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INFORMAL AEC RESEARCH
AND DEVELOPMENT REPORT

GAMD-8552

MASTER

FORT ST. VRAIN PROOF TEST ELEMENT = = = NUMBER ONE

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1. INTRODUCTION

The reference fuel element for the Fort St. Vrain High-Temperature Gas-Cooled Reactor consists of an unpurged hexagonal graphite block containing bonded bed fuel rods (columns of coated fuel particles packed to a high bulk density and bonded together by a carbon matrix). An advanced prototype test element containing various types of fuel rods was fabricated for proof irradiation testing in the Peach Bottom reactor. The active portion of the element consists of four hexagonal graphite blocks 3.54-in. wide, each containing 12 fuel holes and 7 coolant holes. The blocks are joined to form a continuous active length of 89 in. After completion, the element was shipped to Peach Bottom and inserted in the A-11-11 core position. During irradiation, the average fuel centerline temperatures were expected to vary from 510°C at the bottom of the element to a peak of 1240°C at approximately two-thirds the active length of the element up from the bottom and to 1120°C at the top. The average fast-neutron dose after 300 equivalent full-power days is expected to be 1.15×10^{21} n/cm² (E > 0.18 MeV), with a peak dose of 1.50×10^{21} n/cm². An average burnup in the fissile particles of about 6.7% FIMA is expected.

The purpose of this report is to describe in detail the materials and processes used in the fabrication of the proof test element, including the characterization of the coated fuel particles and fuel rods, a description of the "piggyback" irradiation specimens, and the details of assembly of the element. These are discussed in the following sections: 2. Coated

Fuel Particles; 3. Bonded Bed Fuel Rods; 4. "Piggyback" Irradiation Specimens; and 5. Assembly of the Element. The fuel-rod development work leading up to these processes, including various problems encountered and their solutions, has been reported previously. (1-4)

2. COATED FUEL PARTICLES USED IN THE FORT ST. VRAIN PROOF TEST ELEMENT

The data generated for the nine batches of coated fuel particles used in the Fort St. Vrain proof test element are presented in this section.

The particles were fabricated by the Fuel Operation Division (FOD). Five batches of particles with a buffer isotropic (BISO) coating and four batches of particles with an intermediate layer of SiC were used in the proof test element. The particles were made to Gulf General Atomic Specification No. X-18-U-5, Issue A.

2.1. BATCH IDENTIFICATION

The nine batches used in the proof test element are identified in Table 1. The first four batches listed are blends of the individual batches listed in column two. The two low-temperature isotropic (LTI) batches were given a thin sacrificial high-temperature isotropic (HTI) overcoat and then redesignated LTI-CO2 and LTI-CO3, as listed. The third batch, HTI-CO2, which was originally identified as HTI-CO1, was screened to remove the size fraction $-589\text{-}\mu\text{m}$ particles from the original batch, and thus was given the new identification HTI-CO2 (see Table 2). The fifth batch listed in Table 1 was a single FOD batch, not a blend. In the last four batches, an SiC layer was incorporated in the coating: 4639 and 4657 are buffer-SiC-PyC, and 4596 and 4640 are buffer-PyC-SiC-PyC.

For purposes of incorporating the data into an existing data retrieval system, each batch was given a code number, which corresponds to the original batch number. This code number is listed in column four.

TABLE 1
COATED-PARTICLE BATCHES USED IN FORT ST. VRAIN
FUEL TEST ELEMENT

New Batch Identification	Original Run No.	Previous Batch Identification	Data Retrieval Run No.
XETK-HTI-CO1	4551 4555	XETK-HTI-CO1	4000-400 4000-551 4000-555
XETK-LTI-CO2	4553 4554 4557 4558	XETK-LTI-CO1	4000-401 4000-553 4000-554 4000-557 4000-558
XTK-HTI-CO2	4585 4587 4591	XTK-HTI-CO1	4000-402 4000-585 4000-587 4000-591
XTK-LTI-CO3	4573 4574 4576 4577	XTK-LTI-CO2	4000-403 4000-573 4000-574 4000-576 4000-577
XTK-HTI-4581	4581	XTK-HTI-4581	4000-581
XETK-4639 XETK-4596 XTK-4657 XTK-4640		XETK-4639 XETK-4596 3943-23N XTK-4640	4000-639 4000-596 4000-657 4000-640

Note: The batches listed in the first column are identified as follows:

XETK - fissile particles (Enriched plus Thorium).

XTK - fertile particles (Thorium).

HTI - high-temperature isotropic coating.

LTI - low-temperature isotropic coating.

TABLE 2
IDENTIFICATION OF PROOF TEST PARTICLES

Present Identification	Action Taken	New Identification
XTK LTI COMP 2 HTI	Sacrificial HTI coat	XTK LTI HTI COMP 3
XETK LTI COMP 1 HTI	Sacrificial HTI coat	XETK LTI HTI COMP 2
XTK 4581 (HTI)	None	No change
XTK HTI COMP 1	-589 μ m particles screened out	XTK HTI COMP 2
XETK HTI COMP 1	None	No change

The values listed in Tables 3 through 15 were taken, primarily, from the data sheets supplied by FOD, which are listed in a private communication from R. G. Mills to W. V. Goeddel dated December 4, 1967.

2. 2. FUEL CORES

All fissile particles were made with one blended batch of XETS-substrate fuel cores; all fertile particles were made with one batch of XTS-3412 fuel cores. The data on these fuel cores are given in Table 3.

2. 3. BUFFER COATINGS

All fissile particles were made from one batch of buffered particles, XETK-4539. The fertile particles were made from a number of buffered batches. The thickness and density data for the PyC-coated batches are given in Table 4 and those for the SiC-coated batches are given in Table 5.

2. 4. OUTER COATINGS

The physical-property data on the PyC and the SiC coating layers are included in Tables 4 and 5. The SiC thickness measurements listed in Tables 5 and 9 are those determined by the Metallurgy Department. The total coating thickness measurement may not be the sum of the

TABLE 3
DATA ON FUEL CORES FOR FORT ST. VRAIN FUEL TEST ELEMENT

Identification	% U	% Th	Th:U Ratio	% C	Core Density (g/cm ³)	Theoret- ical Density (g/cm ³)	% Theoret- ical Density
XTS-3412	----	89.88	----	10.12	8.61	9.32	92.5
XETS-Substrate	57.0	33.31	0.584	9.69	9.8	10.74	91.3

individual coating thickness measurements since differing techniques were utilized by the personnel from the several areas in Gulf General Atomic who made the measurements.

2.5. FINAL COATED PARTICLES

Selected data on the coated particles without an SiC layer are also listed in Table 4 and those for the SiC-coated particles are listed in Table 5. The Bacon anisotropy factor (BAF) values given in these two tables are those made by the Metallurgy Department, rather than those made by FOD. Chemical analyses for the two types of particles are listed in Tables 6 and 7, and the input data for the calculation of the figure of merit are listed in Tables 8 and 9. Identification of each of the radiographic plates and of each of the photographs made of cross sections of particles is given in Table 10. A comparison of the measured density of the final coated particle with the density calculated using the dimension and density values in Tables 3, 4, and 5 is given in Table 11, which includes the value of the number of particles per gram. Large differences in the two density values probably mean that the thickness values or the coating density values listed in Tables 3, 4, and 5 are in error.

The coating strains for the PyC-coated batches listed in Table 4 are based on the BAF and density values given in that table. These values are plotted in Fig. 1, assuming $K_d = 0.32 \times 10^{21} \text{ nvt}^{-1}$ for the LTI

TABLE 4
PARTICLES FABRICATED FOR FORT ST. VRAIN PROOF TEST ELEMENT
(Not including SiC-coated batches)

Combined Batch No.	Coating							Particle				Irradiation			Coated Particle Density (g/cm ³)
	Individual Batch No.	Buffer		Isotropic			Total Thickness (μ m)	Type	Size (μ m)	Th:U Ratio	Merit Factor	Coating Strain	Design Burnup % FIMA	Design Temp. (° C)	
		Thickness (μm)	Density (g/cm ³)	Thickness (μm)	Density (g/cm ³)	BAF									
XETK-HTI-CO1							121				2330		20.0	1300	2.39
	XETK-4539 ^(a)	62	1.03	--	--	--	--	--	--	--	--	--	20.0	1300	--
	XETK-4551	--	--	60	1.91	1.12	122	Th, UC ₂	189	0.58	2370	1.9	20.0	1300	2.45
	XETK-4555	--	--	61	1.85	1.14	121	Th, UC ₂	189	0.58	2330	2.5	20.0	1300	2.31
XETK-LTI-CO2							120				2300		20.0	1300	2.49
	XETK-4539 ^(a)	62	1.03	--	--	--	--	--	--	--	--	--	20.0	1300	--
	XETK-4553	--	--	58	1.94	1.04	120	Th, UC ₂	189	0.58	2300	1.1	20.0	1300	2.52
	XETK-4554	--	--	57	1.89	1.01	119	Th, UC ₂	189	0.58	2260	1.1	20.0	1300	2.53
	XETK-4557	--	--	55	1.88	1.02	117	Th, UC ₂	189	0.58	2190	1.3	20.0	1300	2.54
	XETK-4558	--	--	63	1.88	1.05	125	Th, UC ₂	189	0.58	2470	1.6	20.0	1300	2.38
XTK-HTI-CO2							140				2390		7.0	1300	2.99
	XTK-4585	68	1.09	77	1.87	1.12	145	ThC ₂	395	--	2420	2.2	7.0	1300	2.94
	XTK-4587	71	0.95	72	1.87	1.12	143	ThC ₂	395	--	2640	2.2	7.0	1300	2.86
	XTK-4591	70	1.06	68	1.88	1.15	138	ThC ₂	395	--	2270	2.4	7.0	1300	3.06
XTK-LTI-CO3							145				2520		7.0	1300	3.16
	XTK-4573	69	1.13	73	1.99	1.06	142	ThC ₂	395	--	2260	1.0	7.0	1300	3.14
	XTK-4574	70	1.11	77	1.98	1.04	147	ThC ₂	395	--	2450	0.9	7.0	1300	3.08
	XTK-4576	66	0.94	82	1.98	1.01	148	ThC ₂	395	--	2790	0.6	7.0	1300	3.19
	XTK-4577	68	1.00	70	1.98	1.98	138	ThC ₂	395	--	2380	0.7	7.0	1300	3.23
XTK-HTI-4581	XTK-HTI-4581	68	1.00	83	1.90	1.05	151	ThC ₂	395	--	2780	1.4	7.0	1300	2.87

^(a) Buffer batch.

TABLE 5
PARTICLES FABRICATED FOR FORT ST. VRAIN PROOF TEST ELEMENTS
(SiC-coated batches)

Final Batch No.	Buffer			Inner Isotropic Coating				Silicon Carbide		Outer Isotropic Coating				Overcoating				Total Thickness (μ m)	Particle			Irradiation		Coated Particle Density (g/cm ³)
	Run No.	Thickness (μ m)	Density (g/cm ³)	Run No.	Thickness (μ m)	Density (g/cm ³)	BAF	Run No.	Thickness (μ m)	Run No.	Thickness (μ m)	Density (g/cm ³)	BAF	Run No.	Thickness (μ m)	Density (g/cm ³)	BAF		Type	Size (μ m)	Th: U Ratio	Design Burnup % FIMA	Design Temp. (° C)	
XETK-4596	XETK-4539	62	1.03	XETK-4556	34	1.92	1.03	3662-63E	13	XETK-4596	8	(a)	(a)	----	---	---	---	113	Th, UC ₂	189	0.58	20.0	1300	2.78
HETK-4639	XETK-4539	62	1.03	----	--	----	----	3662-57E	15	XETK-4560	48	1.93	1.04	HETK-4639	7	(a)	(a)	127	Th, UC ₂	189	0.58	20.0	1300	2.60
HTK-4640	XTK-4601	74	0.96	XTK-4601	33	1.89	1.10	3662-69E	13	HTK-4640	26	1.91	----	----	---	---	---	146	Th, C ₂	397	----	7.0	1300	3.10
XTK-4657	XTK-4648	70	1.07	----	--	----	----	3662-135	15	3943-23N	54	1.77	1.20	----	---	---	---	139	Th, C ₂	397	----	7.0	1300	3.27

(a) Unobtainable because of thickness of coatings.

