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Factors Affecting Defective Fraction of Biso-Coated HTGR Fuel Particles During In-Block Carbonization

A. J. Caputo
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FUEL PARTICLES DURING IN-BLOCK CARBONIZATION

A. J. Caputo, D. R. Johnson, and C. K. Bayne

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A. J. Caputo, D. R. Johnson, and C. K. Bayne*

ABSTRACT

The performance of Biso-coated thorium fuel particles during the in-block processing step of HTGR fuel element refabrication was evaluated. The effect of various process variables (heating rate, particle crushing strength, horizontal and/or vertical position in the fuel element blocks, and fuel hole permeability) on pitch coke yield, defective fraction of fuel particles, matrix structure, and matrix porosity was evaluated. Of the variables tested, only heating rate had a significant effect on pitch coke yield while both heating rate and particle crushing strength had a significant effect on defective fraction of fuel particles.

INTRODUCTION

Fuel particles for the High-Temperature Gas-Cooled Reactor (HTGR) are coated with pyrolytic carbon (PyC) and/or silicon carbide to contain fission products. The coatings must remain intact during fuel fabrication and in service to avoid fission product release. The purpose of this work was to study the effects of fuel refabrication processing variables on the failure of fuel coatings during in-block processing of element refabrication.

The fuel element for the HTGR comprises a machined graphite block that contains coolant holes and fuel holes, which contain the fuel rods. The graphite block acts as the moderator and also as a structural member of the reactor core. The fuel is in the form of thorium (fertile) and uranium oxide-uranium carbide (fissile) microspheres coated with multiple layers of pyrolytic carbon and, in the case of the fissile microspheres, silicon carbide. The fuel particles are bonded together into cylindrical

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*Computer Sciences Division.

rods by a carbonaceous matrix. The prismatic graphite block has a hexagonal cross section 360 mm (14.2 in.) across the flats and is 793 mm (31.2 in.) high. The fuel rods are contained in blind holes drilled parallel to the vertical axis of the element, and coolant holes are drilled parallel to the fuel holes. Figure 1 illustrates the geometry of the fuel element; the hexagonal symmetry of the fuel and coolant hole positions is apparent. Each fuel hole is surrounded by three fuel holes and three coolant holes, but each coolant hole is surrounded by six fuel holes. The graphite web separating the fuel from the coolant is nominally 4.52 mm thick (0.178 in.)

The fuel rods are molded in the following sequence: (1) the fissile, fertile, and graphite shim particles are dispersed into a steel die; (2) a preformed, cold-pressed, matrix slug (consisting of graphite particles and low coke yield additives in a petroleum pitch binder) is added to the die; (3) the mold assembly is heated and the molten matrix is injected into the void between the particles; and (4) the mold assembly is then cooled and the fuel rod ejected from the mold. The molded rods are loaded into the fuel element holes, and the holes are capped with graphite plugs. The fuel element is then heated in an argon atmosphere to 1000°C to carbonize the pitch and drive off volatile decomposition products. The element is then heat treated at 1800°C to stabilize the fuel rods. This processing in the graphite block is referred to as "in-block carbonization," or more simply as "cure-in-place."

During carbonization, volatile decomposition products of the pitch must escape from the fuel holes by diffusing through the graphite webs separating the fuel holes from the adjacent coolant holes. The heating rate during carbonization controls various properties of the fuel rods. These properties include pitch coke yield* of the fuel rod matrix, matrix microstructure, and defective fuel fraction. The defective fuel fraction (failed coatings) is extremely important because the coatings provide the primary means of retaining fission gas.

$$\text{*pitch coke yield (\%)} = \frac{\text{weight of pitch coke in carbonized rod}}{\text{weight of pitch and other volatile constituents in green rod}} \times 100$$

Fig. 1. HTGR Graphite Fuel Element Block.

Failure of a fraction of the fuel particle coatings during in-block carbonization (cure-in-place) processing may be due to an interaction between the matrix (actually the pitch component of the matrix) and the coatings. The thermoplastic pitch shrinks during carbonization. There is considerable evidence that when the shrinkage occurs at the outer coating there can be sufficient stress generated between the shrinking matrix and the nonshrinking coating to either break coatings or initiate tears that fail during further shrinkage in an operating reactor. Also, it could be that tears occur at low temperatures during cooldown from the higher annealing temperatures due to differences in thermal expansion. Recent studies by Moore and Godfrey¹ have shown that the room temperature coefficients of thermal expansion for matrix and coating differ by a factor of nearly two (1.92 vs $3.74 \times 10^{-6} \text{ K}^{-1}$, respectively). An example of broken coatings of irradiated fuel is shown in Fig. 2, while Fig. 3 shows the initiation of a tear in the coating of an as-fabricated fuel rod.

Carbonization at lower heating rates results in higher pitch coke yields and thus more particle-matrix contact. Also, the matrix is denser and stronger when lower heating rates are used. These factors result in a greater possibility for matrix-particle interaction when lower heating rates are used.

Another means of reducing matrix-particle interaction is to increase the potential of the particle coating to withstand stresses. This potential has been related to the fuel particle crushing strength.²⁻⁴ Fuel particle crushing strength is determined for a sample of particles prior to fuel rod fabrication by placing individual particles between two flat plates and measuring the load required to initiate coating fractures. The influence of particle coating conditions on crushing strength has been reported elsewhere.⁵

Prior to this study, effect of the position of the individual fuel rod within the large fuel element block was uncertain. Any differences in heating rate within the block would no doubt have an effect on coke yield. If the potential of the volatile components of the pitch to escape from the fuel holes varied along the long length [787 mm (31 in.)]

Fig. 2. Particles Broken During Irradiation Due to Matrix-Particle Interaction. Carbonized in graphite tube. Irradiated to 3.6×10^{21} n/cm² ($E > 0.18$ MeV) at 940–1145°C. (a) Biso particle. (b) Triso particle.

Fig. 3. As-Fabricated Fuel Rod Showing Tearing of Particle Outer Coating Due to Particle-Matrix Interaction.

of the fuel column (due to differences in graphite permeability), an effect on the pitch coke yield might be expected.

