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**Influence of Process Variables
on Permeability and Anisotropy
of Biso-Coated HTGR Fuel Particles**

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Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
Price: Printed Copy \$4.50 ; Microfiche \$3.00

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ORNL/TM-6087
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Contract No. W-7405-eng-26

METALS AND CERAMICS DIVISION

THORIUM UTILIZATION PROGRAM (189a OH045)

Refabrication Development — Task 300

INFLUENCE OF PROCESS VARIABLES ON PERMEABILITY AND ANISOTROPY OF
BISO-COATED HTGR FUEL PARTICLES

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Date Published - November 1977

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D. P. Stinton, W. J. Lackey, and B. A. Thiele*

ABSTRACT

The effect of several important process variables on the fraction of defective particles and anisotropy of the low-temperature isotropic (LTI) coating layer has been determined for Biso-coated HTGR fuel particles. Process variables that were considered are deposition temperature, hydrocarbon type, diluent type, and percent diluent. The effect of several other variables such as coating rate and density that depend on the process variables were also considered in this analysis. The fraction of defective particles was controlled by the dependent variables coating rate and LTI density. Coating rate was also the variable controlling the anisotropy of the LTI layer. Diluent type and diluent concentration had only a small influence on the deposition rate of the LTI layer. High-quality particles in terms of anisotropy and permeability can be produced by use of a porous plate gas distributor if the coating rate is between 3 and 5 $\mu\text{m}/\text{min}$ and the coating density is between about 1.75 and 1.95 g/cm^3 .

INTRODUCTION

Coated microspheres less than 1 mm in diameter are being developed as fuel for use in High-Temperature Gas-Cooled Reactors. These particles act as miniature fuel elements containing fissile or fertile material. The reference recycle fertile particles consist of coated thorium kernels. They are first coated with a low-density pyrolytic carbon buffer layer, which provides void volume for gaseous fission products and protects outer coatings from recoiling fission fragments. Following the buffer coating is a high-density pyrolytic carbon layer referred to as the low-temperature isotropic (LTI) (Fig. 1). The LTI coating of Biso particles acts as a pressure vessel to retain gaseous fission products and provides mechanical strength necessary for fuel rod molding and pneumatic

*On assignment from KFA, Jülich.

Y-134525

BISO ThO₂ FERTILE

TRISO RESIN FISSILE

Fig. 1. Reference HTGR Fuel Particles.

transfer. Uranium-bearing fissile fuel particles are coated with these same carbon layers plus silicon carbide (Fig. 1), which is the principal barrier to fission product escape, and a final outer layer of LTI coating.

The permeability and anisotropy of LTI coatings are important for both the fertile and fissile particles. If fission products are to be contained in Biso-coated thorium-bearing fuel, LTI coatings must remain intact and impermeable during the in-reactor lifetime of the particles. For Triso-coated fissile particles, the permeability of the inner LTI layer must not be excessive since this would allow HCl present during the SiC coating operation to come into contact and react with the kernel. Also for Triso particles, the permeability and anisotropy of the inner and outer LTI layers likely influence irradiation performance. Thus, because of the importance of the LTI layer, an experiment was conducted to examine the effect of important process variables on LTI coating permeability and anisotropy. Several other coating properties such as shape ratio, crushing strength, density, and porosity were also measured for each coating run, and these results are summarized.

EXPERIMENTAL PROCEDURE

Process variables studied in this statistically designed experiment were deposition temperature (1225 and 1350°C), type of hydrocarbon (propylene vs an acetylene-propylene mixture), type of diluent (Ar, He, H₂), and the diluent concentration (50 and 67 vol %). The effect of numerous dependent variables that are controlled by the process variables was also determined. These dependent variables are coating rate, coating thickness, LTI immersion density, percent open porosity, and immersion density corrected for open porosity.¹ The experimental plan consisted of 24 combinations of the above process variables (see Fig. 2). These variables were chosen because they were known to influence numerous coating properties. Properties examined were anisotropy, fraction defective, efficiency of the coating process, shape ratio, crushing strength, density, and porosity. Property measurement techniques are described later in this report or have been previously reported.¹

Fig. 2. Experimental Plan.