TABLE 6
CHEMICAL ANALYSIS OF FORT ST. VRAIN FUEL TEST ELEMENT
(Not including SiC-coated batches)

Combined Batch No.	Buffer Batch No.	Individual Batch No.	% U	% Th	% C	Percent Total
XETK-HTI- CO1	XETK-4539		22.92	13.27	63.69	99.88
			42.66	24.92	32.42	100.00
		XETK-4551	----	----	64.44	
		XETK-4555	----	----	61.68	
XTK-LTI- HTI-CO3	XTK-4573 XTK-4574 XTK-4576 XTK-4577		----	49.91	50.31	100.22
			----	----	24.19	
			----	----	24.33	
			----	----	22.92	
			----	----	23.38	
		XTK-4573	----	50.48	----	
		XTK-4574	----	49.28	----	
		XTK-4576	----	50.48	----	
		XTK-4577	----	52.40	----	
XTK-4581	XTK-4581		----	46.95	52.87	99.82
			----	----	25.27	
XTK-HTI- CO2	XTK-4585 XTK-4587 XTK-4591		----	49.83	50.21	100.04
			----	----	22.69	
			----	----	21.05	
			----	----	24.19	
		XTK-4585	----	47.44	52.42	99.86
		XTK-4587	----	47.97	52.19	100.16
		XTK-4591	----	50.85	49.06	99.91
XETK-LTI- HTI-CO2	XETK-4539		22.16	12.70	64.54	99.40
			42.66	24.92	32.42	100.00
		XETK-4553	----	----	63.45	
		XETK-4554	----	----	59.60	
		XETK-4557	----	----	62.62	
		XETK-4558	----	----	65.95	

TABLE 7

CHEMICAL ANALYSIS OF SILICON CARBIDE RUNS FOR
FORT ST. VRAIN FUEL TEST ELEMENT

Final Batch No.	Intermediate Batch No.	% U	% Th	% SiC	Total % C	% Si	Percent Total
XETK-4596		23.5	13.5	20.4	48.6	14.3	99.90
	XETK-4539 (Buffer)	42.6	24.92	----	32.42	----	100.00
	XETK-4556 (Inner isotropic)	31.84	18.78	----	49.27	----	99.89
	3662-63E (SiC)	25.00	14.15	----	----	----	-----
HETK-4639		20.05	11.45	16.7	56.84	11.66	100.00
	XETK-4539 (Buffer)	42.66	24.92	----	32.42	----	100.00
	3662-57E (SiC)	34.05	18.95	----	----	----	-----
HTK-4640		----	46.80	15.0	42.72	10.48	100.00
	XTK-4601 (Buffer)	----	75.19	----	23.90	----	99.09
	XTK-4601 (Inner isotropic)	----	~60.	----	39.23	----	~99.23
	3662-69 (SiC)	----	56.65	----	35.40	----	-----
XTK-4657		----	53.9	9.7	39.3	6.8	100.00
	XTK-4648 (Buffer)	----	78.92	----	20.95	----	99.87
	3662-135 (SiC)	----	----	----	----	----	-----

TABLE 8
INPUT DATA FOR FIGURE OF MERIT CALCULATIONS FOR FORT ST. VRAIN FUEL ELEMENT

Combined Batch No.	Individual Batch No.	Core Diameter (μm)	Core Density (g/cm^3)	Core Theoretical Density (g/cm^3)	Core HM Fraction	HM Mole Wt (g/mole)	Burnup Fraction	Temp. ($^{\circ}\text{C}$)	Buffer Thickness (μm)	Buffer Density (g/cm^3)	Overcoat Thickness (μm)	Tensile Strength (psi)	Figure of Merit ^(a)
XETK-HTI-CO1		189	9.80	10.74	0.903	234	0.200	1300	62	1.03	59	40,000	2332
	XETK-4551	189	9.80	10.74	0.903	234	0.200	1300	62	1.03	60	40,000	2367
	XETK-4555	189	9.80	10.74	0.903	234	0.200	1300	62	1.03	59	40,000	2332
XTK-LTI-CO3		395	8.61	9.32	0.899	232	0.070	1300	68	1.04	77	40,000	2517
	XTK-4573	395	8.61	9.32	0.899	232	0.070	1300	69	1.13	73	40,000	2261
	XTK-4574	395	8.61	9.32	0.899	232	0.070	1300	70	1.11	77	40,000	2447
	XTK-4576	395	8.61	9.32	0.899	232	0.070	1300	66	0.94	82	40,000	2789
	XTK-4577	395	8.61	9.32	0.899	232	0.070	1300	68	1.00	70	40,000	2376
XTK-4581	XTK-4581	395	8.61	9.32	0.899	232	0.070	1300	68	1.00	83	40,000	2777
XTK-HTI-CO2		395	8.61	9.32	0.899	232	0.070	1300	70	1.03	70	40,000	2388
	XTK-4585	395	8.61	9.32	0.899	232	0.070	1300	68	1.09	77	40,000	2420
	XTK-4587	395	8.61	9.32	0.899	232	0.070	1300	71	0.95	72	40,000	2638
	XTK-4591	395	8.61	9.32	0.899	232	0.070	1300	70	1.06	68	40,000	2271
XETK-LTI-CO2		189	9.80	10.74	0.903	234	0.200	1300	62	1.03	68	40,000	2632
	XETK-4553	189	9.80	10.74	0.903	234	0.200	1300	62	1.03	58	40,000	2298
	XETK-4554	189	9.80	10.74	0.903	234	0.200	1300	62	1.03	57	40,000	2264
	XETK-4557	189	9.80	10.74	0.903	234	0.200	1300	62	1.03	55	40,000	2194
	XETK-4558	189	9.80	10.74	0.903	234	0.200	1300	62	1.03	63	40,000	2468

^(a) Calculation from MERIT-2, run No. 22.

TABLE 9

SILICON-CARBIDE RUNS INPUT DATA FOR FIGURE OF MERIT CALCULATIONS FOR FORT ST. VRAIN FUEL TEST ELEMENT

Final Batch No.	Core Diam. (μ m)	Core Density (g/cm ³)	Core Theoret- ical Density (g/cm ³)	Core HM Fraction	HM Mole Wt (g/mole)	Burnup Fraction	Temp. (°C)	Buffer Thick- ness (μ m)	Buffer Density (g/cm ³)	Inner Isotropic Thick- ness (μ m)	SiC Thick- ness (μ m)	Outer Isotropic Thick- ness (μ m)	Overcoat Isotropic Thick- ness (μ m)	Tensile Strength Silicon (psi)	Strength PyC (psi)	Figure of Merit ^(a)	
																Load-Bearing Layer	
																PyC	SiC
XETK-4596	189	9.80	10.74	0.903	234.	0.200	1300	62	1.03	34 ^(b)	13	8	--	100,000	40,000	1410	1380
HETK-4639	189	9.80	10.74	0.903	234.	0.200	1300	62	1.03	--	15	48 ^(b)	7	100,000	40,000	1940	1590
HTK-4640	395	8.61	9.32	0.899	232.	0.070	1300	74	0.96	33 ^(b)	13	26	--	100,000	40,000	1290	1280
XTK-4657	397	8.61	9.32	0.899	232.	0.070	1300	70	1.07	--	15	54 ^(b)	--	100,000	40,000	1800	1280

^(a) Calculations from MERIT-2, run No. 23.^(b) Value represents PyC layer used as load-bearing layer in calculations.

TABLE 10

PHOTOGRAPHIC EVALUATION DATA OF FORT ST. VRAIN
PROOF TEST ELEMENT

Final Batch No.	Individual Batch No.	Radio- graphic Plate No. E-	Metallographic Polaroid No. M-
XETK-HTI-CO1	4551	417	22000
	4555	374	
		377	
XTK-LTI-CO3	4573	418	22302
	4574	396	
	4576	397	
	4577	398	
XTK-4581	4581	399	
XTK-HTI-CO2	4585	402	22214
	4587	442	
	4591	421	
		423	
XETK-LTI-CO2	4553	425	22286
	4554	404	
	4557	375	
	4558	376	
XETK-4596	4596	379	22301
XETK-4639	4639	380	
XTK-4640	4640	428	
XTK-4657	4657	475	
		476	22389
		484	22460
			22461
			22549

Note: 10 X and 50 X enlargements of the radiographic plates listed above have been made.

TABLE 11
COMPARISON OF DENSITIES OF FINAL COATED FUEL PARTICLES

Batch No.	Particle Density (g/cm ³)			Number of Particles per gram
	Measured	Calculated	Difference	
XETK-HTI-CO1	2.39	2.29	0.10	10,400
XETK-LTI-CO2	2.49	2.26	0.23	9,300
XTK-HTI-CO2	2.99	2.97	0.02	2,100
XTK-LTI-CO3	3.16	2.99	0.17	2,000
XTK-HTI-4581	2.87	2.89	-0.02	1,950
XETK-4596	2.78	2.54	0.24	10,000
KETK-4639	2.60	2.40	0.20	8,500
HTK-4640	3.10	3.01	0.09	1,960
XTK-4657	3.27	3.07	0.20	2,000

coatings and $K_d = 0.28 \times 10^{21} \text{ nvt}^{-1}$ for the HTI coatings. Similar values for the PyC layers on the SiC-coated batches are listed in Table 5; these are plotted in Fig. 2.

2.6. FISSION-PRODUCT RELEASE EVALUATION OF FUEL PARTICLES

Fission-product release measurements were made on particles and rods fabricated for the proof test element. These measurements were carried out by the Chemistry Department and were made after completion of the element because of the very tight time schedule. Steady-state fission-gas release measurements were made on all batches of coated particles and metallic fission-product release studies were made on two batches of BISO-coated particles.

Fig. 1. Effect of structural parameters on shrinkage rate at total shrinkage of 2 percent (calculations based on GA-7700(5))

Fig. 2. Effect of structural parameters on shrinkage rate at total shrinkage of 2 percent (calculations based on GA-7700(5))

The data obtained in the metallic fission-product annealing experiments (also called a postactivation release annealing experiment^{*}) are given in Table 12. These data show that the release from HTI particles at 8 hr is somewhat greater than the release from the LTI particles. This higher value may be due to a higher contamination level in the outer pyrolytic carbon of the HTI particles. This hypothesis is supported by the higher Xe¹³⁵ release of HTI particles. At steady state, however, the metallic release for both samples would be expected to level out at a value between 0.5 and 1.0 since the release of metallic fission products from BISO particles is controlled by the kernel, because pyrolytic carbon is not a barrier to the release of Ba and Sr.

Fission-gas release measurements are conducted by irradiating the sample in-core in the TRIGA King furnace⁽⁶⁾ at either room temperature or at elevated temperatures. The fission gases liberated in the 1-hr irradiation are swept into liquid nitrogen-cooled charcoal traps that are gamma counted to determine the quantity released. The experimental results were corrected to steady-state values (R/B) using the equations of Zumwalt, Gethard, and Anderson.⁽⁶⁾ The results of these measurements are given in Tables 13 and 14 and the data are plotted versus half-life in Figs. 3 through 11.

Two comments should be made about the release measurements on the fissile particles: First, for batch No. XETK-LTI-CO2, the release at 1000°C exceeds the release at 1400°C by about one order of magnitude. While the same sample was used for the room temperature and 1400°C measurements, a fresh sample was used for the 1000°C measurement. Since the precision of the measurements is better than the difference

^{*}In a postactivation release annealing experiment a leached sample of particles is irradiated at room temperature in the TRIGA reactor to $\sim 10^{14}$ fissions. The sample, in a graphite crucible, is then annealed at 1400°C in a graphite tube furnace and the Ba¹⁴⁰ and Sr⁹¹ fission products released from the particles are sorbed in the crucible, which is gamma counted to determine the amount released.