Thus, this study was conducted to determine quantitatively the effects of heating rate, particle strength, and rod position in the fuel block on pitch coke yield, defective fuel fraction, and matrix structure. This work was performed with Biso-coated fertile particles; a similar study with Triso-coated fissile particles is planned for the future.

The effect of various process modes on fuel rod characteristics has been reported elsewhere²⁻⁴ over the years. However, prior to this investigation, in-block carbonization was simulated by placing fuel rods in graphite cylinders. This study was the first to determine the effect of various process variables on fuel rod characteristics when processed in large segments of fuel elements.

EXPERIMENTAL PROCEDURE

To statistically test the effects of the four control variables mentioned, an experiment was designed for different levels of processing conditions. The control variables and their levels are as follows:

<u>Controlled Variables</u>	<u>Level</u>
Heating rate	1°C/min; 5°C/min; 10°C/min; 15°C/min
Particle crushing strength	22.9 N (5.14 lb); 25.7 N (5.78 lb); 30.5 N (6.85 lb)
Horizontal position (hole in graphite block)	Hole 1; hole 2, hole 3
Vertical position (location of fuel rod in the graphite block hole)	Top; middle; bottom

A carbonization run was made at each of the four heating rates with fuel rods made from three fertile particle batches. The rods were placed at each of three vertical positions (top, middle, and bottom) and in each of the three horizontal positions (holes 1, 2, and 3). A fifth

run was made using only one particle batch in all vertical and horizontal positions. A sixth run was made to obtain an initial cursory view of the effect of fuel hole permeability, if any, upon coke yield and defective fuel fraction. This was done because an earlier study⁶ showed a significant variation in the relative argon permeability of the fuel holes within the graphite fuel element block.

In each of the four initial runs, which had rods of each particle strength in each horizontal and vertical position, the fuel rods were randomized in each fuel hole (horizontal position) and replicated at each vertical position (top, middle, and bottom). The replication was done to obtain a measure of precision of the process and/or the analytical procedure. A schematic representation of the experimental design for the four runs is shown in Fig. 4. A total of 72 fuel rods were used in these initial four runs, but because there are 108 possible combinations of the levels of the controlled variables, the vertical position effect was not measured by these runs. Any vertical position effect from this experiment would be included in the particle strength effect.

To separate the vertical position effect from the particle strength effect, the fifth run (made at a heating rate of 1°C/min) used 18 fuel rods and only one particle type, crushing strength of 22.9 N (5.14 lb), in all vertical and horizontal positions.

The main emphasis of the initial four runs was to measure the effects of heating rate and particle strength. In these runs, the three holes (horizontal position) used in each fixture were chosen for their uniform relative argon permeability value of about 17 liters/min. This value was chosen since it was the average relative permeability determined for the "regular" type holes in a previous study⁶ of ten Fort St. Vrain type fuel blocks. Since the earlier study showed a significant variation in the permeability of the fuel holes, the sixth run was made to get an initial overall view of the effect of permeability. In this permeability test run, fuel rods made from the same particle batch were placed in holes that had argon permeability values of 4.3, 9.8, 13.3, and 16.6 liters/min. The particle batch with the lowest crushing strength, 22.9 N (5.14 lb), was used since this would most likely show the

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		Heating Rate 1°C/min			Heating Rate 5°C/min		
		Hole			Hole		
		1	2	3	1	2	3
Top	{		P ₃	P ₂	P ₁	P ₃	P ₂
		P ₁	P ₃	P ₂	P ₁	P ₃	P ₂
Middle	{	P ₂	P ₁		P ₃	P ₂	P ₃
		P ₂	P ₁	P ₃	P ₃	P ₂	P ₃
Bottom	{	P ₃	P ₂	P ₁	P ₂	P ₁	P ₁
		P ₃		P ₁	P ₂	P ₁	P ₁

		Heating Rate 10°C/min			Heating Rate 15°C/min		
		Hole			Hole		
		1	2	3	1	2	3
Top	{	P ₃	P ₁	P ₁	P ₃	P ₁	P ₁
		P ₃	P ₁	P ₁	P ₃	P ₁	P ₁
Middle	{	P ₂	P ₃	P ₂	P ₁	P ₃	P ₃
		P ₂	P ₃	P ₂	P ₁	P ₃	P ₃
Bottom	{	P ₁	P ₂	P ₃	P ₂	P ₂	P ₂
		P ₁	P ₂	P ₃	P ₂	P ₂	P ₂

Fig. 4. Design for Four Experimental Carbonization Runs. P₁, P₂, and P₃ represent particle strengths of 22.9 N (5.14 lb) (A-637); 25.7 N (5.78 lb) (A-642); and 30.5 N (6.85 lb) (A-635); respectively. A blank space represents a missing value.

permeability effect on the defective fraction of fuel particles. The fuel was carbonized at the reference heating rate of 10°C/min.

The sample fuel rods were fabricated by the reference slug-injection process in which the softened matrix is pressure-intruded into the void volume of a bed of particles. Since the major objective of this experiment was the detection of defective particles due to in-block processing, it was extremely important that fuel particles not be damaged by forces present during this matrix injection step of fuel

rod formation. Thus, trial rods were made and analyzed in the green state to verify that particles were not broken during rod formation. An injection pressure of 5.52 MPa (800 psi) was used for particle batches A-635 and A-642 that had the higher crushing strength of 30.5 and 25.7 N (6.85 and 5.78 lb) respectively. A pressure of 4.14 MPa (600 psi) was used for batch A-637, which had the lowest crushing strength of 22.9 N (5.14 lb). No fuel particle failure was detectable when rods were molded at these pressures. Other than this injection pressure modification, all other rod formation conditions were those typical for the slug injection process.