Coatings in this experiment were deposited in a 13-cm-diam fluidized bed coating furnace. This furnace has been operating for about ten years and has been described previously.² A contoured porous carbon plate referred to as a frit was used as the gas distributor for these runs. Figure 3 shows a sketch of the frit, which has also been described elsewhere.^{3,4} Coating gas enters the furnace and passes through the thinned regions of the porous carbon, producing very uniform fluidization of particles.

Fig. 3. Porous Plate Gas Distributor.

All coatings in this experiment were deposited onto 2.2-kg batches of 500- μ m-diam thoria kernels. Each batch was carefully riffled from one large batch of material. All coatings consisted of a buffer layer immediately followed by one of the LTI layers specified in Fig. 2. All

buffer layers were produced under identical coating conditions (1350°C, 142 liters/min C₂H₂, 85 liters/min Ar) to yield a buffer coating thickness of about 80 μm . In order to compare properties of LTI layers, their thicknesses must be similar. The desired LTI thickness was $75 \pm 15 \mu\text{m}$, which in general was obtained by adjustment of the coating time. Several coatings do not have the correct thickness (Table 1) because the particular coating conditions were found to be undesirable, so no attempt was made to obtain the desired LTI thickness.

RESULTS AND DISCUSSION

Many properties of the LTI coating layers were studied in relation to the process and dependent variables. These properties are discussed individually below.

Anisotropy

To survive irradiation, LTI coatings must be very isotropic. Anisotropic dimensional changes, which occur during irradiation, cause high stresses and cracking in anisotropic coatings. Anisotropy was measured optically with a Seibersdorf unit and expressed in terms of the Bacon Anisotropy Factor (BAF_0). Anisotropy values are given in Table 1 for each coating run. Completely isotropic coatings would have a BAF_0 value of 1.00; BAF_0 values increase as anisotropy increases.

A statistical multiple regression analysis was performed on all the BAF_0 data in an attempt to determine whether correlations exist between the four coating variables under study and BAF_0 . Such analyses were also performed to determine if coating rate, coating thickness, immersion density, percent open porosity, and immersion density corrected for open porosity correlated with anisotropy. These analyses showed that only coating rate, hydrocarbon type, and deposition temperature influenced BAF_0 . Coating rate was the most important variable in agreement with previous work:⁴ it was significant at the 99% confidence level. To show its importance, BAF_0 is plotted against coating rate in Fig. 4. The BAF_0 decreased very rapidly as coating rate increased up to about 3 $\mu\text{m}/\text{min}$. As coating rate increased above 3 $\mu\text{m}/\text{min}$, BAF_0 decreased gradually to a