TABLE 12

METALLIC FISSION-PRODUCT RELEASE AT 1400°C OF FISSILE PARTICLES^(a) FABRICATED FOR THE FORT ST. VRAIN PROOF TEST ELEMENT NO. 1

Time (hr)	Fractional Release		
	Ba ¹⁴⁰	Sr ⁹¹	Xe ¹³⁵
XETK-LTI-CO2			
0	$< 4.2 \times 10^{-6}$	-----	$< 1.7 \times 10^{-7}$
1.0	2.5×10^{-4}	6.9×10^{-3}	-----
3.0	8.4×10^{-4}	2.1×10^{-2}	-----
5.0	1.8×10^{-3}	4.1×10^{-2}	$< 3.0 \times 10^{-8}$
8.0	6.2×10^{-3}	7.6×10^{-2}	-----
XETK-HTI-CO1			
0	6.2×10^{-5}	-----	5.9×10^{-6}
1.0	1.4×10^{-3}	4.4×10^{-2}	-----
3.0	3.8×10^{-3}	7.3×10^{-2}	-----
5.0	6.4×10^{-3}	9.3×10^{-2}	9.1×10^{-6}
8.0	9.4×10^{-3}	1.1×10^{-1}	-----

(a) Particles were leached by FOD to remove surface contamination and broken particles.

TABLE 13
STEADY-STATE RELEASE OF FISSION GASES FROM FISSILE FUEL PARTICLES^{(a)(b)}

Batch No.	Coating Type	Temp (°C)	Kr ^{85m}	Kr ⁸⁸	Kr ⁸⁷	Kr ⁸⁹	Xe ¹³⁸	Xe ¹³⁹
XETK-LTI-CO2	BISO-LTI	1400 ^(c)	8.5×10^{-6}	4.8×10^{-6}	3.0×10^{-6}	9.8×10^{-7}	1.2×10^{-6}	---
		1000 ^(c)	5.7×10^{-5}	3.7×10^{-5}	3.4×10^{-5}	3.8×10^{-6}	6.4×10^{-6}	2.3×10^{-6}
		room	2.8×10^{-6}	1.1×10^{-6}	6.7×10^{-7}	6.2×10^{-7}	1.2×10^{-6}	---
XETK-HTI-CO1	BISO-HTI	1400	1.7×10^{-4}	1.4×10^{-4}	1.2×10^{-4}	2.0×10^{-5}	1.6×10^{-5}	7.1×10^{-6}
		room	2.5×10^{-5}	1.4×10^{-5}	8.0×10^{-6}	3.6×10^{-6}	5.0×10^{-6}	2.9×10^{-6}
XETK-4596	TRISO-II	1000	1.2×10^{-5}	6.5×10^{-6}	5.7×10^{-6}	1.4×10^{-6}	3.5×10^{-6}	7.3×10^{-7}
HETK-4639	TRISO-I	1000	8.0×10^{-6}	9.8×10^{-6}	4.2×10^{-6}	1.4×10^{-6}	2.3×10^{-6}	2.1×10^{-6}

(a) All measurements were carried out on samples of particles which were leached by FOD to remove surface contamination.

(b) All data were obtained with 1-hr irradiations and were corrected to steady-state values using the equations of Zumwalt, Gethard, and Anderson. (6)

(c) The higher level of release at 1000°C instead of 1400°C can only be attributed to sampling differences since the difference exceeds the precision of the measurements.

TABLE 14
STEADY-STATE RELEASE OF FISSION GASES FROM FERTILE FUEL PARTICLES^{(a)(b)}

Batch No.	Coating Type	Temp (°C)	Kr ^{85m}	Kr ⁸⁸	Kr ⁸⁷	Kr ⁸⁹	Xe ¹³⁸	Xe ¹³⁹
XTK-4657	TRISO-I	1000	4.5×10^{-5}	8.5×10^{-5}	3.2×10^{-5}	$<8 \times 10^{-6}$	$<3 \times 10^{-5}$	$<2 \times 10^{-5}$
XTK-HTI-4581	BISO-HTI	1000	1.2×10^{-4}	2.1×10^{-4}	8.7×10^{-5}	$<5 \times 10^{-6}$	$<2 \times 10^{-5}$	$<3 \times 10^{-5}$
XTK-HTI-CO2	BISO-HTI	1000	$<8 \times 10^{-5}$	$<1 \times 10^{-4}$	$<5 \times 10^{-6}$	$<6 \times 10^{-6}$	$<7 \times 10^{-6}$	$<2 \times 10^{-5}$
XTK-4640	TRISO-II	1000	1.2×10^{-4}	3.1×10^{-4}	8.0×10^{-5}	9.4×10^{-6}	4.1×10^{-5}	3.0×10^{-5}
XTK-LTI-CO3	BISO-LTI	1000	$<4 \times 10^{-5}$	$<3 \times 10^{-5}$	$<7 \times 10^{-6}$	$<5 \times 10^{-6}$	$<1 \times 10^{-5}$	$<3 \times 10^{-5}$

(a) All measurements were carried out on samples of particles which were leached by FOD to remove surface contamination.

(b) All data were obtained with 1-hr irradiations and were corrected to steady-state values using the equations of Zumwalt, Gethard, and Anderson. (6)

Fig. 3. Gaseous fission-product release data from XETK-LTI-CO₂ fuel particles at room temperature, 1000°C, and 1400°C

Fig. 4. Gaseous fission-product release data from XETK-HTI-CO1 fuel particles at room temperature and 1400°C

Fig. 5. Gaseous fission-product release data from XETK-4596 fuel particles at 1000°C

Fig. 6. Gaseous fission-product release data from XETK-4639 fuel particles at 1000°C

Fig. 7. Gaseous fission-product release data from XTK-4657 fuel particles at 1000°C

Fig. 8. Gaseous fission-product release data from XTK-HTI-4581 fuel particles at 1000°C

Fig. 9. Gaseous fission-product release data from XTK-HTI-CO₂ fuel particles at 1000°C

Fig. 10. Gaseous fission-product release data from XTK-4640 fuel particles at 1000°C

Fig. 11. Gaseous fission-product release data from XTK-LTI-CO3 fuel particles at 1000°C

between the 1400° and 1000°C data, the anomalous behavior can only be attributed to sampling differences. Second, if the 1000°C data for XETK-LTI-CO2 are ignored, the elevated temperature release of the BISO-HTI particles (batch No. XETK-HTI-CO1) exceeds the elevated-temperature releases of the other fissile batches by about one order of magnitude.

In many cases, fission-gas release measurements on fertile particles yielded only upper limits for the release. However, it should be noted that the release levels for the fertile particles are generally about an order of magnitude higher than the release levels for the fissile particles except for the BISO-HTI sample.

The results of fission-gas release measurements on the fuel particles show that

1. The specification for fission-gas release* for particles to be used in the proof test element were met within the limits of error by fissile batches XETK-4596 and XETK-4639. Batch No. XETK-LTI-CO2 also meets the specification if the 1400°C data are accepted rather than the 1000°C data.
2. Fissile batch No. XETK-HTI-CO1 (BISO-HTI) exceeds the fission-gas release specifications by about one order of magnitude.
3. If upper limits of release for fertile particles are accepted as valid measurements, none of the fertile-particle batches met the fission-gas release specifications.
4. The steady-state fission-gas release from fuel particles generally shows a $\sqrt{t_{1/2}}$ dependence on half-life ($t_{1/2}$) of the isotope.

The fission-gas release measurements on fuel rods are discussed in Section 3 and are summarized in Table 20.

*The fission-gas release specification for the Fort St. Vrain Proof Test Element No. 1 states that "The steady-state fission-gas release (R/B) rate for noble gas fission products with half-lives less than 4.4 hours shall be less than 1×10^{-5} ."

3. BONDED BED FUEL RODS

The fuel rods basically consist of columns of both fertile and fissile coated fuel particles packed to a high density and bonded together by a carbon matrix. A bond between the coated particles is desirable to prevent the particles from pouring out of the element in the event any damage should occur to the element that would open a cavity in the fuel rod. However, the fuel rod is not bonded to the graphite container wall. In fact, a 1- to 3-mil gap is desirable. The matrix contains carbonized resin binder and carbon filler. In addition to acting as a bonding agent, the carbon matrix also serves to sorb metallic fission products that may escape from the coated particles.

3.1. REQUIREMENTS AND TYPES OF FUEL RODS

The proof test element design required approximately 994 in. of fuel rod containing a total of 447 g of enriched uranium or 0.45 g of uranium per inch of fuel rod. A total thorium content of approximately 1670 g was also specified, although this number could vary depending on the particle loadings obtained. The fertile coated fuel particles contained all thorium carbide, whereas the fissile particles contained both enriched uranium and thorium carbide with a Th:U ratio of 0.58:1. A total of 153 individual fuel rods having nominal lengths of 1 in., 6 in., and 11 in. were used in the element. The majority (141) of the rods were solid and had a diameter of 0.467 in., the remaining 12 rods had an annular design to allow insertion of a thermocouple. These rods were 0.515-in. OD and 0.206 in. ID, and they contained a graphite tube with a 0.025-in. -thick wall.

The fuel-rod composition variables, including four types of coated particles, two types of binder, two types of filler, and two fabrication methods, are listed in Table 15.

TABLE 15
FUEL-ROD COMPOSITION VARIABLES

Types of Coated Particles(a):

- LTI - Low-temperature isotropic pyrocarbon coating
- HTI - High-temperature isotropic pyrocarbon coating
- SiC 1 - Silicon carbide-pyrocarbon Type 1 coating
- SiC 2 - Silicon carbide-pyrocarbon Type 2 coating

Types of Resin Binder Materials:

- Plyophen 5023S phenolic resin
- Furfuryl alcohol polymer

Types of Filler Materials:

- GP-38 graphite powder
- Blend of two parts GP-38 and one part Barneby-Cheney 589 charcoal powder

Types of Fabrication Processes:

- Injection Molding
- Wet Molding

(a) Fuel rods containing SiC-coated particles were included since this element provided a convenient opportunity for irradiation testing of these advanced coatings under desired conditions, as noted in the HTGR Base Program Quarterly Progress report for November 1967. (7)

Most of the calculated and measured data for fuel-rod fabrication and the measurements on the completed rods were recorded and stored in a bonded-bed data retrieval computer program (DRO34). In this program, all information is indexed by the fuel-rod number.

3. 2. FABRICATION OF RODS BY INJECTION MOLDING

There are four main steps to the injection-molding fuel-rod fabrication process:

1. The fissile and fertile coated particles are blended and loaded into the metal mold cavities to form columns of loose, closely packed particles.
2. A thermosetting liquid resin binder mix is injected under pressure into the columns of particles.
3. The thermosetting resin in the rods is then heat-cured to a hard, green state.
4. The green rods are carbonized.

3. 2. 1. Molds and Accessories Used in the Process

Each of the Type 304 stainless steel molds used for forming the green fuel rods have six rod cavities in a "telephone-dial" arrangement. A schematic diagram of a mold assembly is shown in Fig. 12; the detailed assembly illustrated in Fig. 13 shows both typical solid rod and annular rod cavities.

Other accessories used with the mold include brass ring inserts for forming a 1/16-in. radius on the rim of the rod at each end and numbered plastic discs. These discs are inserted in the top of a filled and injected mold cavity to extrude excess binder and to mold the identification number on the top of the rod. Three molds, 1.120 in., 5.700 in., and 11.650 in. in length, are shown in Fig. 14 along with accessories and completed fuel rods.

Before loading the coated particles, the mold cavities, end caps, and other accessories are coated with a release agent (RAM-225), which is dried for 30 min at 65°C.

A brass insert ring is placed in the bottom of each mold cavity with the radius side facing inward in the hole. The bottom plate with the O-ring in place is assembled and secured. The mold is turned upright and the bottom radius insert ring tamped in place. The top plate and radius insert ring are not assembled until the particle loading and injection are complete.

TYPE	MOLD LENGTHS	HOLD DIAM
ROD	± 0.005 IN.	± 0.0005 IN.
SOLID	1.120	0.4675
SOLID	5.700	0.4675
ANNULAR	6.179	0.5150
SOLID	11.650	0.4675

SPECIFICATIONS FOR ALL MOLDS

MATERIAL: 304 STAINLESS STEEL

CAVITY FINISH: 16μ IN.