Because of the size limitation of the laboratory carbonization and the heat-treating furnaces, this work was conducted using segments of the full-size fuel element block. The segments were full size in length, but 1/6 in cross section. The six block segments were then reduced in diameter to 133.4 mm (5 1/4 in.) in order to fit the carbonization and the heat-treating furnaces. This produced a fixture as shown in Fig. 5. The fixture contained 24 fuel holes as shown more clearly in Fig. 6. However, holes along the outside surfaces no longer have the typical hole layout (that is, each fuel hole having three fuel holes and three coolant holes adjacent to it). Thus, sample rods were not placed in the outside holes, but were limited to the holes in the segment that had the normal layout. These holes are referred to as "regular holes." The area containing these holes is shown in Fig. 6 and all of these holes were filled with fuel rods.

All the fuel rods within a hole (that is, the fuel column) were essentially loaded as we interpret the reference loading requirements for HTGR recycle fuel.^{7,8} That is, the fuel column was composed of 15 fuel rods [nominal 13-mm-diam $\times$ 49.3-mm-long (1/2 $\times$ 1.94 in.)] and seven plastic spacers, each 3.8-mm-thick (0.150-in.), distributed along the fuel column. A plastic spacer was placed beneath the top fuel rod and beneath every second rod thereafter. This arrangement is shown in Fig. 7(a). The rod loading arrangement required more fuel rods than were necessary for the test. The additional "filler rods" were identical to the test rods except that the filler rods were made from a different

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Fig. 5. In-Block Carbonization Fixture 787.4 mm Long (31 in.).

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Fig. 6. Top Surface of Carbonization Fixture.

Fig. 7. Fuel Column Loading Plan. (a) Reference loading plan. (b) Loading plan for fuel holes containing sample rods.

batch of particles. This placement of plastic spacers was used for the holes without the sample rods and was modified only slightly for the holes with the sample rods. Since the sample rods had to be weighed to determine the coke yield and analyzed for defective fuel particles, the possibility of adjacent rods sticking together could not be tolerated. Thus, thinner plastic spacers [2.54 instead of 3.81 mm (0.100 instead of 0.150 in.)] were used and were placed on both sides of all the sample rods, as shown in Fig. 7(b). An additional spacer was used between two of the filler rods so that the total amount of plastic spacer material used [$10 \times 2.54 = 25.4$ mm ($10 \times 0.100 = 1.0$ in.)] was essentially the same as that used for the regular, reference loading scheme [$7 \times 3.81 = 26.67$ mm ($7 \times 0.150 = 1.05$ in.)].

The fuel rods are not normally unloaded from the graphite block after processing; the completed and inspected fuel element is inserted

into the reactor core. However, in this experiment, the fuel rods had to be unloaded for analysis. There is a tendency for some rods to stick to the walls of the fuel holes, so if a large force were used to remove the rods from the block, fuel particles could be damaged. This would make it impossible to distinguish the source of any defective particles. Thus, filler rods were placed on either side of each pair of sample rods so that the fuel block could be cut through the filler rods and the sample rods removed.

After forming the fuel rods and loading the graphite blocks as described above, the four main carbonization runs were made at 1, 5, 10, and 15°C/min up to 800°C in an argon atmosphere. The fixture was then cooled and unloaded from the carbonization furnace and then heat-treated (at 10°C/min) up to 1800°C in an induction graphite furnace, again using an argon atmosphere. After this, the fixtures were cut as described earlier and the sample rods removed. The fifth run, made to separate the position effect from the particle strength effect, was made at 1°C/min and the sixth run to study the block permeability effect was made at 10°C/min. The pitch coke yields were calculated using the fuel particle weight and the green and fired rod weights. The defective particle content of the rods was determined using the chlorine leach method.⁹ With this method, gaseous chlorine is passed through the rods for 2 hr at 1500°C. If a fuel particle has a defective (cracked) outer PyC coating, the chlorine will pass through the porous buffer PyC coating and react with the ThO₂ kernel forming volatile ThCl₄. The amount of thorium leached from the rod versus the original amount of thorium in the formed rod defined the "defective fuel fraction." The defective fraction is also defined as the number of defective particles divided by the total number of particles in a rod. The total number of particles in a rod is listed in Table 1. The amount of thorium leached from a rod can be used to determine the number of defective particles by using the nominal thorium content per particle shown in Table 1. The specification for desired defective fuel fraction¹⁰ is $<1 \times 10^{-4}$.

In addition to the coke yield and defective particle fraction, the matrix microstructure, microporosity, and macroporosity were also evaluated.

Table 1. Characteristics of Fuel Rods and Fuel Particles

Particle Batch Number	Particle Crushing Strength (lb)	Thorium Content Per Rod (g)	Thorium Content Per Particle (μ gm)	Average Number of Particles per Rod
P ₁ (A-637)	5.14	7.198	555	12,970
P ₂ (A-642)	5.78	6.173	578	11,620
P ₃ (A-635)	6.85	8.774	565	15,540

Three metallographic samples were taken from each of the four initial runs, one of each of the three particle types with the samples representing the top, middle, and bottom vertical position. The samples were evaluated visually and the matrix macroporosity and microporosity were determined using a Quantimet model 720; an advanced model quantitative television microscope (QTM).

EXPERIMENTAL RESULTS

The results of the experiment, discussed below, are divided into the general areas of (1) pitch coke yield, (2) defective fraction of fuel particles, and (3) matrix microstructure (microporosity and macroporosity). A brief description of the statistical method used to analyze the data is attached as Appendix A, and the experimental data are attached as Appendix B.

DISCUSSION OF RESULTS

Pitch Coke Yield

The pitch coke yield for all the individual fuel rods in the initial four runs is shown in Appendix B, Table B-1. A summary of the pitch coke yield data, including the mean and the 95% confidence level about the mean, for the four heating rates is given in Table 2. The decrease in coke yield with increases in heating rate is quite evident. In addition, the statistical analysis showed that heating

Table 2. Pitch Coke Yield of the In-Block Carbonization Process Versus Heating Rate

Heating Rate ^a (°C/min)	Mean Pitch Coke Yield (%)	95% Confidence Interval About the Mean (%)
1	37.8	37.3-38.3
5	32.5	32.0-33.0
10	29.3	29.0-29.6
15	28.0	27.6-28.4

^aHeating rate of the carbonization cycle between 300 and 600°C.

rate is the only process variable that had a significant effect on coke yield. Variations in the heating rate within the fixtures were not large enough to significantly influence the coke yield.