Table 1. Characterization Data for All Coating Runs

	Hydrocarbon Type	Diluent	Deposition Temperature (°C)	EAF ₀	Fraction Defective by Cl ₂ Leach	Ne/He Ratio	Fraction Defective by R/B	Efficiency (%)	Shape Ratio	Coating Thickness (μm)	Crushing Strength		Immersion Density (g/cm ³)	Immersion Density Corrected for Percent Porosity (g/cm ³)	Open Porosity (%)	Coating Rate (μm/min)
											(N)	(lb)				
A-747	Propylene	50% He	1225	1.474	1.9×10^{-3}	0.15	2×10^{-5}	11.26	1.087	25.3	19.4	4.37	1.9481	1.8603	4.507	1.17
A-754	Propylene	67% H ₂	1250	1.226	1.1×10^{-3}	0.21	9.2×10^{-6}	0.12	1.175	31.6	28.9	6.49	1.9553	1.8752	4.098	0.35
A-758	Mixed	67% He	1350	1.027	1.0×10^{-4}	0.48	5×10^{-6}	39.45	1.098	81.1	32.2	7.22	1.7554	1.6635	5.237	4.92
A-762	Mixed	67% He	1225	1.043	1.3×10^{-5}	0.36	$<1 \times 10^{-6}$	42.19	1.119	71.4	26.4	5.94	1.9620	1.9040	2.956	3.25
A-764	Propylene	67% He	1350	1.033	4.9×10^{-6}	0.35	$<1 \times 10^{-6}$	37.91	1.111	92.0	30.8	6.92	1.8635	1.8109	2.824	3.29
A-765	Propylene	50% Ar	1350	1.035	1.8×10^{-5}	0.34	$<2 \times 10^{-6}$	35.87	1.110	78.6	31.1	6.99	1.9153	1.8580	2.994	3.14
A-766	Mixed	50% H ₂	1350	1.024	8.3×10^{-6}	0.51	$<2 \times 10^{-6}$	58.84	1.088	66.0	29.6	6.65	1.7638	1.6722	5.195	4.78
A-768	Propylene	50% H ₂	1225	1.343	2.1×10^{-3}	0.13	2×10^{-6}	3.86	1.093	44.2	25.9	5.82	2.0046	1.9568	2.386	0.61
A-770	Propylene	67% Ar	1225	1.107	5.7×10^{-4}	0.15	3×10^{-6}	15.09	1.099	93.6	28.9	6.49	2.0120	1.9919	1.001	1.25
A-771	Mixed	67% H ₂	1350	1.030	5.0×10^{-6}	0.44		51.47	1.129	69.0	19.6	4.40	1.6981	1.6194	4.631	4.18
A-772	Mixed	67% Ar	1350	1.031	2.0×10^{-5}	0.66		53.10	1.150	72.7	23.0	5.17	1.6934	1.5929	5.935	4.41
A-775	Mixed	50% Ar	1350	1.030	6.4×10^{-4}	0.7	7×10^{-3}	60.76	1.141	79.1	23.1	5.19	1.7290	1.5578	9.903	4.79
A-779	Propylene	50% Ar	1225	1.055	5.7×10^{-5}	0.19			1.097	125.9	39.3	8.84	2.0191	1.992	1.32	2.10
A-780	Propylene	50% He	1350	1.022	4.2×10^{-6}	0.37	$<2 \times 10^{-6}$	44.53	1.098	78.2	28.0	6.30	1.8644	1.7726	4.923	4.03
A-782	Propylene	50% H ₂	1350	1.026	5.2×10^{-5}	0.27	$<1 \times 10^{-6}$	32.53	1.088	88.4	32.3	7.26	1.8546	1.8096	2.428	2.95
A-783	Mixed	50% He	1350	1.023	7.9×10^{-4}	0.60	2×10^{-3}	68.14	1.097	86.3	24.4	5.47	1.7322	1.5295	11.701	5.95
A-784	Mixed	50% He	1225	1.029	1.5×10^{-6}	0.43	$<3 \times 10^{-6}$	62.76	1.088	88.9	31.2	7.00	1.9289	1.8418	4.516	4.44
A-785	Mixed	67% Ar	1225	1.035	8.6×10^{-7}	0.32	$<1 \times 10^{-6}$	38.84	1.129	76.3	24.8	5.56	1.8717	1.8152	3.020	3.05
A-786	Mixed	50% Ar	1225	1.030	8.9×10^{-7}	0.36	$<2 \times 10^{-6}$	50.71	1.115	88.0	31.3	7.03	1.8369	1.8258	3.750	3.67
A-787	Propylene	67% H ₂	1350	1.031	1.5×10^{-5}	0.35	4×10^{-6}	27.85	1.107	80.5	28.2	6.34	1.7381	1.6910	2.713	2.68
A-803	Propylene	67% Ar	1350	1.017	4.2×10^{-5}	0.43	$<4 \times 10^{-6}$	25.59	1.138	71.3	18.0	4.04	1.7984	1.7280	3.920	2.46
A-804	Propylene	67% He	1225	1.055	4.4×10^{-5}	0.27		27.68	1.123	69.7	19.6	4.39	1.9873	1.9313	2.819	2.25
A-806	Mixed	67% H ₂	1225	1.056	1.1×10^{-6}	0.38	$<2 \times 10^{-6}$	39.15	1.151	70.6	21.9	4.93	1.9426	1.8780	3.326	2.82
A-807	Mixed	50% H ₂	1225	1.032	5.8×10^{-5}	0.28	$<2 \times 10^{-6}$		1.135	98.1	25.9	5.82	1.9212	1.8671	2.818	3.63