CAVITY BOW PER FT : 0.001 IN.

OUTSIDE DIAMETER: 2-1/2 IN.

Fig.12, Schematic drawing of metal molds for the fabrication of bonded bed fuel rods by injection molding

Fig. 13. Detail of assembly of mold for the fabrication of bonded bed fuel rods by injection molding

HT59098

Fig. 14. Stainless-steel fuel-rod molds and accessories with six cured fuel rods

3.2.2. Blending and Loading of Fuel Particles

The blending and loading of the fissile and fertile particles in the mold cavities is done with a double hopper—double orifice metering system. The two types of particles are fed simultaneously through two controlled orifices into a blending funnel and then into the mold cavity. The mold is vibrated as the particles are loaded to achieve close packing.

The double hopper—double orifice device, as shown in Fig. 15, consists of two triangular cavities with one side of each cavity adjustable to control the orifice opening at the bottom. A micrometer on each side is used to determine the orifice opening. The flow rate through each orifice can be adjusted independently of each other. The orifices are opened and closed simultaneously by a timed, solenoid-operated orifice gate valve. The tapered conical blending funnel is designed to accept the two streams of flowing particles from each orifice on its wide outer rim, causing the particles to deflect into the tapered lower end of the funnel. Using this technique, an intimate mixture of the two types of particles is obtained with the desired particle distribution.

The lower end of the blending funnel, which is the same diameter as a mold cavity, is aligned with the cavity by means of an adaptor plate.

The blending funnel and a mold with the adaptor plate attached is shown in Fig. 16. The complete loading assembly can be seen in Fig. 17. This includes the double hopper—double orifice device and the blending funnel with the adaptor plate attached to an 11.65-in. -mold. The mold is resting on a vibrator. The timer for the solenoid-operated orifice gate valve is also shown.

In operating the loading assembly, the required amount of fissile particles for a given uranium loading is weighed and loaded into the hopper; the total flow time through the orifice is then measured. (In setting the flow rate, usually about 2 g/sec, orifice openings of less than 3 particle diameters should not be used because of the possibility of particle bridging.) The required amount of fertile particles is then loaded into the other

- 1 HOPPER-ADJUSTING MICROMETER
- 2 ADJUSTABLE SIDE OF HOPPER
- 3 HOPPER SIDE PLATE
- 4 HOPPER SLIDE VALVE
- 5 ADJUSTABLE ORIFICE OPENING

Fig. 15. Detailed assembly of the double hopper-double orifice apparatus for the simultaneous metering of fissile and fertile coated fuel particles into the blending funnel

HT59113

Fig. 16. Blending funnel for blending and loading fissile and fertile coated fuel particles into a fuel-rod mold; a mold with adaptor plate attached is also shown

HT59103

Fig. 17. Complete fissile and fertile coated-particle metering, blending, and loading assembly with fuel-rod mold

hopper and this orifice adjusted to give a total flow time equal (to within 0.1 sec) to that of the fissile particles. Once the settings are determined, weighed amounts of both types of particles can be loaded into each cavity with no changes. As the particles fall into the mold cavity, a high packing fraction (60 to 65 volume percent) is obtained by vibrating the mold. A mold with two cavities filled with loose particles and the blending funnel are shown in Fig. 18.

From earlier developmental work, this method of blending and loading the fuel particles was determined to be the most successful and resulted in particle distributions which deviated from the ideal by only $\pm 4\%$ maximum.

3. 2. 3. Injection of Binder Mix with "Grease-Gun" Apparatus

The binder mix consists of a liquid, thermosetting phenolic resin (Plyophen 5023S) blended with powdered graphite (GP-38) and charcoal (Barneby-Cheney 589) filler materials. The Plyophen 5023S resin is a viscous, high-solids resin with a coking value of 55% to 60%. It also shows good wetting and bonding characteristics to carbon and graphite. A binder found to be satisfactory for the current injection molding consisted of 70% by weight of resin plus 20% GP-38 graphite powder and 10% Barneby-Cheney 589 charcoal powder as filler material. A few rods were also made with a 70% resin-30% GP-38 formulation. It is necessary that the filler material have a particle size of less than 30 μ m for complete injection of an 11-in. column of particles. Using substantial amounts of a larger particle-size filler resulted in a layer filtering out at the top of the fuel-particle column and plugging the binder flow. The charcoal was a special material with a particle size considerably less than 30 μ m. However, the GP-38 as received from the manufacturer contains about 52% by weight of particles greater than 30 μ m. This material can be separated with a cyclone particle classifier type of apparatus using a high air flow. The apparatus shown in Fig. 19 was constructed for this purpose and it proved highly successful in achieving the desired separation.

HT59105

Fig. 18. Blending funnel and fuel-rod mold with two cavities containing loose fissile and fertile coated fuel particles

HT59097

Fig. 19. High-air-flow cyclone-type particle classifier for separating fraction of powdered graphite less than 30- μ m particle size

Blending of the binder formulation is accomplished by combining the preweighed components in the following order: The powdered filler materials are first dry mixed and then they are added to the resin and stirred until complete wetting occurs. Additional mixing is done with a motor-driven stirring blade to assure homogeneity.

For binder injection, the binder mix is loaded into the reservoir of the "grease-gun" injection apparatus. This apparatus consists basically of a binder mix reservoir, a pressure chamber with two check valves to direct the flow of the binder, a double-acting air cylinder to develop injection pressures up to 1280 psi, a set of multiple-injection loading orifices with a mold seal surface, and a mold support structure. A schematic diagram of this apparatus is shown in Fig. 20. The whole assembly and a 6-in. -long mold being loaded are shown in Fig. 21. The assembly turned to a horizontal position to show the multiple loading orifices and mold seal surface is shown in Fig. 22. After the reservoir is filled with binder mix, the mold containing the loose coated particles is placed in the mold support structure and the top tightened against the mold seal surface. The double-acting air cylinder is then activated and cycled to begin pumping the binder mix down through the column of particles. The multiple-loading injection molding orifices allow all six cavities of a mold to be injected simultaneously. Completion of the injection is indicated by a stopping of the air cylinder action and the appearance of binder mix at the edge of the bottom end mold plate. After injection is completed and the mold removed from the apparatus, the top brass ring inserts and plastic discs are put in place. The top mold plate is assembled and tightened down to force the rings and discs into place; this also displaces any excess binder remaining at the top of the mold cavity.

Fig. 20. Fuel-rod injection-molding apparatus (grease gun)

HT59111

Fig. 21. Complete assembly of fuel-rod injection-molding apparatus (grease gun) with 6-in. mold in place

HT59107

Fig. 22. Horizontal view of fuel-rod injection-molding apparatus (grease gun) showing multiple-loading orifices and mold seal surface

3.2.4. Curing and Carbonization of the Fuel Rods

To cure the phenolic resin and harden the fuel rods, the loaded mold is placed in a circulating-air oven at 90 °C for 16 hr and at 120 °C for 4 hr. This completes the "green" or thermosetting cure. The mold is then disassembled and the green cured rods, which are quite strong at this stage, are pushed out, examined, and measured. They are then placed in a graphite holder assembly in preparation for carbonization, as shown in Fig. 23. Carbonization is accomplished in a resistance heated furnace, shown in Fig. 24, under a nitrogen purge. The rods are heated to 900 °C in 38 hr and held at that temperature for 1 hr. The heating is programmed as follows:

<u>Carbonization Heating</u>	<u>Total Heating Time</u>
Room temp. to 150 °C at 130 °C/hr	1 hr
150 °C to 250 °C at 33.3 °C/hr	3 hr
250 °C to 500 °C at 10 °C/hr	25 hr
500 °C to 600 °C at 33.3 °C/hr	3 hr
600 °C to 900 °C at 50 °C/hr	6 hr
Held at 900 °C	<u>1 hr</u>
Total accumulated carboniza- tion heating time	39 hr

This program was designed to give the slowest heating rate while the maximum amount of volatile material was being released from the rod. After carbonization and cooldown, the rods are removed and again examined and measured. They are then stored until ready for assembly.

It was discovered that occasionally cracked particle coatings were observed, apparently from a strong matrix-to-particle coating bond and subsequent shrinkage of the matrix during carbonization. To overcome this problem, a controlled weakening of both the matrix and

HT59112

Fig. 23. Graphite-holder assembly for the carbonization of cured fuel rods

HT59106

Fig. 24. Resistance-heated furnace and atmosphere tube for carbonizing fuel rods at 900 °C (note furnace controls and cam for giving programmed heat-up cycle)

matrix-to-coating bond was used. This consisted of using a 10% charcoal, 20% GP-38 filler matrix mix and of overcoating the main LTI or SiC particle coatings with a thin HTI coating. Also, prior to loading, all particles were overcoated with Spraygraph, a colloidal graphite mold release agent. The Spraygraph was applied from a pressurized can to 200-g batches of coated particles contained in an open hexagonal shaped tumbling drum. The drum was rotated at about 100 rpm and heated to 120 °F while a uniform coating was sprayed on the tumbling fuel particles. Enough coating was applied to give a 0.3- to 0.5-g weight increase per 200-g batch of particles.

3.3. FABRICATION OF FUEL RODS BY WET MOLDING

A few of the rods were fabricated by a process developed by FOD known as wet molding. This process consists of mechanically mixing the required loose particles with the liquid matrix mix to form a wet granulated fuel mix. A weighed amount of this wet mix is then packed into both halves of a longitudinally split mold to give the required particle density and uranium loadings. The mold is assembled and the mix is heat-cured for 16 hr at 100 °C to form a solid rod. The rod is then removed from the mold and carbonized in graphite holders in the same manner as the injection molded rods (39-hr cycle to 900 °C under nitrogen). The matrix used with this particular process consisted of 70 wt-% furfuryl alcohol polymer and 30 wt-% GP-38 graphite flour. The furfuryl alcohol polymer is formed by mixing 10 parts furfuryl alcohol with 1 part maleic anhydride.

3.4. EVALUATION OF FUEL RODS PRODUCED

A total of 221 fuel rods were fabricated; 209 by the Metallurgy Department using injection molding and 12 by FOD using wet molding. Of these, 153 rods were loaded in the proof test element. The types and lengths of rods used in the proof test element are described in Table 16.

TABLE 16

SUMMARY OF TYPES OF FUEL RODS FABRICATED BY THE
INJECTION- AND WET-MOLDING PROCESSES AND LOADED
IN THE PROOF TEST ELEMENT

Fuel Rod Compo- sition ^(a)	Number of Rods Required	Length of Rods Required (in.)	Total Rod Length of Each Type Required (in.)	Figure of Metal- lographic Section of Rod
HPBI	20	1.00	20.0	25
	11	5.57	61.2	26
	5	5.93 annular	29.6	27
	25	11.53	288.2	28
HPGI	4	1.00	4.0	29
LPBI	23	1.00	23.0	30
	4	5.57	22.3	31
	7	5.93 annular	41.5	--
	23	11.53	265.2	32
LPGI	4	1.00	4.0	33
	4	11.53	46.1	--
HFGW	1	5.57	5.6	--
	2	11.53	23.0	34
LFGW	2	5.57	11.1	35
	4	11.53	46.1	--
SPBI	2	1.00	2.0	36
	2	5.57	11.1	--
	4	11.53	46.1	--
TPBI	2	1.00	2.0	37
	1	5.57	5.6	--
	2	11.53	23.0	--
SHPBI ^(b)	1	11.53	11.5	--
TOTALS	153		992.2	

TABLE 16 (continued)

(a) Rod Composition Coding

First letter—type of coated particle:	H = high-temperature isotropic pyrocarbon coating (HTI)
	L = low-temperature isotropic pyrocarbon coating (LTI)
	S = silicon carbide coating Type 1 (SiC-1)
	T = silicon carbide coating Type 2 (SiC-2)
Both fissile and fertile particles with the same type coatings are used in the rods, except the one designated SHPBI.	
Second letter—type of binder	P = Plyophen 5023S phenolic resin (70 wt-%)
	F = furfuryl alcohol polymer (70 wt-%)
Third letter—type of filler	G = GP-38 graphite flour (30 wt-%)
	B = blend of GP-38 (20 wt-%) and Barneby-Cheney 589 charcoal powder (10 wt-%)
Fourth letter—type of process	I = injection-molding process
	W = wet-molding process

(b) For this rod, a combination of SiC-coated fissile particles (S Type 1) and PyC-coated fertile particles (HTI) were used.