The statistical method to analyze the coke yield data was the method of analysis of variance (AOV).¹¹ This method partitions the adjusted total sum of squares [i.e., $\sum(\text{coke yield} - \text{mean})^2$] into each source that may cause the coke yield to vary from an overall mean. For the initial four runs, the sources of variation are the heating rate (heat), the particle crushing strength (strength), the different holes nested in each heating rate [hole (heat)], the interactions of heating rate with particle strength (heat $\times$ strength), and particle strength with the different holes nested in each heating rate [strength $\times$ hole (heat)]. The remaining sum of squares is due to measurement errors, which are assumed to be independent and identically distributed as a normal distribution with zero mean and constant variance.

The partition of the sum of squares for the initial four runs is summarized in Table 3. An outlier test showed that the low coke yield value of 27.03% (see Appendix B, Table B-1) was significant at the 1% significance level. This point was deleted from the analysis of the coke yield data.

The mean square for a source, which is the sum of squares divided by the degrees of freedom (DF), can be used to test the significance

Table 3. Coke Yield AOV Table for the Initial Four Experimental Runs

Source	DF	Sum of Squares	Mean Squares	F-Statistic
Heat	3	879.15	293.05	395.35 ^a
Strength	2	0.96	0.48	0.65
Hole (Heat)	8	8.26	1.03	1.39
Heat × Strength	6	2.07	0.35	0.47
Strength × Hole (Heat)	16	11.89	0.74	1.00
Error	<u>32</u>	<u>23.72</u>	0.74	
Total	67	926.05		

^aSignificant at the 1% level.

of a variable by the F-statistic. F-statistics are the ratios of the mean squares for the variables to the error mean square. These ratios are compared to the percentage points of the F-distribution. Only heating rate (the HEAT variable) had a significant effect on coke yield with a significance level of 1%. The heating rate variable was then considered as a continuous variable and a quadratic function was fitted to the coke yield measurements using the method of least squares:

$$\text{COKE YIELD} = 39.131 - 1.539 \text{ HEAT} + 0.053 (\text{HEAT})^2 .$$

An estimate of the variance for the quadratic model is the error mean square of $\hat{\sigma}^2 = 0.760$ as shown in the AOV Table 4. Because there are repeated measurements within each heating rate, the quadratic model can be tested to see if there is a significant lack of fit. This test is performed by partitioning the error sum of squares into the sum of squares due to the lack of fit of the model and the sum of squares due to the repeated observations in each heating rate. The lack of fit test in Table 5 shows there is no significant lack of fit at the 5% level, and the quadratic model is an appropriate model for coke yield within the range of heating rates tested. The quadratic prediction model and the 95% confidence interval for the prediction model are plotted in Fig. 8.

Table 4. AOV Table for the Quadratic Coke Yield Model

Source	DF	Sum of Squares	Mean Squares	F-Statistics
Linear	1	789.66	789.66	1039.02 ^a
Quadratic	1	87.00	87.00	114.48 ^a
Error	65	49.39	0.76	
Total	67	926.05		

^aSignificant at the 1% level.

Table 5. Lack of Fit Test for the Quadratic Coke Yield Model

Source	DF	Sum of Squares	Mean Squares	F-Statistic
Lack of Fit	1	2.49	2.50	3.41
Repeated Observations	64	46.90	0.73	
Total	65	49.39		

Fig. 8. Pitch Coke Yield of the In-Block Carbonization-Heat Treating Process is a Function of the Heating Rate of the Carbonization Cycle.

Since an earlier study⁶ found the permeability of fuel element holes to vary significantly, the sixth run (C-204) was made to get a cursory, overall view of the effect of fuel hole permeability (within a range of 4 to 17 liters/min) on coke yield and defective fraction. The run was made at 10°C/min since this is the reference heating rate. The complete data for the run are shown in Appendix B, Table B-2, and summarized in Table 6. The AOV Table 7 for this run shows that the F-value for the permeability source of variation on coke yield is $F = 0.54$, which is well below the 5% percentile of the F-distribution with 3 and 28 degrees of freedom $F(3,28) = 2.96$. Therefore, permeability (within the range tested) is not a significant source of variation for the coke yield measurements.

The experiment showed that the heating rate is the only significant process variable that affects coke yield. The effect of heating rate on coke yield can be represented by a quadratic function, which shows that coke yield decreases as the heating rate increases.

Table 6. Pitch Coke Yield and Defection Fraction of Fuel Particles Versus Fuel Hole Permeability

Fuel Hole Number	Relative Argon Permeability (liters/min)	Mean Pitch ^a Coke Yield (%)	Defective Fraction ^b of Fuel Particles ($\times 10^{-4}$)
17	4.51	29.75	0.034
18	4.12	28.99	c
28	9.82	29.16	0.035
29	9.72	30.23	c
43	13.49	29.82	0.008
44	13.19	30.35	c
57	17.41	29.33	0.219
59	15.76	30.78	c

^aUsing additive type matrix at a heating rate of 10°C/min. Each value listed is an average of four samples.

^bUsing particle batch P₁ (A-637) with a crush strength of 5.14 lb. Each value listed is an average of two samples.

^cNot determined.

Table 7. AOV Table for Permeability
Effect Upon Coke Yield

Source	DF	Sum of Squares	Mean Squares	F-Statistic
Permeability	3	2.74	0.92	0.54 ^a
Error	<u>28</u>	<u>46.64</u>	1.67	
Total	31	49.38		

^aNot significant at 5% level.