Fig. 4. Correlation of BAF₀ with Average Coating Rate.

value of about 1.023. The analysis shows that some of the variation about this curve was caused by hydrocarbon type and deposition temperature. Both these variables were statistically significant at the 99% confidence level. On the average, propylene-derived coatings had BAF_O values that were about 0.005 units lower than similar coatings made with a mixture of propylene and acetylene. Higher deposition temperatures produced lower BAF_O values in agreement with previous results.⁴ However, the effect of temperature was quite small (about -0.002 BAF_O units/ 100°C).

Previous studies⁵ have indicated that coatings with BAF_O values above about 1.05 have a high probability of failure during irradiation. Note that BAF_O values below this limit can be obtained if coatings are deposited at rates above about $3\text{ }\mu\text{m/min}$.

Fraction of Defective Particles

Since the purpose of the LTI layer for Biso-coated particles is to retain gaseous fission products, it must be intact and impermeable at the start of irradiation. The present HTGR specifications state that there must be no more than one defective particle in 10^4 at the beginning of irradiation. Defective particles are defined as having either cracked or permeable LTI coatings. This implies that the fraction defective after coating must be lower since subsequent operations (particle transfer, fuel rod molding, fuel rod transfer, and fuel rod carbonization and annealing) might damage additional particles. Presently three techniques are used after coating to measure the number of defective particles. These are hot chlorine leach, Ne/He permeability, and fission gas release. These techniques and the results obtained from each technique will be discussed individually below.

With the chlorine leach technique,⁶ particles are exposed to chlorine gas at 1500°C for 2 hr. The chlorine penetrates cracked or permeable coatings and reacts with the thorium kernels. Volatile heavy metal chlorides escape from the particles and are collected downstream from the furnace and quantitatively analyzed for thorium. Fraction defective is therefore the ratio of the heavy metal released to the heavy metal inventory in the sample. Chlorine leach results are shown in Table 1.

A multiple regression analysis of the chlorine leach results with all the variables showed that only coating rate correlated with chlorine leach (Fig. 5). Surprisingly, density and open porosity did not effect the chlorine leach value. The statistical analysis showed that coating rate was significant at the 99% confidence level, but there was a large amount of unexplained variation (see Fig. 5).

The variation in the data on Fig. 5 can be explained by two mechanisms. A visual examination showed that the high fraction defectives at low coating rates ($< \text{about } 2 \text{ } \mu\text{m}/\text{min}$) were caused by cracked LTI layers. These coatings were deposited with a high degree of order of the pyrocarbon crystallites and therefore cracked when cooled from the coating temperature down to room temperature. This behavior has been previously observed.⁷ As coating rate increases to about $2 \text{ } \mu\text{m}/\text{min}$, the degree of order is no longer very high and the coatings do not crack on cooling. A second mechanism explains why the fraction defective increases at coating rates above about $5 \text{ } \mu\text{m}/\text{min}$. As the degree of order of the crystallites decreases (coating rate increases), coatings become permeable because of interconnected porosity between the pyrocarbon crystallites. Between coating rates of about 2 and $5 \text{ } \mu\text{m}/\text{min}$ (fraction defectives $< \text{about } 1 \times 10^{-4}$), it is not clear what causes the large amount of variation in fraction defective.