Various evaluations were made on samples of the rods produced, including metallography, dimensional measurements, gamma scanning, compressive strength measurements, and fission-product release measurements.

Metallographic sections were made of a number of rods that were typical of those put in the element; these are shown in Figs. 25 through 37. Refer to Table 16 for rod composition coding and figure number for each type of rod.

M22836-1

75×

Fig. 25. Rod No. 260; HPBI 1.00 in. long; HTI pyrocarbon-coated fissile and fertile particles overcoated with Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22834-2

75x

Fig. 26. Rod No. 197; HPBI 5.57 in. long; HTI pyrocarbon-coated fissile and fertile particles overcoated with Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22833-2

75×

Fig. 27. Rod No. 9; HPBI annular 5.93 in. long; HTI pyrocarbon-coated fissile and fertile particles overcoated with Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix.

M22835-1

75×

Fig. 28. Rod No. 243; HPBI 11.53 in. long; HTI pyrocarbon-coated fissile and fertile particles overcoated with Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22841-1

75×

Fig. 29. Rod No. 684; HPGI 1.00 in. long; HTI pyrocarbon-coated fissile and fertile particles overcoated with Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—30% graphite powder filler matrix

M22839-1

75×

Fig. 30. Rod No. 295; LPBI 1.00 in. long; LTI pyrocarbon-coated fissile and fertile particles overcoated with $2\text{ }\mu\text{m}$ of HTI coating and Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22837-1

75×

Fig. 31. Rod No. 274; LPBI 5.57 in. long; LTI pyrocarbon-coated fissile and fertile particles overcoated with 2 μm of HTI coating and Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22838-1

75×

Fig. 32. Rod No. 283; LPBI 11.53 in. long; LTI pyrocarbon-coated fissile and fertile particles overcoated with $2\mu\text{m}$ of HTI coating and Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22840-1

75×

Fig. 33. Rod No. 678; LPGI 1.00 in. long; LTI pyrocarbon-coated fissile and fertile particles overcoated with $2\mu\text{m}$ of HTI coating and Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—30% graphite powder filler matrix

M22487-1

75x

Fig. 34. Rod No. 437; HFGW 11.53 in. long; HTI pyrocarbon-coated fissile and fertile particles overcoated with wax film mold release in a wet-molded fuel rod with a 67% furfuryl alcohol resin binder—33% graphite powder filler matrix

M22486-2

75×

Fig. 35. Rod No. 425; LFGW 5.57 in. long; LTI pyrocarbon-coated fissile and fertile particles overcoated with $2\mu\text{m}$ of HTI coating and wax film mold release in a wet-molded fuel rod with a 67% furfuryl alcohol resin binder—33% graphite powder filler matrix

M28843-1

75×

Fig. 36. Rod No. 712; SPBI 1.00 in. long; SiC-pyrocarbon Type 1 - coated fissile and fertile particles over-coated with $2\mu\text{m}$ of HTI coating and Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder—20% graphite powder—10% charcoal powder filler matrix

M22842-1

75×

Fig. 37. Rod No. 694; TPBI 1.00 in. long; SiC-pyrocarbon Type 2 coated fissile and fertile particles over-coated with $2\mu\text{m}$ of HTI coating and Spraygraph mold release in an injection-molded fuel rod with a 70% phenolic resin binder-20% graphite powder-10% charcoal powder filler matrix

In general, the metallographic pictures show the good releasing characteristics of the overcoating treatments on the particles, which allows the matrix to shrink away from the particles during carbonization. This release of the matrix from the particle coating prevents gross stresses from being built up and possible coating damage as the matrix changes during carbonization. There appears to be no difference in the matrix structure or matrix-to-particle bond between rods of different lengths and containing different particle types. Some difference can be noted between rods of different matrix composition.

The desired dimensional tolerances for the final fuel rods were as follows:

<u>Average Length (in.)</u>	<u>Average Diameter (in.)</u>
1.00 ± 0.005	0.468 ± 0.002
5.57 ± 0.008	0.468 ± 0.002
5.93 ± 0.008	0.515 ± 0.002
11.53 ± 0.010	0.468 ± 0.002

Each fuel rod was measured and weighed after the cure and carbonization stages. Figure 38 shows the measurement procedures used and typical values obtained for a rod as they appear in the rod data records. All of the average rod lengths and diameters were within the required tolerances. The dimensional data were also included in the data retrieval computer program mentioned earlier.

The total enriched uranium content and loading variations within the rod of several typical fuel rods was checked by a gamma counting technique developed by FOD. The rods are positioned in a rotating and indexing fixture modified to accept dual detector probes located diametrically opposite each other. Glass tubes are used to contain the rods for counting to prevent any possible damage to the rods and cross contamination.

ROD NO.	AFTER CURE DIMENSIONS					AFTER CARBONIZATION DIMENSIONS				
		⊥		LENGTH	WT.		⊥		LENGTH	WT.
187	S	.4651	.4648			S	.4682	.4692		
	M	.4649	.4648	1.023	5.72	M	.4681	.4690	1.024	5.49
	B	.4648	.4647			B	.4678	.4687		

Fig. 38. Typical dimensioning procedure used on each fuel rod

The apparatus is calibrated with standards of known enriched uranium content. The uranium loading of the test rods can then be determined from the counts obtained from that rod. For loading variation within the rods, the 11.53-in. rods were counted at four places along the rod, whereas the 5.57-in. rods were counted at three places. The results for the FOD wet-molded rods are given in Table 17 and those for the Metallurgy injection-molded rods are given in Table 18.

The compressive strength of various typical proof test element fuel rods was determined by loading 1-in.-long specimens axially to failure in an Instron test machine. The specimens had 1/8-in. graphite discs cemented on each end as bearing surfaces. For 1-in.-long rods, the whole rod was used as the specimen, but for longer rods, 1-in. segments were broken off. The results are listed in Table 19. In general, the compressive strength values were low, although the rods apparently had enough strength to survive handling and loading in the element. Also, it is believed the rods had enough strength to generally hold together as chunks when fractured rather than completely disintegrating into individual particles.

3. 5. FISSION-PRODUCT RELEASE EVALUATION OF FUEL RODS

The steady-state release (R/B) of noble fission gases from several sample fuel rods (1 in. long) was measured at 1000°C in the Mark I TRIGA reactor. These data are given in Table 20 and are plotted in Figs. 39 through 44 as a function of half-life.

Generally, the release from the fuel rods exceeds the release from the particles from which they were fabricated by one-half of one order of magnitude. Examination of the data in Figs. 39 through 44 shows that

1. For fuel rods that contain BISO-coated particles (rods No. 296, 679, 685, and 261), the release from the fissile particles exceeds the release from the fertile particles and appears to be the controlling factor in the release from the rod.

TABLE 17

URANIUM LOADING IN FOD WET-MOLDED FUEL RODS DETERMINED BY GAMMA COUNTING

Rod No.	Rod Type	Rod Length (in.)	Average Counts per Min. per Rod Section	Per- cent Spread in Counts	Percent Deviation from Standard Uranium Loading at Location along Rod				
					1	2	3	4	Average
383	LFGW	11.53	260,800	14.38	-3.5	-2.5	Nominal	+7.0	Nominal
393	LFGW	11.53	261,200	7.66	-1.0	-2.0	+2.0	+2.5	+0.5
420	LFGW	5.57	260,800	6.36	+1.5	+2.0	-3.0	--	Nominal
436	LFGW	5.57	256,200	6.17	-4.0	Nominal	Nominal	--	-1.5
443	LFGW	11.53	265,700	6.32	Nominal	+3.5	+2.0	+2.5	+2.0
444	LFGW	11.53	259,400	5.71	-1.0	Nominal	-1.0	+1.0	Nominal
445	LFGW	11.53	260,200	7.49	-2.5	Nominal	+1.0	+2.0	Nominal
448	HFGW	11.53	264,600	9.56	-2.0	+1.0	+3.5	+3.5	+1.5
449	HFGW	5.57	261,900	8.78	-3.0	+2.0	+3.0	--	+0.5
450	HFGW	5.57	281,100	15.55	-1.0	+3.0	+3.0	--	+1.5
451	HFGW	11.53	291,300	5.60	+4.0	+3.0	+6.0	+9.0	+5.5

TABLE 18

URANIUM LOADING IN METALLURGY INJECTION-MOLDED FUEL RODS DETERMINED BY GAMMA COUNTING

Rod No.	Rod Type	Rod Length (in.)	Average Counts per Min. per Rod Section	Per- cent Spread in Counts	Percent Deviation from Standard Uranium Loading at Location along Rod				
					1	2	3	4	Average
228	HPBI	11.53	260,400	2.38	+0.5	+1.0	+0.5	-1.0	Nominal
229	HPBI	11.53	258,000	1.51	-1.0	-0.5	Nominal	-1.5	-1.0
230	HPBI	11.53	259,000	2.01	+0.5	-1.0	Nominal	-1.0	-0.5
231	HPBI	11.53	260,700	1.04	Nominal	+0.5	+0.5	Nominal	Nominal
233	HPBI	11.53	262,300	5.95	-2.0	Nominal	+3.0	+2.5	+1.0
234	HPBI	11.53	261,600	4.13	+1.5	+1.5	+1.0	-2.0	+0.5
235	HPBI	11.53	252,800	4.23	-1.0	-2.0	-4.0	-3.0	-2.5
236	HPBI	11.53	259,100	1.04	-1.0	-0.5	Nominal	Nominal	-0.5
239	HPBI	11.53	258,500	5.11	+1.0	Nominal	-3.5	Nominal	-0.5
249	HPBI	11.53	257,200	1.21	-1.0	-1.0	-0.5	-1.0	-1.0

TABLE 19

COMPRESSIVE STRENGTH OF TYPICAL PROOF-TEST-ELEMENT
FUEL RODS

Rod No.	Type of Rod ^(a)	Original Rod Length (in.)	Rod Test Specimen Length (in.)	Rod Diameter (in.)	Compressive Load (lb)	Compressive Strength (psi)
263	HPBI	1.010	1.010	0.471	59	340
264	HPBI	1.008	1.008	0.469	78	450
265	HPBI	1.002	1.002	0.468	57	330
297	LPBI	1.021	1.021	0.469	72	415
298	LPBI	1.018	1.018	0.469	82	475
299	LPBI	1.021	1.021	0.469	70	405
696	TPBI	1.020	1.020	0.469	55	320
697	TPBI	1.026	1.026	0.469	65	375
714	SPBI	1.041	1.041	0.469	58	335
715	SPBI	1.036	1.036	0.469	76	440
687-a	LPGI	11.517	1.380	0.468	109	635
687-b	LPGI	11.517	1.250	0.468	94	545
687-c	LPGI	11.517	1.060	0.468	98	570
700-a	TPBI	11.519	1.250	0.467	91	530
700-b	TPBI	11.519	1.250	0.467	70	410
700-c	TPBI	11.519	1.380	0.467	95	555
243-a	HPBI	11.518	1.250	0.468	63	365
243-b	HPBI	11.518	1.130	0.468	65	380
243-c	HPBI	11.518	1.060	0.468	41	240
283-a	LPBI	11.516	1.250	0.467	147	860
283-b	LPBI	11.516	1.060	0.467	225	1315
283-c	LPBI	11.516	1.250	0.467	157	920
197-a	HPBI	5.618	1.000	0.468	153	890
197-b	HPBI	5.618	0.940	0.468	136	790
197-c	HPBI	5.618	0.880	0.468	170	990

(a) See Table 16 for rod composition.