Defective Fraction of Fuel Particles

The defective fraction of fuel particles for all the individual fuel rods in the initial four runs is shown in Appendix B, Table B-1. A cursory examination of the table shows the defective fraction approached or exceeded the desired maximum of 1×10^{-4} at some combinations of heating rate and particle crushing strength, but the important point is that the defective fraction was well below the maximum limit at other combinations of these two variables. A summary of the defective fraction data is given in Table 8, which indicates the defective fraction is influenced by both heating rate and particle crushing strength. It should be noted that the defective fraction was well below the desired maximum limit of 1×10^{-4} when using particles with: (1) a crushing strength of 6.85 lb at any heating rate, (2) a crushing strength of 5.78 lb at a heating rate of 5°C/min and (3) a crushing strength of 5.14 lb at a heating rate of 10°C/min or greater.

The defective fraction data had to be transformed to analyze the sources of variations because the data did not follow the assumptions of normality and constant variance required by the analysis of variance procedure. The following responses were examined to see if they satisfied the analysis of variance assumptions: leached thorium weight (WT), defective fraction (F), $\sqrt{F}$, arcsin F, and log (F) (base e). Transformations were judged equivalent if the means, standard deviations, skewness, and kurtosis statistics had about the same values for each heating rate and

Table 8. Defective Fraction^{a,b} of Bisco-Coated HTGR Fertile Particles Processed Using In-Block Carbonization

Fertile Particle Crushing Strength N (lb)	Heating Rate, ^c °C/min Mean Coke Yield, wt %			
	1	5	10	15
	37.8	32.5	29.3	28.0
22.9 (5.14)	1.76	0.78	0.02	0.19
25.7 (5.78)	1.64	0.23	0.01	0.16
30.5 (6.85)	0.01	0.01	0.01	0.01

^aValues listed for the 1°C/min run are an average of five samples, all other values listed are an average of six samples ($\times 10^{-4}$).

^bDetermined by chlorine leach for 2 hr at 1500°C.

^cHeating rate between 300 and 600°C.

crushing strength combination. Since the log (F) transformation seemed to both stabilize the variance and satisfy the normality assumption, the data were analyzed using log (F).

The AOV Table 9 for the sources of variation for log (F) shows that the HEAT, STRENGTH and HEAT $\times$ STRENGTH variables have a significant effect on log (F) at the 5% significance level, but the HOLE variable (horizontal position) is not a significant effect at the 5% significance level. Since there are three missing values in the experiment, the sum of squares are adjusted for the other effects in the AOV model. This adjustment made only minor differences from the unadjusted values. Because of the lack of a significant HOLE (horizontal position) effect, a subsequent analysis was made using just the HEAT, STRENGTH, and HEAT $\times$ STRENGTH sources of variation. This alternate AOV table is given in Table 10.

The meaning of the significant HEAT $\times$ STRENGTH interaction is that the effect of heating rate on defective fraction depends on the particle strength. This interaction is illustrated in Fig. 9, which is a graph of the means of the defective fractions for each crushing strength versus

Table 9. AOV of Log (F) for the Initial Four Tests

Source	DF	Sum of Squares	Mean Squares	F-Statistic
Heat	3	72.48	24.16	12.15 ^a
Strength	2	85.88	42.94	21.59 ^a
Hole (Heat)	8	20.31	2.54	1.28
Heat × Strength	6	32.45	5.41	2.72 ^b
Strength × Hole (Heat)	16	33.95	2.12	1.07
Error	33	65.63	1.99	
Total	68	299.61 ^c		

^aSignificant at 1% level.

^bSignificant at 5% level.

^cUnadjusted sum of squares do not add up to total sum of squares.

Table 10. Alternate AOV of Log (F) for the Initial Four Tests

Source	DF	Sum of Squares	Mean Squares	F-Statistic
Heat	3	70.95	23.65	11.26 ^a
Strength	2	77.78	38.89	18.52 ^a
Heat × Strength	6	31.16	5.19	2.47 ^b
Error	57	119.72	2.10	
Total	68	299.61		

^aSignificant at 1% level.

^bSignificant at 5% level.

the four heating rates. One physical meaning of this effect is that the defective fraction of very strong particles does not change with heating rate.

The initial four runs could not distinguish between the crushing strength and vertical position effect upon defective fraction. The fifth run (C-214 at 1°C/min) was made in which only one particle type, crushing strength of 22.9 N (5.14 lb), was used in all the 18 rods placed in all the horizontal and vertical positions. The defective fraction data for this run are shown in Appendix B, Table B-3 and the

Fig. 9. Mean Defective Fraction versus Heating Rate at Different Crushing Strength Levels Shows Heating Rate Times Crushing Strength Interaction.

mean values of the data are summarized in Table 11. The analysis of this run did verify that both the vertical and the horizontal position effects upon defective fraction were not significant at the 5% significance level. The AOV table using the log of defective fraction is shown in Table 12.

The effect of fuel hole permeability (within a range of 4 to 17 liters/min) upon defective fuel fraction was investigated in the sixth run. The data for this test are given in Appendix B, Table B-2 and the mean values of the defective fraction data at the various permeability levels are shown in Table 6. The analysis of variance showed the permeability had no significance on the log (F) defective fraction at the 5% significance level. This lack of effect is shown in AOV Table 13.

Table 11. Defective Fraction^a of Fuel Particles^b versus Horizontal and Vertical Position

Vertical Position	Horizontal Position		
	Hole 1	Hole 2	Hole 3
Top	0.65	3.58	1.88
Middle	5.05	0.84	1.92
Bottom	4.08	1.18	1.63

^a $\times 10^{-4}$.

^b Particle Batch P₁ (A-637) with a crushing strength of 22.9 N (5.14 lb) carbonized at a heating rate of 1°C/min in Run C-214 with a mean pitch coke yield of 38.0%.

Table 12. AOV for Log (F) to Test the Effects of Horizontal and Vertical Position

Source	DF	Sum of Squares	Mean Squares	F Statistic
Hole ^a	2	10.04	5.02	1.17 ^c
Location ^b	2	0.31	0.15	0.03 ^c
Hole ^a $\times$ Location ^b	4	20.34	5.09	1.18 ^c
Error	9	38.70	4.30	

^a Horizontal position.

^b Vertical position.

^c Not significant at the 5% significance level.

