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**Performance of HTGR Fuel
in HFIR Capsule HT-33**

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IN HFIR CAPSULE HT-33

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ABSTRACT

Irradiation capsule HT-33 was a cooperative effort between General Atomic Company (GA) and Oak Ridge National Laboratory (ORNL). In this capsule ThO_2 particles (fabricated by GA), low-enriched uranium particles, inert carbon particles, and various fuel rod matrices were tested under accelerated irradiation in the High-Flux Isotope Reactor. Our report covers only the ORNL portion of the experiment.

Visual examination showed good irradiation behavior for fuel rods with slug-injected matrices (using a pitch binder) and warm-molded matrices (using a thermosetting resin binder). Rod debonding improved somewhat with fuel rods that used GLCC H-451 ground graphite shim particles rather than Speer fluid coke shim particles.

Measurements of permeability (by inert gas intrusion) of the pyrocarbon on the inert particles showed that the disorder created by the neutron flux did not increase the inert gas permeability.

Metallographic examination of Triso-coated particles irradiated both with and without an outer pyrocarbon coating revealed that the outer coating is necessary to suppress SiC degradation at temperatures above approximately 1375°C.

The fission product behavior (determined by the electron microprobe) was similar in both low-enriched and high-enriched uranium particles made from weak-acid resins. Furthermore, fission product palladium caused severe SiC corrosion at time-averaged temperatures above 1400°C.

INTRODUCTION

Irradiation capsule HT-33 was a cooperative effort between General Atomic Company (GA) and the Oak Ridge National Laboratory (ORNL) to test High-Temperature Gas-Cooled Reactor (HTGR) fuel. Our report describes the physical design of the total experiment, the rationale for the ORNL objectives, and the performance of ORNL-made fuel. General Atomic has issued a preirradiation report¹ covering their portion of the experiment.

The HT-33 capsule was designed as a five-cycle experiment in the High-Flux Isotope Reactor (HFIR) with two temperature zones, 900 and 1250°C,

measured at the surface of the graphite particle holders. The capsule contained 52 positions: 32 positions for loose-coated-particle holders, which were occupied by loose Biso- and Triso-coated ThO_2 particles manufactured by GA, and 20 positions occupied by ORNL rods containing low-enriched uranium (LEU; 7.073 wt % ^{235}U) particles for heat production during the early portion of the irradiation. In previous HT capsules specimens in the driver particle positions were used for heat generation only and were discarded as useless after irradiation. However, in HT-33 experimental objectives were identified that could be met by use of the LEU particle positions. This was done with no loss of fertile particle test space and without interference with the primary test objectives. The LEU particles later became an important part of the experiment when the federal government changed its policy on the use of high-enriched uranium (HEU).

The ORNL design objectives were as follows:

1. Comparison of a slug-injected (SI) matrix with a pitch binder and a warm-molded (WM) matrix with a thermosetting resin binder.
2. Comparison of two types of shim particles in SI specimens.
3. Comparison of Triso-coated and SiC-coated (no outer LTI coating) particles in WM specimens and Triso-coated particles in SI specimens.
4. Irradiation of inert carbon coated particles for testing of inert gas permeability. This data was added to existing results on the effect of irradiation on permeability.

As mentioned previously, the interest in LEU fuel prompted another objective: To evaluate the performance of LEU fuel particles and to compare it with HEU fuel particles irradiated in other experiments.

Our report covers only the above objectives, which were conducted by ORNL in the LEU particle positions of the HT-33 experiment.

DESCRIPTION OF EXPERIMENT

The design of the HT-33 capsule was identical to that of capsules HT-12, -13, -14, and -15.² A schematic of the capsule design is shown in Fig. 1. The test matrix shown in Table 1 was selected to simulate the maximum loading estimated for equilibrium recycle fuel elements in a large

ORNL-DWG 75-2595R

Fig. 1. The HT-33 Capsule Design, 1 in. = 25.4 mm.