TABLE 20
STEADY-STATE FISSION-GAS RELEASE (R/B) OF 1-IN.-LONG FUEL ROD SAMPLES AT 1000°C

Rod No.	Rod Type	Fissile Particle	Fertile Particle	Kr ^{85m}	Kr ⁸⁸	Kr ⁸⁷	Kr ⁸⁹	Xe ¹³⁸	Xe ¹³⁹
296	LPBI	XETK-LTI-CO2	XTK-LTI-CO3	2.7×10^{-4}	2.2×10^{-4}	1.9×10^{-4}	4.9×10^{-5}	6.6×10^{-5}	2.2×10^{-5}
679	LPGI	XETK-LTI-CO2	XTK-LTI-CO3	2.4×10^{-4}	2.3×10^{-4}	1.6×10^{-4}	1.1×10^{-4}	1.3×10^{-4}	5.3×10^{-5}
685	HPGI	XETK-HTI-CO1	XTK-HTI-CO2	1.1×10^{-3}	6.5×10^{-4}	6.2×10^{-4}	3.2×10^{-4}	4.0×10^{-4}	1.3×10^{-5}
695	TPBI	XETK-4596	XTK-4640	3.5×10^{-4}	2.6×10^{-4}	2.2×10^{-4}	5.1×10^{-5}	8.3×10^{-5}	5.9×10^{-5}
713	SPBI	XETK-4639	XTK-4657	1.2×10^{-4}	5.0×10^{-5}	4.8×10^{-5}	4.8×10^{-5}	6.7×10^{-5}	1.5×10^{-5}
261	HPBI	XETK-HTI-CO1	XTK-HTI-CO2	5.5×10^{-4}	7.6×10^{-4}	5.1×10^{-4}	3.6×10^{-5}	6.2×10^{-5}	1.6×10^{-5}

Fig. 39. Gaseous fission-product release data from XETK-LTI-CO2 and XTK-LTI-CO3 fuel particles and from fuel rod No. 296 at 1000°C

Fig. 40. Gaseous fission-product release data from XETK-LTI-CO2 and XTK-LTI-CO3 fuel particles and from fuel rod No. 679 at 1000°C

Fig. 41. Gaseous fission-product release data from XETK-HTI-CO1 (at 1400°C) and XTK-HTI-CO2 fuel particles and from fuel rod No. 685 at 1000°C

Fig. 42. Gaseous fission-product release data from XETK-4596 and XTK-4640 fuel particles and from fuel rod No. 695 at 1000°C

Fig. 44. Gaseous fission-product release data from XETK-HTI-CO1 (at 1400°C) and XTK-HTI-CO2 fuel particles and from fuel rod No. 261 at 1000°C

2. For fuel rods that contain TRISO-coated particles (rods No. 695 and 713), the release from the fertile particles exceeds the release from the fissile particles in the given rod by about one order of magnitude and may be the controlling factor in the release from the rod. *
3. The steady-state release from the fuel rods generally shows a dependence on half-life ($t_{1/2}$) of the fission nuclide, with an approximate slope of $\sqrt{t_{1/2}}$.

In an effort to determine the reason for the increase in release on fabrication of fuel rods from particles, rod No. 261 was subjected to a low-temperature, selective oxidation process[†] to recover the fuel particles from the rod. This process oxidizes the carbonaceous matrix while leaving the coatings on the particles intact. The mixture of fissile and fertile particles in rod No. 261 was recovered by this process and subjected to another fission-gas release measurement. These data are also shown in Fig. 44. If fuel particles were damaged either in the rod fabrication process or in the destructive oxidation, an increase in fission-gas release (above that of the unbonded particles) would be expected. The data in Fig. 44 show a release even higher than that of the fuel rod from which the particles were obtained, indicating at least some damage to the particles during the debonding operation. It is not possible to say whether any damage occurred during rod fabrication.

* Whether this hypothesis is true or not depends on the relative number of fissions in the fertile vis-a-vis the fissile particles and/or the contaminant, if any (perhaps uranium), in the fertile particles.

[†]Carried out by D. W. Stevens of the Metallurgy Department.

4. "PIGGYBACK" IRRADIATION SPECIMENS

"Piggyback" samples of various fuel and graphite materials were prepared for irradiation in the proof test element so that the behavior of various component materials can be compared with that of the composite fuel rods and graphite elements. Also, the performance of these materials can be determined in an HTGR flux spectrum rather than in a water test reactor spectrum.

The samples being tested are:

1. Loose coated particles of each of the nine types being used in the fuel rods,
2. HTI PyC, LTI PyC, and SiC coatings, both restrained and unrestrained,
3. Matrix material of each of the four types being used in the fuel rods, and
4. Graphite samples of current interest for the Public Service Company of Colorado (PSC) reactor.

4.1. DESCRIPTION OF SPECIMENS

4.1.1. Coated Particles

Each of the nine types of coated particles used in the test element fuel rods are also being tested separately as piggyback samples. These include the LTI-BISO, HTI-BISO, and two types of SiC-BISO on fissile and fertile particles. The samples are being tested as loose coated particles contained in individual graphite crucibles. Descriptions of the coated-particle samples are given in Table 21.

TABLE 21

DESCRIPTION OF COATED-PARTICLE SAMPLES BEING TESTED AS PIGGYBACK SAMPLES IN
FORT ST. VRAIN PROOF TEST ELEMENT

Batch Number	COATING										Particle	
	Buffer		Inner Isotropic			SiC Thick- ness (μm)	Outer Isotropic			Total Thick- ness (μm)		
	Thick- ness (μm)	Den- sity (g/ cm ³)	Thick- ness (μm)	Den- sity (g/ cm ³)	BAF		Thick- ness (μm)	Den- sity (g/ cm ³)	BAF		Type	Diam. (μm)
XETK-HTI-CO1	62	1.03	--	--	--	--	61	1.88	1.13	121	(Th, U)C ₂	189
XETK-LTI-CO2	62	1.03	--	--	--	--	58	1.90	1.03	130	(Th, U)C ₂	189
XTK-HTI-CO2	70	1.03	--	--	--	--	72	1.87	1.13	140	ThC ₂	395
XTK-LTI-CO3	68	1.05	--	--	--	--	76	1.98	1.03	145	ThC ₂	395
XTK-HTI-4581	68	1.00	--	--	--	--	83	1.90	1.05	151	ThC ₂	395
XETK-4596	62	1.03	34	1.92	1.03	13	8	(a)	(a)	113	(Th, U)C ₂	189
HETK-4639	62	1.03	--	--	--	15	48	1.93	1.04	127 ^(b)	(Th, U)C ₂	189
HTK-4640	74	0.96	33	1.89	1.10	13	26	1.91	(a)	146	ThC ₂	397
XTK-4657	70	1.07	--	--	--	15	54	1.77	1.20	139	ThC ₂	397

NOTE: In many cases the values in this table are average values. Complete details on coated particles may be found in Section 2.

(a) To be determined.

(b) Includes an additional 7- μm overcoating on the outer isotropic coating.

4.1.2. Pyrocarbon and Silicon Carbide Strips

The three types of coatings applied to the fuel particles in the test element are HTI-PyC, LTI-PyC, and SiC. These coatings are being tested as piggyback samples in the form of unrestrained strips: 12 HTI-PyC strips, 4 LTI-PyC strips, and 12 SiC strips. Also included are 4 HTI-PyC restrained samples and 1 annealed pyrographite sample. A summary of these samples is given in Table 22.

4.1.3. Matrix Material

One sample of each of the four types of matrix material used in the test element fuel rods is being tested as a piggyback specimen. The four samples were all made by the injection-molding process, and they include both Plyophen and furfuryl alcohol/maleic anhydride binders and both graphite and graphite-charcoal fillers. A description of the samples is given in Table 23. A photograph of the four specimens is shown in Fig. 45.

4.1.4. Graphite Samples

Four graphites of current interest (H327B, RC4, TS688, and 9567) for PSC are being tested as piggyback samples. Samples cut from both the parallel and perpendicular extrusion directions are being tested for each graphite. The samples are located in two zones of the element (see Section 4.3), and all samples are being tested in both zones (with the exception of RC4-parallel and TS-688-parallel, which are being tested in zone 3 only). The samples in zone 3 are rods approximately 1/8 in. in diameter and 1/2 in. long; those in zone 2 are discs approximately 1/2 in. in diameter and 1/8 in. thick. A detailed description of the graphite samples is given in Table 24.

4.2. LOCATION OF SPECIMENS

The piggyback samples are in two locations in the proof test element: fuel zone 3 and fuel zone 2. The samples in zone 3 are located in

TABLE 22

DESCRIPTION OF PyC AND SiC STRIPS BEING TESTED AS PIGGYBACK
SAMPLES IN THE FORT ST. VRAIN PROOF TEST ELEMENT

Speci- men Number	Coating Run Number	Density (g/cm ³)	BAF	Remarks
41-1	HTK-4641	2.030	1.22	} High-temperature ISO PyC 40-mil-wide strips
41-2	HTK-4641	2.033		
41-3	HTK-4641	2.024		
41-4	HTK-4641	2.018		
7-1	XTK 4527	1.926	1.02	} Low-temperature ISO PyC 40-mil-wide strips
7-2	XTK-4527	1.920		
7-3	XTK-4527	1.920		
7-4	XTK-4527	1.925		
1-1	XTK-4581	1.897	1.05	} High-temperature ISO PyC 40-mil-wide strips
1-2	XTK-4581	1.892		
1-3	XTK-4581	1.897		
1-4	XTK-4581	1.898		
1-5	XTK-4581	1.899		
1-6	XTK-4581	1.898		
1-7	XTK-4581	1.891		
1-8	XTK-4581	1.896		
1	ANNPGR	2.246		A-direction annealed 3000 °C for 35 min + 3150 °C for 30 min + 3300 °C for 5 min
41-4-R	HTK-4641		1.22	} Restrained HTI speci- mens
41-3-R	HTK-4641			
41-2-R	HTK-4641			
41-1-R	HTK-4641			
- - -	3662-111			12 SiC strips 40 mil wide

TABLE 23
DESCRIPTION OF MATRIX MATERIALS BEING TESTED AS PIGGYBACK SAMPLES IN
FORT ST. VRAIN PROOF TEST ELEMENT

Sample No.	Matrix	Outside Diameter (in.)	Length (in.)	Weight (g)	Method of Manufacture
GA3734-123-1	FA/MA ^(a) 30% graphite	0.1508-0.1514	0.4890	0.1260	Injection molded
GA3734-123-3	Plyophen 70 wt-% 5023 10% charcoal 20% graphite	0.1522-0.1530	0.4892	0.1799	Injection molded
GA3734-123-6	Plyophen 70 wt-% 5023 30 wt-% graphite	0.1378-0.1453	0.4602	0.1440	Injection molded
GA3734-123-8	FA/MA 10% charcoal 20% GP38 graphite	0.1502-0.1519	0.4947	0.1384	Injection molded

(a) FA/MA is furfuryl alcohol/maleic anhydride.

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(4x)

Fig. 45. Four specimens of matrix materials being tested as piggyback samples: (1) Plyophen binder-graphite filler (sample 3734-123-6); (2) Plyophen binder-graphite-charcoal filler (sample 3734-123-3); (3) furfuryl binder-graphite filler (sample 3734-123-1); (4) furfuryl binder-graphite-charcoal filler (sample 3734-123-8)

TABLE 24
DESCRIPTION OF GRAPHITE BEING TESTED AS PIGGYBACK
SAMPLES IN FORT ST. VRAIN PROOF TEST ELEMENT

Specimen No. (a)	Extrusion Direction	Type of Graphite	Weight (g)	Diameter (in.)	Thickness (in.)
Rod A	Parallel	RC4	0.2390	0.146	0.501
Rod B	Parallel	RC4	0.2394	0.146	0.501
Rod C	Perpendicular	RC4	0.2369	0.146	0.496
Rod D	Perpendicular	RC4	0.2365	0.146	0.492
Rod E	Parallel	H327B	0.2479	0.147	0.500
Rod G	Perpendicular	H327B	0.2485	0.147	0.500
Rod I	Parallel	TS688	0.2391	0.147	0.500
Rod K	Perpendicular	TS688	0.2384	0.147	0.500
Rod M	Parallel	9567	0.2499	0.146	0.507
Rod O	Perpendicular	9567	0.2400	0.146	0.507
Disc C	Perpendicular	RC4	0.5526	0.440	0.129
Disc E	Parallel	H327B	0.5740	0.441	0.129
Disc G	Perpendicular	H327B	0.5756	0.441	0.129
Disc K	Perpendicular	TS688	0.5475	0.440	0.129
Disc M	Parallel	9567	0.5669	0.441	0.129
Disc O	Perpendicular	9567	0.5575	0.440	0.129

(a) Rod samples are in zone 3; disc samples are in zone 2.

the center of annular fuel rods in hole 7. This zone contains all four types of samples described above. The samples are 1/8 in. in diameter and occupy a total length of 17 in. They will be irradiated at a temperature of 1150° to 1200°C, which will be monitored by a nearby thermocouple, and they will receive a fast neutron dose of about $1.3 \times 10^{21} \text{ n/cm}^2$ ($E > 0.18 \text{ MeV}$). A loading map of samples in this zone is given in Fig. 46.