Table 13. AOV Table for Log (F) to Test the Permeability Effect

Source	DF	Sum of Squares	Mean Squares	F-Statistic
Permeability	3	3.91	1.30	0.47 ^a
Error	4	11.01	2.75	
Total	7	14.92		

^a Not significant at 5% level.

To obtain the defective fraction within a known confidence level, both the total number of defective particles found and the total number of particles examined must be known, not just the ratio. If it is required that the defective fraction be known with a 95% confidence, the graph shown in Fig. 10 can be used.¹² If a point representing the number of defective particles found versus total number of particles examined is below the shaded area, there is 95% confidence that the defective fraction is less than 1×10^{-4} . Conversely, if the point is above the shaded area, there is, 95% confidence that the defective fraction is greater than 1×10^{-4} . If the point is within the shaded area, it is not known with 95% confidence, if the defective fraction is less than, equal to, or greater than 1×10^{-4} .

The points in Figure 10 are equivalent to the defective fractions shown in Table 8. The number of particles examined was the average number of particles per rod (see Table 1) times the number of rods sampled (see Table 3). The number of defective particles in a rod was calculated as

Fig. 10. Confidence Limits for Fuel Defective Fraction.

mentioned earlier. A calculated fractional particle of 1/4 or more was considered a fuel particle. Note that the two points representing 1°C/min with particle crushing strengths of 5.15 and 5.78 lb are above the shaded area and thus there is 95% confidence that the defective fraction is greater than 1×10^{-4} . The point representing 5°C/min with a particle crushing strength of 5.14 lb is in the shaded area and thus it is not known if the failure is greater than or less than 1×10^{-4} . All other points are below the shaded area and thus we are 95% confident that the defective fraction is less than 1×10^{-4} . In other words, if the reference in-block carbonization process operated at 10 to 15°C/min, an acceptable defective fraction would be obtained. Even if the heating rate were reduced to as low as 5°C/min, an acceptable defective fraction would be obtained with particles having a crushing strength of 25.7 N (5.78 lb) or greater.

Matrix Microstructure and Porosity

Three metallographic samples were taken from each of the four initial carbonization tests and the data for the 12 samples are listed in Appendix B, Table B-4. The samples from each test included one of each of the three particle types with samples representing the top, middle, and bottom vertical positions. The samples were visually examined at magnification levels of 40×, 250×, and 750×. Visual examination at all magnification levels showed no readily discernable differences among the samples. A representative sample from each of the four runs is shown in Fig. 11. The similarity of the microstructure among the samples is apparent. Although one to seven defective particles per rod were found by the chlorine leach technique, none were found in the metallographic samples, including the examination at 750×. This was not surprising since the metallographic mount examines a plane through the rod and sees only a limited number of the fuel particles. The number of defective particles would have to be much higher than the maximum permitted (essentially one per rod) before detection by metallographic means would be possible. By contrast, the chlorine leach technique evaluates all

Fig. 11. Matrix Microstructure of In-Block Carbonized HIGR Fuel Rods Using Additive-Type Matrix. (a) Heating rate, $1^{\circ}\text{C}/\text{min}$, mean coke yield, 37.8%. (b) Heating rate, $5^{\circ}\text{C}/\text{min}$, mean coke yield 32.5%. (c) Heating rate, $10^{\circ}\text{C}/\text{min}$, mean coke yield, 29.3%. (d) Heating rate, $15^{\circ}\text{C}/\text{min}$, mean coke yield 28.0%.

the particles in the rod and was able to detect as little as an individual particle. Indeed, this was the prime reason for developing the chlorine leach technique.

The porosity in the fuel rod matrix is basically of two different types: gross, large pores equal to or greater in size than the fuel particles, and much smaller pores that tend to be uniform in size and distribution. The large pores, called macropores can be the result of gross flaws in the fuel rod such as large gas bubbles. The finer microporosity is associated with the relatively uniform decomposition of the pitch. The microporosity might be expected to correlate with pitch coke yield, which in turn is determined by the heating rate. In this study, macropores are defined as pores larger than 50 μm and microporosity includes all smaller pores.

The micro- and macroporosity were quantitatively measured on an automatic television image analyzer.* The macroporosity was measured at 100 $\times$ and the microporosity at 870 $\times$. In each case 10 determinations were made along the rod. Increasing the number of determinations to 20 did not significantly improve the precision of the data. The porosity data for the twelve metallographic samples are shown in Appendix B, Table B-4. As the pitch coke yield of the matrix decreases, the porosity of the matrix must increase. Thus, a positive correlation between coke yield and heating rate was expected. Although there are variations in the data, generally the porosity values increased as the heating rates increased (decrease in coke yield) as shown in Fig. 12 for macroporosity and Fig. 13 for microporosity. However, as shown in the figures, the porosity values, unexpectedly, did not consistently increase with increases in heating rate. The reasons for this anomaly in the data are not understood.

CONCLUSIONS

The most important conclusion that can be drawn from this work is that the in-block carbonization process can produce acceptable HTGR

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*Quantimet Model 720

ORNL-DWG 77-17669

Fig. 12. Matrix Macroporosity versus Heating Rate.

ORNL-DWG 77-17667

Fig. 13. Matrix Microporosity versus Heating Rate.

fuel rods. The rods will have the desired pitch coke yield and the defective fraction of fuel particles will be below the desired maximum limit of 1×10^{-4} . The above was accomplished using Biso-coated fertile particles and the reference General Atomic matrix at the proposed heating rate of $10^{\circ}\text{C}/\text{min}$. In addition, the study showed that the heating rate and the particle crushing strength could vary considerably and the process could still produce an acceptable product. However, at combinations of low heating rates and low particle crushing strength, an unacceptable number of defective particles (due to matrix-particle interactions) could be produced.

Other conclusions include:

1. The pitch coke yield was controlled by the heating rate of the carbonization cycle.
2. Within the range studied, none of the other variables tested (particle strength, horizontal position, vertical position, and fuel hole permeability) had a significant effect on the coke yield.
3. The defective fraction of fuel particles was controlled by the heating rate and particle crushing strength.
4. Within the range studied, none of the other variables tested (horizontal position, vertical position, and fuel hole permeability) had a significant effect on the defective fraction of fuel particles.