Table 1. Test Matrix for Irradiation Capsule HT-33

Item	Fabrication Method (Binder)	Particle Type	Volume % Loading	
			1250°C	900°C
1, 5	Slug injection (pitch)	TRISO fissile	17	12
		TRISO inert	23	28
		Shim (GLC H-451)	18	18
			<u>58</u>	<u>58</u>
2, 6	Slug injection (pitch)	TRISO fissile	17	12
		TRISO inert	23	28
		Shim (Speer Fluid coke)	18	16
			<u>58</u>	<u>58</u>
3, 7	Warm molding (resin)	TRISO fissile	17	12
		TRISO inert	23	28
			<u>40</u>	<u>40</u>
4, 8	Warm molding (resin)	SiC fissile	12	8
		SiC inert	28	32
			<u>40</u>	<u>40</u>

HTGR. Many other loadings will be required for the entire fuel cycle scheme, but this experiment was designed to obtain a direct comparison between the shimmed slug-injected matrices and the warm-molded continuous matrices with identical heavy-metal loadings. Characteristics of the coated particles and the shim particles used in the driver rod specimens are shown in Tables 2 and 3, respectively. The shapes of the shim particles are shown in Fig. 2. The Triso-coated particles were annealed at 1800°C for 10 min before incorporation into the bonded specimens; the SiC-coated particles were used as coated. The shim particles and graphite filler flour (H-451*) were heat-treated at 2800°C for purification. The H-451 shim particles were impregnated with pyrolytic carbon to reduce open porosity and increase strength. The Speer[†] shim was used as heat-treated. The loading scheme and heavy-metal loadings for the ORNL specimens are shown in Tables 4 and 5, respectively.

*Manufactured by Great Lakes Carbon Corp.

†Manufactured by Speer Carbon Co.

Table 2. Characteristics of Coated Particles in Driver Rods for Capsule HT-33

	Type Particles ^a					
	Fissile		Inerts			
	TRISO (OR-2576H)	SiC (SC-375)	TRISO (OR-2577H)	SiC (SC-376)	BISO (OR-2293-HT)	BISO (OR-2294-HT)
Kernel						
Stoichiometry	WAR ^b IRC-72 U-C _{4.40} O _{1.43}	WAR ^b IRC-72 U-C _{4.40} O _{1.43}	Carbon	Carbon	Carbon	Carbon
Nominal Conversion, %	30	30				
Diameter, μ m	376.1 (10.10)	376.1 (10.10)	484.7 (19.1)	484.7 (19.1)	451.2 (27.6)	459.8 (29.3)
Density, g/cm ³	3.26	3.26	1.31	1.31	1.33	1.33
Enrichment, %	7.073	7.073				
Buffer						
Thickness, μ m	53.7 (7.02)	53.7 (7.02)	72.9 (8.42)	72.9 (8.42)	83.2 (10.2)	81.4 (9.6)
Density, g/cm ³	1.12	1.12	0.75	0.75	1.14	1.13
Inner LTI						
Thickness, μ m	34.57 (3.20)	34.57 (3.20)	17.3 (2.07)	17.3 (2.07)	90.2 (5.6)	86.9 (4.3)
Density, g/cm ³	1.939 (0.007)	1.939 (0.007)	ND	ND	ND	ND
SiC						
Thickness, μ m	35.6 (1.50)	35.6 (1.50)	68.8 (2.45)	68.8 (2.45)		
Density, g/cm ³	3.200 (0.00066)	3.200 (0.00066)	3.197 (0.0012)	3.197 (0.0012)		
Coating Rate, μ m/min	0.228	0.228	0.275	0.275		
Outer LTI						
		None		None		
Thickness, μ m	37.18 (2.60)		38.6 (2.86)			
Density, g/cm ³	2.020 (0.0046)		2.028 (0.004)			
Coating Rate, μ m/min	3.8		4.8			

^aNumbers in parentheses are standard deviations. ND - not determined.

^bWAR - weak-acid resin.

Table 3. Characteristics of Shim Particles for HT-33

Material	Size Range (μm)	Bulk Density ^a (g/cm ³)	Open Porosity ^b (%)
Speer fluid coke ^c	840-1000	1.92	10.54
GLCC H-451 ^d	710-1190	1.94	8.99

^aDetermined by mercury porosimetry at 0.52 MPa (75 psi).