Fig. 46. Piggyback-sample loading map for zone 3 (hole 7) of Fort St. Vrain proof test element

The samples in zone 2 are discs, which are inserted between fuel rods in hole 2. These discs are made of the various graphites described above, and each is nominally $1/2$ in. in diameter and $1/8$ in. thick. They will be irradiated at a center temperature of 1000° to 1150°C , with a radial gradient decreasing to 600° to 700°C at the edge. The samples will receive approximately the same fast dose as in zone 3.

5. LOADING AND ASSEMBLY OF PROOF TEST ELEMENT

The Capsule Irradiation Group's responsibilities for the PSC proof test element were primarily in the following areas: (1) loading of the fuel, (2) assembly of the element, (3) placement of thermocouples within the element, and (4) loading of the assembled element into the shipping container. To obtain adequate data to properly complete these various steps, it was necessary to perform the following tasks and investigations:

1. Dosimetry experiment;
2. Thermocouple experiment (cycling of a test piece);
3. Cement-curing experiment;
4. Pressure buildup experiment (dissociation of the polyethylene padding when heated in a closed vessel).
5. Padding compression test;
6. Assembly of a dummy element;
7. Design the assembly jig, the shipping container, and the element support within the shipping container;
8. Trucking of the element within the container (rough-ride test) and drop testing the shipping container;
9. Fuel loading and slide photography;
10. Assembly of element sections;
11. Loading of the element into the shipping container.

5.1. DOSIMETRY EXPERIMENT

Initial plans were to install within the element the same kind of dosimetry normally used in irradiation capsules. For standard dosimetry, this consists of enclosing one length each of pure Fe, pure Ti, and Ni-0.4% Co wire in a supersil quartz vial back-filled with a partial helium

gas pressure. Special dosimetry is composed of a pure V, V-Fe⁵⁴ and a V-Co wire.

In a capsule, the dosimetry is located in a cool region where the temperature does not exceed 300°F. This experiment was initiated to test the compatibility between the supersil quartz and graphite at the expected operating temperature of ~1000°F.

Four vials of each kind of dosimetry were placed within drilled holes in a graphite block. The assembly was then heated to 1000°F in an atmosphere of purified flowing helium gas for a period of 30 days.

Examination showed that the standard dosimetry wires were very brittle and had undergone a loss in diameter. All of the vials for the standard dosimetry were coated with a white deposit; some of the vials were broken. The special dosimetry vials appeared grey in color; some of these vials were also broken and all of them were bent. The wires were fused together; in some cases, one of the wires was partially gone, and in others completely gone. Spectrochemical analysis results showed a reaction between the wires and the container. Because of the lack of a suitable container, it was decided not to include dosimetry in the proof test element.

5.2. THERMOCOUPLE EXPERIMENT

A 4-3/4-in.-long piece of thermocouple, including the hot-junction end, was made by the Continental Sensing Company in accordance with GGA specifications. This thermocouple was then subjected to the cycling test described below.

Disassembly revealed that the swaged hot-junction slug measured 0.061 in. in diameter by 0.088 in. long. The wires are 0.020 in. in diameter.

A simple cycling test was set up. It consisted of 25 cycles from ~375°F to 2000°F, an examination of the thermocouple, and then 25 more cycles.

The cycling of the hot junction was performed in a vacuum using a 10-kW induction unit. Immediately upon reaching the high temperature, heating was ceased and purified argon gas was introduced for the cool down. When the cool down had been achieved, the cycle was reversed. Each cycle took 3-1/2 min.

The thermocouple survived the cycling and a visual examination showed no change in the appearance of the sheath or the thermoelements.

5.3. CEMENT-CURING EXPERIMENT

The cement recommended for assembly of the proof test element sections was Grade P-514 carbon cement made by the Great Lakes Carbon Corporation. The recommended curing to 260°F at 15°F/hr increase could not be applied because the Styrofoam plug inserted on top of each of the fuel-rod stacks as a motion restrainer lose their integrity at a temperature of 180°F. It was thus necessary to test the joints after a cure of 160°F.

Two mating graphite sections were supplied to perform the test. Thermometer holes were drilled to reach the roots of the threads. The male and female threads were cleaned by brushing with a stiff brush to remove loose particles. Both threads were coated with a primer and allowed to dry for 20 min. The P-514 cement was then brushed on both threads and the two pieces of graphite were assembled. The small amount of cement that had worked its way to the surface was removed. A heating tape, connected to a variable transformer, was wrapped around the joint and the tape and the element were then covered with a fiberglass blanket. In addition, four spots of P-514 cement were applied to a flat surface on the graphite — two were applied over a primer-coated surface and two on an unprimed surface. Since it would be difficult to examine the condition of the threaded surfaces, these spots would give an indication of the condition of the cement. The assembly was then heated to a temperature of 155°F and held there for 24 hr.

Examination of the spots at the end of the test showed that the primed spots were somewhat tackier than the unprimed spots. Scraping with a sharp object showed tenacious adhesion in both instances.

To test the integrity of the joint, one-half of the cemented section was held in a vise and an estimated 1500 in.-lb of force was applied to the other half. There was no indication of unthreading of the joint. It was concluded that the cement was sufficiently cured to prevent unscrewing during shipment. Final cure and carburization of the cement will occur when the element reaches operating temperature in the reactor.

5.4. PRESSURE BUILDUP TEST

Expanded polyethylene foam with a density of 2 lb/ft³ was selected as the cradle material for the shipment of the proof test element. Originally, full foam support was to be provided. However, if the shipping container (8 in. OD schedule 40 pipe 148 in. long) is exposed to a temperature of 1475°F, the foam dissociates and the full foam support builds up excessive pressure within the pipe. (The limits are specified by the Atomic Energy Commission.) To determine the extent of disassociation and the resulting pressure, a pressure buildup test was performed. The test consisted of filling a piece of Type 304 stainless steel tubing, 1 in. OD by 0.065 in. thick wall, with the foam, heating it to a temperature of 1475°F, and observing the rise in pressure. The highest pressure attained was 950 psi at 1475°F.

Disassembly after cooling showed only a sooty deposit on the tubing wall. A small Fiberfrax plug that had been included at one end of the pipe also disassociated and resulted in a glassy deposit on the tubing wall. Other than the deposits on the wall, the tube was empty.

Based on this experiment, calculations showed that the maximum allowable foam content should be 3.2 lb. The Project recommended using 2.8 lb. The design of the cradle was so adjusted that 2.5 lb of the foam were used.

5.5. PADDING COMPRESSION TEST

To test the resiliency of the expanded polyethylene foam, three lead bricks, each weighing ~26 lb, were placed on pieces of the foam. The resulting pressures were 3.25 psi, 1.68 psi, and 0.84 psi. The assemblies were left in this condition for a period of 24 hr. Examination showed that a definite impression remained in the 3.25 psi and the 1.68 psi cases, but this gradually disappeared after the removal of the bricks.

The total surface of foam in the lower half of the cradle in contact with the element amounted to 334.5 in.², and one flat of the element is in contact with 111.5 in.². The weight of element plus the handling tool is ~100 lb. The pressure with all of the surfaces in contact with the element is equal to 0.334 psi, and the pressure with only one flat in contact with the element is equal to 1.003 psi.

It was concluded that the foam would support the element without setting permanently.

5.6. THE DUMMY ELEMENT

In the development of fabrication techniques for making the graphite parts, many pieces were made that were not suitable for the end product. Nevertheless, the best of them were selected for a practice (dummy) assembly of a complete element. The practice assembly was used to prove out the assembly jig and assembly techniques and to establish a procedure for removal of the cement from the gas flow passages. In addition, the dummy element was used for the "rough ride" in the shipping container and for performing a drop test of the container.

The following drawings list the pertinent assemblies and show the final designs:

90-SK-2071 - Proof Test Element Assembly Fixture

90-SK-2209 - Jig Assembly for the Proof Test Element

90-SK-2309 - Shipping Cask Assembly

90-SK-2354 - Proof Test Fuel Element and Shipping Cask Assembly

5.7. TESTING OF THE SHIPPING CONTAINER

Two tests were performed to ensure the soundness of the foam support, the container, and the shipping crate.

The first test involved loading the dummy element for shipment and trucking it over rough roads. The crate, which had a 4 by 4 base with 1/4-in. plywood sides reinforced with 3/4-in. by 2-1/2 in. strips, was placed in the middle of the truck bed and lashed with four ropes to prevent lateral movement. Nine Shockmaster overload indicators were used to measure overloads due to the roughness of the ride. A 20g and a 50g Shockmaster was fastened to the truck bed; two 10g Shockmasters were attached to the crate floor; and the remainder, rated at 5, 10, 15, 20, 30, and 50g were fastened to the shipping container. The truck was driven over 46 miles of extremely rough roads at varying speeds; frequent inspections were made of the Shockmasters. At the end of the test, none of the indicators had been tripped. The element was removed from its containment and examined for damage. None was found.

The element was then repackaged and the drop test was performed. This test consisted of supporting one end of the loaded crate on a wooden 4 by 4 plank 1 ft high and dropping the crate onto blacktop pavement by driving the plank out from under the crate. This was then repeated for the opposite end of the crate. Examination of the element after this test revealed no damage.

5.8. FUEL LOADING

Because of the known weaknesses of the fuel rods, two ground rules were established regarding the loading of the fuel: (1) holes would be loaded only when full hole loadings were available and (2) the fuel rods would not be handled unless absolutely necessary. For this latter reason, two double troughs of the required height to slip-feed a fuel rod into its hole were made. A handling sequence was adopted to minimize the possibility of accidental dropping or otherwise damaging the fuel rods. Three areas

were prepared with masslin cloth covering to handle the enriched fuel. The fuel rods were emptied from the glass tubes in which they were received by sliding them directly into the troughs. The rods were then identified by sticking a numbered tag to the trough alongside the fuel rod. If the tag protruded outward of the trough it indicated that the numbered end was photographed; if the tag protruded into the trough it indicated that the opposite end was photographed. Entire fuel rods were not photographed. The intention was to photograph the worst end and to include a representative surface condition.

Once identified, the trough with the fuel was moved to a masslin-covered pan and positioned for photographing it. After it was photographed, the trough was moved to another masslin-covered table prepared for loading the fuel rod into the test element.

Each fuel hole within the graphite sections was visually examined and then cleaned with a special adapter attached to a portable vacuum cleaner.

A flat area, $1/4$ in. wide by 2 in. long, to be used as an orientation mark for hole numbering was made by filing away a selected point between two flats of the hexagonal configuration of the graphite fuel zone. At the end opposite of this mark, $3/16$ in. by $3/16$ in. flats were filed in suitable numbers to denote the zone number. Cutoff marks were scribed circumferentially for post-test separation of the zones in the Hot Cell Facility. Two cuts are required, one to open up the holes at the bottom of each fuel stack and the other above each fuel stack. Figure 47 shows a typical fuel section during the assembly procedure.