RECOMMENDATIONS FOR FUTURE WORK

The conclusions drawn from this study are for fertile particles. As analytical techniques are established for evaluating fissile particles, a similar investigation should be made for fissile particles. Naturally, the conclusions of any investigation are valid only under the conditions tested. As process materials, process conditions, and analytical techniques for fertile particles are significantly changed, the investigations should be redone to confirm the validity of the previous conclusions. The study of matrix microstructure and porosity should be continued in an attempt to derive a useful quantitative parameter that does not require any prior history (particle weight, rod weight, etc.) of the fuel rod.

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APPENDIX A

STATISTICAL ANALYSIS OF THE DATA

Introduction

HTGR fuel particles of three different strengths were used to make fuel rods which were placed in four graphite blocks. Each of these graphite blocks was heated at a different rate to a final processing temperature. An experimental design was constructed to examine the effects of different particle crushing strengths, horizontal and vertical positions within the block, and heating rates on the response of coke yield and the defective fraction of fuel particles in a fuel rod.

Analysis of the Data

The sources of variation for an experimental measurement or a response can be represented by an analysis of variance (AOV) model. The sources of variation are identified by using the following notation:

$(\text{HEAT})_i$ = i th heating rate; $i = 1, 2, 3, 4$;

$(\text{STRENGTH})_j$ = j th fuel particle strength, $j = 1, 2, 3$;

$\text{HOLE}(\text{HEAT})_{ki}$ = k th hole (horizontal position) *nested* within the i th heat, $k = 1, 2, 3$;

$(\text{HEAT} \times \text{STRENGTH})_{ij}$ = i th heat and j th fuel particle strength interaction;

$[\text{STRENGTH} \times \text{HOLE}(\text{HEAT})]_{jki}$ = j th fuel particle strength interaction with the k th hole (horizontal position) *nested* within the i th heat;

$(\text{ERROR})_{ijkh}$ = h th random error in the ijk th cell, $h = 1, 2, \dots, n_{ijk}$.

The random error variables are assumed to be independent and identically distributed as a normal distribution with zero mean and constant variance.

The AOV model for a response is:

$$\begin{aligned} \text{RESPONSE} = & \text{MEAN} + (\text{HEAT})_i + (\text{FUEL})_j + (\text{HOLE}(\text{HEAT}))_{ij} + (\text{HEAT} \times \text{FUEL})_{ij} \\ & + [\text{FUEL} \times \text{HOLE}(\text{HEAT})]_{jki} + (\text{ERROR})_{ijkh} . \end{aligned}$$

The parameters in the AOV model are estimated to minimize the sum of the squared error terms. The adjusted total sum of squares [i.e., $\Sigma(\text{RESPONSE} - \text{MEAN})^2$] can then be partitioned into sum of squares corresponding to each source of variation.

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Table B-1. Coke Yield and Defective Particle Fraction Data
for Runs C-200, -201, -202, and -203

Vertical Position	Horizontal Position											
	Hole 1				Hole 2				Hole 3			
	Par- ticle Batch	Fuel Rod Number	Coke Yield (%)	Defective Fraction ($\times 10^{-4}$)	Par- ticle Batch	Fuel Rod Number	Coke Yield (%)	Defective Fraction ($\times 10^{-4}$)	Par- ticle Batch	Fuel Rod Number	Coke Yield (%)	Defective Fraction ($\times 10^{-4}$)
<u>Run C-203, 1°C/min</u>												
Top-1	P ₁	M152-264	a	a	P ₃	M152-257	38.66	0.01	P ₂	M152-277	38.54	0.79
Top-2	P ₁	M152-265	38.25	5.24	P ₃	M152-258	27.03 ^b	0.02	P ₂	M152-278	37.48	2.37
Middle-1	P ₂	M152-273	38.21	0.02	P ₁	M152-266	38.44	2.08	P ₃	M152-259	a	a
Middle-2	P ₂	M152-274	37.74	4.25	P ₁	M152-267	38.57	0.74	P ₃	M152-260	37.03	0.01
Bottom-1	P ₃	M152-255	37.91	0.01	P ₂	M152-275	37.76	0.79	P ₁	M152-268	36.54	0.01
Bottom-2	P ₃	M152-256	35.51	0.01	P ₂	M152-276	a	a	P ₁	M152-269	38.24	0.73
<u>Run C-202, 5°C/min</u>												
Top-1	P ₁	M152-172	33.24	0.75	P ₃	M152-228	34.23	0.01	P ₂	M152-203	33.07	0.78
Top-2	P ₁	M152-173	32.80	0.01	P ₃	M152-229	32.74	0.01	P ₂	M152-204	33.17	0.35
Middle-1	P ₃	M152-226	32.61	0.01	P ₂	M152-201	32.30	0.02	P ₃	M152-230	29.76	0.01
Middle-2	P ₃	M152-227	33.37	0.01	P ₂	M152-202	32.81	0.06	P ₃	M152-231	32.46	0.01
Bottom-1	P ₂	M152-199	32.91	0.02	P ₁	M152-174	32.46	0.02	P ₁	M152-176	31.46	1.48
Bottom-2	P ₂	M152-200	32.57	0.17	P ₁	M152-175	30.44	1.68	P ₁	M152-177	32.79	0.77

Table B-1. (Continued)