Coating permeability can also be measured with the Ne/He technique.⁸ Coated particles are annealed at 1375°C for 1 hr in an atmosphere of one-half helium and one-half neon. During annealing helium penetrates the LTI coating and fills voids within the buffer coating, whereas the larger neon atoms penetrate with greater difficulty. Therefore the neon buildup within the particle depends on coating permeability. After annealing, particles are individually crushed in a vacuum and the amounts of neon and helium released are recorded by a mass spectrometer. As discussed above, coatings applied at very low deposition rates are subject to cracking.

A cracked particle is not included in the reported Ne/He ratio because it yields no reading. Neon and helium can pass into and out of the particle through the crack so that when the particle is broken in the mass spectrometer no more gas escapes. Therefore the Ne/He ratio is

Fig. 5. Correlation of Coating Rate with Fraction Defective.

a measure only of permeability and ignores any cracked particles. Low Ne/He ratios (about 0.10) represent nearly impermeable coatings. Ratios of Ne/He are shown in Table 1. A statistical analysis of all the data showed the Ne/He ratio to depend on coating rate and density both at the 99% confidence level. The Ne/He ratio increases as coating rate increases (Fig. 6). A portion of the variation about the line in Fig. 6 is caused by coating density. High densities cause the Ne/He ratio to decrease. An increase in the immersion density of 0.1 g/cm^3 above the mean causes a decrease of about 0.05 in the Ne/He ratio.

Fig. 6. Correlation of Neon per Helium Ratio with Average Coating Rate.

A third technique to determine the fraction of defective particles is the fission gas release-to-birth ratio (R/B) for krypton and xenon during short irradiations. Release-to-birth ratios of many particle batches in this experiment were measured by General Atomic Company

personnel (Table 1). The technique is quite expensive, so not all the batches were measured. Results show that R/B ratio depends on coating rate (Fig. 7) similarly to the chlorine leach results. Results from other studies are included in Fig. 7 to verify the increase in R/B at high coating rates. High coating rates have high fraction defectives, but low coating rates have R/B values only slightly above average. Notice that R/B values are lower by a factor of about 10 than the chlorine leach result. This is because a portion of the fission gases is retained by the kernel even in a defective particle. Also the R/B technique is insensitive to marginally permeable coatings because of the low number of fissions produced.

A combination of these techniques can be used to determine the fraction of cracked or permeable coatings before irradiation. However, it is not known at this time which technique if any is most useful in predicting performance during long-term irradiation. The irradiation stability of eight particle batches from this study is currently being tested in irradiation test capsule HT-34. Results from this test will help indicate which coating property is the best indicator of particle performance.

Efficiency

During the coating operation, not all the carbon present in the hydrocarbon gas ends up as coating on the particles. Some of the gas leaves the furnace without decomposing or after only partial decomposition. Also some carbon deposits on the frit and furnace liner. The term efficiency, as used here, refers to the percentage of carbon in the input hydrocarbon gas that ends up as carbon on the particles. Efficiency is important to the coating process from the viewpoint of economics, frequency of replacement of coater components, and quantity of soot that must be from the effluent gas. Efficiency in this study ranged from nearly 0 up to about 70% (Table 1). An analysis of the data showed that all process variables (temperature, hydrocarbon type, diluent type, and percent diluent) affected efficiency. Mixed gas produced coatings at efficiencies nearly 25% higher than propylene. The second

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Fig. 7. Correlation of Coating Rate with Fraction Defective.

most important variable was deposition temperature. Coatings were produced about 15% more efficiently at 1350°C than at 1225°C. The type diluent also affected efficiency; helium was found to be the most efficient. It was nearly 10% more efficient than hydrogen. Argon was the next most efficient diluent; it was nearly 6% more efficient than hydrogen. The percent diluent also affects efficiency: 50% diluent produced about 8% higher efficiencies than the 67% diluent.