As each zone was prepared for loading, the pertinent drawing illustrating the kind of rod make-up required was posted on the wall. The numbers of the fuel rods and their materials were listed and published as the rods were made up. The numbers from these lists were assigned to the proper positions by locating them on the drawing. The loading of a fuel rod into its assigned place was made by one individual and witnessed

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Fig. 47. Assembly of fuel section

by another. As each rod was inserted in place, the identifying tag was lifted from the trough and placed on the drawing opposite the written assigned number as a verification of the correctness of the loading. An arrow pointed to the direction in which the numbered end of the fuel rod's face was inserted into the graphite. This was also marked on the drawing as a part of the record. Drawings No. 90-SK-1996, 90-SK-1997, 90-SK-1998, and 90-SK-1999 locate the loadings and indicate the orientation of each fuel rod. The void-space gap above each fuel stack is also indicated on these drawings. The location of the various fuel rods in the element, the fuel zone, the hole number, and the hole position of each numbered rod are given in Table 25.

All of the holes, with the exception of hole 4 and hole 10 of zone 3, were loaded with carbonized fuel rods. Holes 4 and 10 were loaded with green fuel rods that were carbonized in place. After carbonization, it was determined that the rods in these holes adhered to the walls of the graphite element. Hole 7 in fuel zones 1, 2, and 3 was fitted with annular fuel rods to accommodate a thermocouple. After each of these fuel zones were loaded, alignment of the thermocouple holes was checked by inserting a rod of approximately the same diameter as the thermocouple in the hole. The annular fuel rods, above the termination point of the thermocouple, were loaded with the piggyback samples described in Section 4.

Before each fuel rod was loaded, a graphite "pusher plug" ~0.250 in. long was inserted in each of the full-rod holes. The ultimate use of this plug is to assist in the removal of the fuel rods for post-irradiation examination.

After each hole was fully loaded, a polyethylene foam plug was fitted in the void space between the fuel-rod stack and the graphite top plug. The top plugs were cemented in place with carbon cement P-514. Any excess cement was carefully removed.

5.9. ELEMENT ASSEMBLY AND SHIPPING-CONTAINER LOADING

Prior to the assembly of the element, the handling-tool anchor plug was pushed into place. When all of the zones had been loaded with fuel and capped off by cementing the plugs in place, the threaded portions of the graphite element components were brushed and vacuum cleaned to remove dust and particles. All of the threads were coated with the primer and the P-514 cement was applied to both male and female threads just prior to assembling the sections. Immediately after assembling any two sections together, a wooden dowel with a cheesecloth-covered end was passed through the gas holes of each joint to remove any cement that had extruded into them. The gas passages were then examined visually as each section was added. The thermocouple holes were checked for alignment after each addition was made by inserting a rod of approximately the same diameter as the thermocouple.

The assembly was started by joining the bottom-most piece to the transition piece and was continued by using the assembly fixture in Drawing No. 90-SK-2071 and 90-SK-2209 until fuel zone 3 was in place. At that point, the two tungsten-3% rhenium/tungsten-26% rhenium thermocouples were installed in the element. Thermocouple response, resistance, and thermoelement identification were determined prior to installation. The installation was made so that the positive and negative leg of the elements aligned with the proper contacts. Joining of the thermoelements and the contacts was made by tungsten inert-gas welding. The screws holding the contact assembly were firmly seated and tack-welded to prevent loosening. The element area at the thermocouple hot junction was gently heated with a hair dryer and the thermocouple response checked with a potentiometer. After ensuring that both thermocouples were operating properly, fuel zone 4 and the top reflector were added to complete the unit.

TABLE 25

LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nom-inal Length of Rod (in.)
1	1	1	629	LPBI	1.00
1	1	2	646	LPBI	11.53
1	2	1	630	LPBI	1.00
1	2	2	647	LPBI	11.53
1	3	1	631	LPBI	1.00
1	3	2	648	LPBI	11.53
1	4	1	632	LPBI	1.00
1	4	2	649	LPBI	11.53
1	5	1	633	LPBI	1.00
1	5	2	650	LPBI	11.53
1	6	1	634	LPBI	1.00
1	6	2	651	LPBI	11.53
1	7	1	16	LPBIA	5.93
1	7	2	114	LPBIA	5.93
1	8	1	680	HPGI	1.00
1	8	2	224	HPBI	11.53
1	9	1	635	LPBI	1.00
1	9	2	663	LPBI	11.53
1	10	1	636	LPBI	1.00
1	10	2	664	LPBI	11.53
1	11	1	637	LPBI	1.00
1	11	2	666	LPBI	11.53
1	12	1	638	LPBI	1.00
1	12	2	668	LPBI	11.53

TABLE 25 (continued)

LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nom- inal Length of Rod (in.)
2	1	1	640	LPBI	5.57
2	1	2	665	LPBI	11.53
2	1	3	686	LPGI	11.53
2	2	1	420	LFGW	5.57
2	2	2	393	LFGW	11.53
2	2	3	383	LFGW	11.53
2	3	1	259	HPBI	1.00
2	3	2	293	LPBI	1.00
2	3	3	258	HPBI	1.00
2	3	4	257	HPBI	1.00
2	3	5	292	LPBI	1.00
2	3	6	677	LPGI	1.00
2	3	7	683	HPGI	1.00
2	3	8	256	HPBI	1.00
2	3	9	254	HPBI	1.00
2	3	10	676	LPGI	1.00
2	3	11	639	LPBI	1.00
2	3	12	627	LPBI	1.00
2	3	13	253	HPBI	1.00
2	3	14	252	HPBI	1.00
2	3	15	657	LPBI	1.00
2	3	16	662	LPBI	1.00
2	3	17	251	HPBI	1.00

TABLE 25 (continued)

LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nominal Length of Rod (in.)
2	3	18	250	HPBI	1.00
2	3	19	208	HPBI	1.00
2	3	20	207	HPBI	1.00
2	3	21	206	HPBI	1.00
2	3	22	661	LPBI	1.00
2	3	23	660	LPBI	1.00
2	3	24	659	LPBI	1.00
2	3	25	682	HPGI	1.00
2	3	26	675	LPGI	1.00
2	3	27	205	HPBI	1.00
2	3	28	658	LPBI	1.00
2	4	1	449	HFGW	5.57
2	4	2	451	HFGW	11.53
2	4	3	448	HFGW	11.53
2	5	1	708	SPBI	5.57
2	5	2	717	SPBI	11.53
2	5	3	716	SPBI	11.53
2	6	1	641	LPBI	5.57
2	6	2	290	LPBI	11.53
2	6	3	689	LPGI	11.53
2	7	1	17	LPBIA	5.93
2	7	2	19	LPBIA	5.93
2	7	3	24	LPBIA	5.93

TABLE 25 (continued)
LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nom-inal Length of Rod (in.)
2	7	4	25	LPBIA	5.93
2	7	5	23	LPBIA	5.93
2	8	1	294	LPBI	1.00
2	8	2	436	LFGW	5.57
2	8	3	444	LFGW	11.53
2	8	4	443	LFGW	11.53
2	9	1	667	LPBI	11.53
2	9	2	669	LPBI	11.53
2	9	3	291	LPBI	1.00
2	9	4	642	LPBI	5.57
2	10	1	643	LPBI	5.57
2	10	2	236	HPBI	11.53
2	10	3	190	HPBI	1.00
2	10	4	703	SHPBI	11.53
2	11	1	720	SPBI	11.53
2	11	2	719	SPBI	11.53
2	11	3	711	SPBI	1.00
2	11	4	707	SPBI	5.57
2	12	1	699	TPBI	11.53
2	12	2	698	TPBI	11.53
2	12	3	693	TPBI	1.00
2	12	4	704	TPBI	5.57
3	1	1	193	HPBI	5.57

TABLE 25 (continued)

LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nom-inal Length of Rod (in.)
3	1	2	199	HPBI	11.53
3	1	3	201	HPBI	11.53
3	2	1	202	HPBI	11.53
3	2	2	195	HPBI	5.57
3	2	3	203	HPBI	11.53
3	3	1	222	HPBI	11.53
3	3	2	223	HPBI	11.53
3	3	3	196	HPBI	5.57
3	4	1	644	HPBI	5.57
3	4	2	652	HPBI	11.53
3	4	3	653	HPBI	11.53
3	5	1	226	HPBI	11.53
3	5	2	198	HPBI	5.57
3	5	3	235	HPBI	11.53
3	6	1	228	HPBI	11.53
3	6	2	244	HPBI	11.53
3	6	3	212	HPBI	5.57
3	7	1	8	HPBIA	5.93
3	7	2	7	HPBIA	5.93
3	7	3	12	HPBIA	5.93
3	7	4	11	HPBIA	5.93
3	7	5	5	HPBIA	5.93
3	8	1	213	HPBI	5.57

TABLE 25 (continued)

LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nom-inal Length of Rod (in.)
3	8	2	209	HPBI	1.00
3	8	3	230	HPBI	11.53
3	8	4	231	HPBI	11.53
3	9	1	216	HPBI	5.57
3	9	2	188	HPBI	1.00
3	9	3	234	HPBI	11.53
3	9	4	233	HPBI	11.53
3	10	1	255	HPBI	1.00
3	10	2	654	HPBI	11.53
3	10	3	655	HPBI	11.53
3	10	4	645	HPBI	5.57
3	11	1	245	HPBI	11.53
3	11	2	218	HPBI	5.57
3	11	3	191	HPBI	1.00
3	11	4	239	HPBI	11.53
3	12	1	247	HPBI	11.53
3	12	2	249	HPBI	11.53
3	12	3	192	HPBI	1.00
3	12	4	219	HPBI	5.57
4	1	1	281	LPBI	11.53
4	2	1	280	LPBI	11.53
4	3	1	282	LPBI	11.53
4	4	1	289	LPBI	11.53

TABLE 25 (continued)
LOCATION OF THE VARIOUS FUEL RODS IN PROOF TEST ELEMENT

Fuel Zone No.	Fuel Hole No.	Rod Position in Hole from Bottom to Top	Rod No.	Type Rod	Nom-inal Length of Rod (in.)
4	5	1	284	LPBI	11.53
4	6	1	285	LPBI	11.53
4	7	1	628	LPBI	1.00
4	7	2	286	LPBI	11.53
4	8	1	674	LPGI	1.00
4	8	2	688	LPGI	11.53
4	9	1	681	HPGI	1.00
4	9	2	690	LPGI	11.53
4	10	1	204	HPBI	1.00
4	10	2	248	HPBI	11.53
4	11	1	710	SPBI	1.00
4	11	2	287	LPBI	11.53
4	12	1	692	TPBI	1.00
4	12	2	288	LPBI	11.53

The six joints were then cured, three each on two successive days, using the techniques described earlier. A higher curing temperature (170°F) was attainable because of the change from Styrofoam plugs, which collapse at 180°F, to polyethylene plugs, which start to collapse at 210°F.

The handling tool (GGA Dwg. No. 90-SK-1988) was installed in the fuel element and locked in place.

The foam shipping support consisted of four lengths of 1-in. aluminum schedule 40 pipe equipped with 3-1/4-in. -long aluminum spacers made of 1-1/4-in. schedule 40 pipe. Polyethylene foam pads die cut to

the proper configuration were inserted between the spacers. The support is made up in two halves. One half of the foam shipping support (GGA Dwg. No. 90-SK-2354) was positioned to receive the element. The element was lifted by four individuals and placed in this cradle. A protective cap (GGA Dwg. No. 90-SK-2314) was placed over the thermocouple contact points and secured to the cradle with glass-reinforced tape. The second half of the cradle was positioned and the two halves were bound together with glass-reinforced tape.

The shipping container (Dwg. No. 90-SK-2309) is constructed of a piece of 8-in. schedule 40 pipe that is threaded on both ends and equipped with fork-lift brackets, supports, and eye bolts for horizontal and vertical lifting.

A length of 3-ft-wide Kraft paper was fed through the 8-in. pipe and an equal length was allowed to extend out of one end. The element in its foam support was wrapped in the protruding piece of paper. When all was ready, one individual pulled on the Kraft paper at one end of the 8-in. pipe while four individuals lifted and slid the cradle into the pipe at the other end.

Once the element was in place, the excess paper was removed and discarded, the padded pipe caps were attached, wire-lock holes were drilled, and the caps were wire-locked in place. Both ends of the pipe were enclosed with a polyethylene bag and sealed with tape.

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