three regimes (i.e. small divot, transition, and large divot). During the large divot regime, σ_{1pk} is no longer present at the inner apex, rendering the analytic solution invalid. The large divot regime also introduces more complications, such as a drop in σ_{1pk} and deviation from biaxial tension.
3. The two Weibull distribution inputs of σ_{fi}^L and S_{Ei}^L cannot currently be generated by a single solution method. This is likely due to an inability to accurately represent individual test article geometry. In lieu of a single solution, these values can be derived from separate solutions (i.e. two different FEA models).

The research herein shows that it cannot be assumed that the maximum σ_{1pk} for the crush test is present at the time of fracture or that σ_{1pk} occurs at a fixed location throughout the test. It has also been shown that if true test article geometry is not modeled, two different FEA simulations are necessary to obtain acceptable inputs for the Weibull distribution.

These conclusions reinforce the previously stated need to accurately model test article geometry. Additionally, there are two more recommendations:

1. Control test conditions so that all (or nearly all) of the test articles fracture near the end of the small divot regime.
2. Implement a ceramic fracture post-processing methodology which has the capability to consider varying stress fields over time, for articles that fracture in the large divot regime.

This research is part of the history of incremental improvements in TRISO SiC shell strength testing. All recommendations herein are included with the intention to assist in future improvements to the diametrical compression crush test.

6. Acknowledgments

This research was funded by Battelle Energy Alliance, LLC (DOE-NEUP DE-AC07-05ID14517). The authors are thankful to Dr. Paul Demkowicz of Idaho National Laboratory for helpful discussions.

References

- [1] D. A. Petti, P. A. Demkowicz, J. T. Maki, et al., Triso-coated particle fuel performance, *Comprehensive Nuclear Materials* 3 (INL/JOU-09-16642).
- [2] K. A. Terrani, L. L. Snead, J. C. Gehin, Microencapsulated fuel technology for commercial light water and advanced reactor application, *Journal of Nuclear Materials* 427 (1) (2012) 209–224.
- [3] A. G. Evans, C. Padgett, R. W. Davidge, Strength of pyrolytic sic coatings of fuel particles for high-temperature gas-cooled reactors, *Journal of the American Ceramic Society* 56 (1) (1973) 36–41.
- [4] Idaho National Laboratory, Summary for the Next Generation Nuclear Plant Project in Review INL/EXT-10-19142 (Revision 1).
- [5] K. Bongartz, E. Gyarmati, H. Nickel, H. Schuster, W. Winter, Measurement of young's modulus and fracture stress on htr particle coatings by the brittle ring test, *Journal of Nuclear Materials* 45 (3) (1972) 261 – 264.
- [6] K. Gilchrist, J. Brocklehurst, A technique for measuring the strength of high temperature reactor fuel particle coatings, *Journal of Nuclear Materials* 43 (3) (1972) 347 – 350.
- [7] T. S. Byun, J. D. Hunn, J. H. Miller, L. L. Snead, J. W. Kim, Evaluation of fracture stress for the sic layer of triso-coated fuel particles using a modified crush test method, *International Journal of Applied Ceramic Technology* 7 (3) (2010) 327–337.
- [8] T. S. Byun, J. W. Kim, I. Dunbar, J. D. Hunn, Fracture stress data for sic layers in triso-coated fuel particles, Tech. Rep. ORNL/TM-2008/167, Oak Ridge National Laboratory (September 2008).
- [9] S.-G. Hong, T.-S. Byun, R. A. Lowden, L. L. Snead, Y. Katoh, Evaluation of the fracture strength for silicon carbide layers in the tri-isotropic-coated fuel particle, *Journal of the American Ceramic Society* 90 (1) (2007) 184–191.
- [10] T. S. Byun, S. G. Hong, L. Snead, Y. Katoh, Influence of specimen type and loading configuration on the fracture strength of sic layer in coated particle fuel, *Ceramics in Nuclear and Alternative Energy Applications, Ceramic Engineering and Science Proceedings, Cocoa Beach* 310 (2009) 77.
- [11] W. C. Young, R. G. Budynas, Roark's formulas for stress and strain; 7th ed., McGraw-Hill, New York, NY, 2002.
- [12] B. C. Davis, Fracture strength of the sic layer in triso coated fuel particles, Master's thesis, Colorado School of Mines (2013).
- [13] E. Reissner, Stresses and small displacements of shallow spherical shells, ii, *Journal of Math and Physics* 25 (4) (1947) 233 – 242.
- [14] L. L. Snead, T. Nozawa, Y. Katoh, T.-S. Byun, S. Kondo, D. A. Petti, Handbook of sic properties for fuel performance modeling, *Journal of Nuclear Materials* 371 (13) (2007) 329 – 377.
- [15] Committee, ASM International Handbook, ASM Handbook: Properties and Selection : Nonferrous Alloys and Special-Purpose Materials (ASM Handbook) VOL. 2, 10th Edition, ASM International, 1990.
- [16] D. Davies, The statistical approach to engineering design in ceramics, in: *Proc. Brit. Ceram. Soc.*, Vol. 22, 1973, pp. 429–452.
- [17] A. M. Freudenthal, Statistical approach to brittle fracture, *Fracture* 2 (1968) 591–619.
- [18] B. Bergman, On the estimation of the weibull modulus, *Journal of Materials Science Letters* 3 (8) (1984) 689–692.
- [19] ASTM International, ASTM G115 - Standard Guide for Measuring and Reporting Friction Coefficients (2013).
- [20] ASTM International, ASTM G133 - Standard Test Method for Linearly Reciprocating Ball-on-Flat Sliding Wear (2010).