Other Properties

Coated particles ideally should be nearly spherical to enhance irradiation performance. Particles become faceted during coating because carbon is deposited unevenly around the particle. An index termed "shape ratio" has been developed to measure the sphericity of coated particles.⁹ The shape ratio is obtained for each particle by dividing the coating thickness on one side of a particle by the thickness on the opposite side. The particle shape for a batch is characterized by averaging 50 individual ratios.

Shape ratios were measured for all the coatings in this experiment (Table 1). A multiple regression analysis performed on all the data showed that percent diluent was the only variable to influence shape ratio at the 95% confidence level. Lower flow rates of diluent produced more spherical particles. Unfortunately percent diluent alone could not account for all the variation in the data.

Whole-particle crushing strengths¹⁰ have been measured on all the particles in this experiment (Table 1). No variables except thickness could be found to explain the variation in the data. The effect of thickness is consistent with previous results.¹⁰

Coating density is important because highly dense coatings retain a higher percentage of the fission products than less dense coatings. Density is expressed in two ways. One is the immersion density¹ and the second is the immersion density corrected for the percent open porosity.¹ A statistical analysis revealed that immersion density is controlled by the process variables hydrocarbon type and deposition temperature (Table 1). Deposition at 1225°C produced coatings that

were about 0.2 g/cm^3 denser than coatings produced at 1350°C . Propylene produced coatings that were about 0.1 g/cm^3 denser than mixed-gas-produced coatings.

The immersion density corrected for the open porosity was also controlled by deposition temperature and hydrocarbon type. This relation was identical to the immersion density because the porosities for most coatings were quite similar (Table 1).

CONCLUSIONS

It is clear from these analyses that the important properties of anisotropy and permeability are controlled by coating rate and density. Therefore, it is important to learn how coating rate and density depend on process variables. A statistical analysis showed that all process variables affected coating rate. All variables were significant at the 99% confidence level, with hydrocarbon type causing the largest effect. Use of mixed gas produced coatings with deposition rates nearly $2 \text{ }\mu\text{m/min}$ faster than propylene when all other variables were held constant. A second important variable is deposition temperature. Higher temperatures (1350°C) produced coating rates about $1.5 \text{ }\mu\text{m/min}$ faster than low (1225°C) deposition temperatures. Helium used as a diluent was found to produce the fastest deposition rate ($0.9 \text{ }\mu\text{m/min}$ faster than hydrogen) while argon produced a deposition rate about $0.4 \text{ }\mu\text{m/min}$ faster than hydrogen. The use of 50% diluent instead of 67% increased in the coating rate by about $0.5 \text{ }\mu\text{m/min}$. The variables affecting density were deposition temperature and hydrocarbon type, as discussed previously.

This experiment has shown that the type diluent and percent diluent have only an indirect effect on the LTI properties of anisotropy and fraction defective. Very isotropic coatings ($\text{BAF}_0 < 1.04$) can be produced with the frit type gas distributor and any combination of the process variables as long as the coating rate is above $3 \text{ }\mu\text{m/min}$. Impermeable and uncracked coatings can also be produced by any combination of process variables if the coating rate is between 2 and $5 \text{ }\mu\text{m/min}$ and the coating has an acceptable density. It is important to note that coatings were produced with both very acceptable values for anisotropy and fraction

defective. It is also important to point out that the conclusions drawn are perhaps valid only over the particular range of process variables investigated.

ACKNOWLEDGMENTS

We are grateful to C. E. DeVore and J. B. Flynn for their careful work during the coating of particles and measurement of coating thicknesses. Numerous members of the staff of General Atomic Company are thanked for their assistance in measuring all the anisotropy and fission gas release measurements. The authors also wish to thank Terry Tiegs and Steve McNeany for reviewing this report.

The manuscript was edited by Sigfred Peterson and prepared for submission for publication by Kathryn S. Witherspoon of the Metals and Ceramics Division Reports office.

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