^bDetermined by mercury porosimetry at 0.03 MPa (15,000 psi).

^cFrom Speer Carbon Company; heat-treated at 2800°C for 1 h.

^dGround H-451 graphite from Great Lakes Carbon Corporation; heat-treated at 2800°C for 1 h and impregnated at ORNL with MAPP gas.

The inert and LEU particles were fabricated into rod specimens as shown in Table 1. Basically, the slug-injection technique for fabrication rod specimens requires that the dry coated particles (and shim when used) be confined in a fixed-volume mold while the hot viscous matrix material (185°C) is forced through the bed of particles (4.83 MPa, 700 psi). The matrix is then solidified, and the rod specimens are ejected from the mold. A 32 wt % H-451 graphite filler (-325 mesh) and 68 wt % Ashland A-240* binder are used to fabricate the HT-33 slug-injected rods. In warm-molded fabrication particles and matrix material are mixed and then formed into rods with a die set. The HT-33 experiment rods are fabricated with 58.74 wt % H-451 graphite powder (-200 mesh), 27.97 wt % Varcum binder,† 10.49 wt % Thermax carbon black,‡ and 2.8 wt % maleic anhydride (used as a catalyst). Both the slug-injected and warm-molded rods were heated to 900°C (at 90°C/h) and then heated to 1800°C for 0.5 h in packed GLCC-1008 natural flake graphite to remove all the volatiles before use. The data for the ORNL specimens used in HT-33 are summarized in Table 6.

*Manufactured by Ashland Oil Co.

†Manufactured by Allied Chemical Co.

‡Manufactured by R. T. Vanderbilt Co.

Fig. 2. Shim Particles Used in Irradiation Experiment HT-33. (a) GLCC H-451. (b) Speer fluid coke.

Table 4. Loading Scheme for Capsule HT-33

Capsule Position ^a	Type Specimen ^b	Item ^c	Fuel (Heavy Metal)	Batch Number	Type Coating
1-T ^d	IN			OR-2293-HT	BISO
1	SI	5	C	OR-2576	TRISO
2	CA		Th	6542-39-0161-001	BISO
3	SI	6	U	OR-2576	TRISO
4	CA		Th	6542-40-0161-001	BISO
5	CA		Th	6542-40-0261-901	BISO
6	WM	7	U	OR-2576	TRISO
7	CA		Th	6542-40-0260-001	BISO
8	CA		Th	6542-27-0161-001	BISO
9	WM	8	U	SC-375	SiC
10	CA		Th	6542-29-0261-001	BISO
11	CA		Th	6542-41-0161-001	BISO
12	SI	5	U	OR-2576	TRISO
13	CA		Th	6542-41-0160-001	BISO
1-B ^d	IN			OR-2293-HT	BISO
2-T ^d	IN			OR-2293-HT	BISO
14	SI	1	U	OR-2576	TRISO
15	CA		Th	6542-29-0261-002	BISO
16	WM	4	U	SC-375	SiC
17	CA		Th	6542-27-0161-002	BISO
18	CA		Th	6542-41-0161-002	BISO
19	WM	3	U	OR-2576	TRISO
20	CA		Th	6542-41-0160-002	BISO
21	CA		Th	6542-39-0161-002	BISO
22	SI	2	U	OR-2576	TRISO
23	CA		Th	6542-40-0161-002	BISO
24	CA		Th	6542-40-0261-002	BISO
25	SI	1	U	OR-2576	TRISO
26	CA		Th	6542-40-0260-002	BISO
2-B ^d	IN			OR-2293-HT	BISO
RMP					
3-T ^d	IN			OR-2294-HT	BISO
27	CA		Th	6252-08-0161-004	TRISO
28	WM	3	U	OR-2576	TRISO
29	CA		Th	6252-10-0161-004	TRISO
30	CA		Th	6252-05-0160-008	TRISO
31	WM	3	U	OR-2576	TRISO
32	CA		Th	6252-06-0261-004	TRISO
33	CA		Th	6252-06-0161-004	TRISO
34	SI	2	U	OR-2576	TRISO
35	CA		Th	6252-07-0261-004	TRISO
36	CA		Th	6252-07-0161-004	TRISO
37	SI	1	U	OR-2576	TRISO
38	CA		Th	6252-09-0161-004	TRISO
39	WM	4	U	SC-375	SiC
3-B ^d	IN			OR-2294-HT	BISO
4-T ^d	IN			OR-2294-HT	BISO
40	CA		Th	6252-09-0161-003	TRISO
41	SI	6	U	OR-2576	TRISO
42	CA		Th	6252-07-0161-003	TRISO
43	CA		Th	6252-07-0161-003	TRISO
44	WM	7	U	OR-2576	TRISO
45	CA		Th	6252-05-0160-007	TRISO
46	CA		Th	6252-06-0261-003	TRISO
47	WM	8	U	SC-375	SiC
48	CA		Th	6252-06-0161-003	TRISO
49	CA		Th	6252-08-0161-003	TRISO
50	SI	5	U	OR-2576	TRISO
51	CA		Th	6252-10-0161-003	TRISO
52	WM	7	U	OR-2576	TRISO
4-B ^d	IN			OR-2294-HT	BISO