Vertical Position	Horizontal Position											
	Hole 1				Hole 2				Hole 3			
	Par-ticle Batch	Fuel Rod Number	Coke Yield (%)	Defective Fraction ($\times 10^{-4}$)	Par-ticle Batch	Fuel Rod Number	Coke Yield (%)	Defective Fraction ($\times 10^{-4}$)	Par-ticle Batch	Fuel Rod Number	Coke Yield (%)	Defective Fraction ($\times 10^{-4}$)
Run C-200, 10°C/min												
Top-1	P ₃	M152-235	29.44	0.01	P ₁	M152-183	30.38	0.01	P ₁	M152-185	28.82	0.01
Top-2	P ₃	M152-236	29.53	0.01	P ₁	M152-184	28.96	0.02	P ₁	M152-186	28.59	0.01
Middle-1	P ₂	M152-208	29.26	0.01	P ₃	M152-237	28.91	0.00	P ₂	M152-212	29.19	0.02
Middle-2	P ₂	M152-209	28.87	0.01	P ₃	M152-238	29.00	0.00	P ₂	M152-213	30.06	0.02
Bottom-1	P ₁	M152-181	29.62	0.02	P ₂	M152-210	29.67	0.00	P ₃	M152-239	30.66	0.01
Bottom-2	P ₁	M152-182	28.65	0.03	P ₂	M152-211	29.20	0.00	P ₃	M152-240	28.72	0.01
Run C-201, 15°C/min												
Top-1	P ₃	M152-244	28.79	0.01	P ₁	M152-192	28.14	0.05	P ₁	M152-194	28.45	0.04
Top-2	P ₃	M152-245	25.76	0.00	P ₁	M152-193	28.07	0.01	P ₁	M152-195	27.78	0.02
Middle-1	P ₁	M152-190	26.19	0.01	P ₃	M152-246	28.60	0.02	P ₃	M152-248	28.62	0.01
Middle-2	P ₁	M152-191	27.61	1.02	P ₃	M152-247	29.05	0.02	P ₃	M152-249	28.30	0.01
Bottom-1	P ₂	M152-217	27.30	0.01	P ₂	M152-219	28.39	0.04	P ₂	M152-221	28.02	0.01
Bottom-2	P ₂	M152-218	28.20	0.00	P ₂	M152-220	28.06	0.85	P ₂	M152-222	28.35	0.03

^a Indicates a missing value due to difficulties in unloading the fuel rods.

^b A statistical test showed this point to be an outlier and was deleted from the analysis of the coke yield data.

Table B-2. Pitch Coke Yield and Defective Fraction Data
versus Fuel Hole Permeability for
Carbonization Run C-204^a

Fuel Hole Number	Relative Argon Permeability (liters/min)	Fuel Rod Number	Pitch Core Yield (%)	Defective Fraction ^b Fuel Particles ($\times 10^{-4}$)
17	4.51	M152-284	27.87	0.046
		M152-285	28.85	0.022
		M152-286	30.76	
		M152-287	31.50	
18	4.12	M152-290	28.17	
		M152-291	28.16	
		M152-292	28.53	
		M152-293	31.09	
28	9.82	M152-296	29.45	0.053
		M152-315	29.23	0.016
		M152-316	30.86	
		M152-317	27.10	
29	9.72	M152-320	29.18	
		M152-321	30.29	
		M152-322	30.96	
		M152-323	30.47	
43	13.49	M152-326	29.96	0.006
		M152-327	30.86	0.009
		M152-328	30.86	
		M152-329	27.60	
44	13.19	M152-332	29.98	
		M152-333	29.67	
		M152-334	30.48	
		M152-335	31.25	
57	17.41	M152-338	29.63	0.005
		M152-339	30.34	0.432
		M152-340	28.68	
		M152-341	28.67	
59	15.76	M152-344	30.58	
		M152-345	30.73	
		M152-346	32.41	
		M152-347	29.38	

^aCarbonized at a heating rate of 10°C/min using the additive type matrix.

^bUsing particle batch P₁ (A-637) with a crushing strength of 5.14 lb.

Table B-3. Defective Fraction Data versus
Horizontal and Vertical Positions for
Carbonization Run C-24^a

Vertical Position	Horizontal Position					
	Hole 1		Hole 2		Hole 3	
	Fuel Rod Number	Defective Fraction ($\times 10^{-4}$)	Fuel Rod Number	Defective Fraction ($\times 10^{-4}$)	Fuel Rod Number	Defective Fraction ($\times 10^{-4}$)
Top-1	M152-348	0.58	M152-354	0.38	M152-360	0.03
Top-2	M152-349	0.72	M152-355	6.78	M152-361	3.72
Middle-1	M152-350	7.27	M152-356	0.12	M152-362	0.01
Middle-2	M152-351	2.82	M152-357	1.56	M152-363	3.82
Bottom-1	M152-352	2.21	M152-358	0.03	M152-364	0.33
Bottom-2	M152-353	5.94	M152-359	2.33	M152-365	2.92

^aCarbonized at a heating rate of 1°C/min using additive type matrix at a mean pitch coke yield of 38.0% and using particle batch P₁ (A-637) with a crushing strength of 5.14 lb.

Table B-4. Matrix Macroporosity and Microporosity Data for Carbonization
Runs C-200, -201, -202, and -203

Run Number	Heating Rate (°C/min)	Mean Pitch ^a Coke Yield (%)	Fuel Rod Number	Rod Pitch Coke Yield (%)	Vertical Position	Particle ^b Batch	Porosity (% of Matrix)		
							Micro	Macro	Total
C-203	1	37.8	M152-264	41.5	Top	P ₁	30.4	38.2	68.6
			M152-276	36.8	Bottom	P ₂	31.3	36.7	68.0
			M152-259	37.3	Middle	P ₃	30.3	37.9	68.2
C-202	5	32.5	M152-234	32.55	Bottom	P ₃	31.5	33.4	64.9
			M152-180	31.79	Middle	P ₁	35.3	32.3	67.6
			M152-207	32.35	Top	P ₂	36.7	35.0	71.7
C-200	10	29.3	M152-243	29.12	Bottom	P ₃	36.4	35.1	71.5
			M152-189	28.28	Middle	P ₁	36.8	46.3	83.1
			M152-216	29.41	Top	P ₂	34.5	40.7	75.2
C-201	15	28.0	M152-224	27.99	Top	P ₂	32.1	41.7	73.8
			M152-251	27.36	Middle	P ₃	33.0	45.0	78.0
			M152-198	25.64	Bottom	P ₁	33.2	38.9	72.1

^aFrom Table 2.

^bP₁ = A-637; P₂ = A-642; P₃ = A-635.

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