^aTop of capsule.^bSI - slug-injected, WM - warm-molded, IN - inert particles. All CA specimens are loose particles.^cSee Table 1.^dLoose coated inert particles located in end plug positions. For pyrocarbon permeability studies.

Table 5. Heavy-Metal Loadings for Capsule HT-33

Capsule Position ^a	Contents, g			Number of Thorium Particles
	Total Uranium ^b	²³⁵ U	Thorium	
1	0.0191	0.0014		
2			0.0233	38
3				
4	0.0191	0.0014	0.0234	41
5			0.0234	41
6				
7	0.0191	0.0014	0.0234	41
8			0.0232	40
9				
10	0.0191	0.0014	0.0236	39
11			0.0235	40
12				
13	0.0191	0.0014	0.0234	40
14				
15				
16	0.0268	0.0019	0.0332	55
17				
18			0.0330	57
19	0.0268	0.0019	0.0329	56
20			0.0329	56
21			0.0331	54
22	0.0268	0.0019		
23			0.0331	58
24			0.0331	58
25	0.0268	0.0019		
26			0.0331	58
Horizontal midplane				
27	0.0268	0.0019	0.0331	80
28				
29			0.0333	79
30	0.0268	0.0019	0.0333	51
31				
32			0.0330	77
33	0.0268	0.0019	0.0330	78
34				
35			0.0330	70
36	0.0268	0.0019	0.0332	80
37				
38			0.0332	78
39	0.0268	0.0019		
40			0.0226	53
41				
42	0.0191	0.0014	0.0224	53
43			0.0224	54
44				
45	0.0191	0.0014	0.0224	34
46			0.0223	52
47				
48	0.0191	0.0014	0.0225	53
49			0.0224	54
50				
51	0.0191	0.0014	0.0223	53
52				

^aTop of capsule.^bAll uranium 7.073% enriched.

Table 6. Properties of ORNL Specimens in Capsule HT-33

Capsule Position	Item ^a	Average OD		Average Length		Weight (g)	Density, Mg/m ³		Particle Loading, Vol %		Coke Yield (%)
		(mm)	(in.)	(mm)	(in.)		Matrix	Total	Particles ^a	Shim ^a	
1	5	9.7282	0.3830	6.8839	0.2711	0.7079	0.61	1.38	40.56	14.08	20.63
3	6	9.7409	0.3835	6.8250	0.2687	0.7031	0.60	1.38	40.16	14.55	19.46
6	7	9.7358	0.3833	6.8859	0.2711	0.8437	1.37	1.65	39.66		40.85
9	8	9.7536	0.3840	7.1704	0.2823	0.8777	1.34	1.64	38.10		39.59
12	5	9.7511	0.3839	6.8148	0.2683	0.7013	0.59	1.38	40.11	14.82	18.97
14	1	9.7409	0.3835	6.6878	0.2633	0.6902	0.58	1.38	40.92	13.69	20.37
16	4	9.7535	0.3840	7.1806	0.2827	0.8786	1.32	1.64	38.03		42.49
19	3	9.7434	0.3836	7.0917	0.2792	0.8716	1.36	1.65	39.17		43.51
22	2	9.7561	0.3841	6.6040	0.2600	0.6824	0.55	1.38	41.31	13.55	18.66
25	1	9.7409	0.3835	6.6954	0.2636	0.6913	0.58	1.39	40.87	13.66	18.12
28	3	9.7460	0.3837	7.0561	0.2778	0.8685	1.37	1.65	38.81		45.44
31	3	9.7434	0.3836	7.0587	0.2779	0.8738	1.38	1.66	38.62		44.17
34	2	9.7307	0.3831	6.4668	0.2546	0.6693	0.53	1.39	42.38	13.91	16.24
37	1	9.7460	0.3837	6.6827	0.2631	0.6853	0.55	1.37	40.91	13.68	16.47
39	4	9.7561	0.3841	7.1526	0.2816	0.8740	1.31	1.63	38.25		40.18
41	6	9.7282	0.3830	6.8072	0.2680	0.6956	0.57	1.37	40.23	14.65	18.69
44	7	9.7104	0.3823	6.7662	0.2467	0.8694	1.41	1.67	39.06		44.32
47	8	9.7536	0.3840	7.1298	0.2807	0.8777	1.36	1.65	38.30		41.07
50	5	9.7384	0.3834	6.9012	0.2717	0.7019	0.58	1.37	39.70	14.69	17.94
52	7	9.7358	0.3833	7.0333	0.2769	0.8656	1.39	1.65	38.82		43.64

^aSee Table 1.

CAPSULE OPERATION

Capsule HT-33 was inserted into HFIR target position G-5 on June 17, 1976, and removed October 21, 1976, after having operated for 2759 h at 100-MW reactor power. The reactor operating history is shown in Table 7. The capsule was then transferred to the hot cells for subsequent post-irradiation examination. The thermal and fast fluences and burnups for the capsule are presented in Tables 8 and 9, respectively.

Table 7. Reactor Operating History During Irradiation of Capsule HT-33

Cycle	Begin		End		Irradiation Time, h	
	Time	Date	Time	Date	During Cycle	Accumulated
1 ^a	0152	6-17-76	0800	7-9-76	534	534
2	0355	7-10-76	2350	8-1-76	547	1081
3	1655	8-2-76	0400	8-25-76	540	1621
4	1223	9-2-76	2400	9-25-76	564	2185
5	0645	9-27-76	0400	10-21-76	574	2759

^aThe first cycle of operation for the HT-33 capsule was HFIR cycle 134.

Table 8. Thermal and Fast Neutron Fluxes and Fluences for Capsule HT-33

Capsule Position	Flux $n/m^2 s$		Fluence n/m^2	
	Thermal (<0.414 eV)	Fast (>0.18 MeV)	Thermal	Fast
1, 52	1.44×10^{19}	5.60×10^{18}	1.43×10^{26}	5.56×10^{25}
2, 51	1.50	5.95	1.49	5.91
3, 50	1.56	6.40	1.55	6.36
4, 49	1.62	6.65	1.61	6.61
5, 48	1.68	6.95	1.67	6.90
6, 47	1.74	7.30	1.73	7.25
7, 46	1.80	7.60	1.79	7.55
8, 45	1.86	7.90	1.85	7.85
9, 44	1.90	8.19	1.89	8.14
10, 43	1.94	8.50	1.93	8.44
11, 42	2.00	8.80	1.99	8.74
12, 41	2.06	9.09	2.05	9.03
13, 40	2.10	9.30	2.09	9.24
14, 39	2.36	10.37	2.34	10.30
15, 38	2.41	10.57	2.39	10.50
16, 37	2.46	10.77	2.44	10.70
17, 36	2.56	10.97	2.48	10.90
18, 35	2.55	11.07	2.53	11.00
19, 34	2.58	11.17	2.56	11.10
20, 33	2.62	11.28	2.60	11.20
21, 32	2.65	11.38	2.63	11.30
22, 31	2.68	11.48	2.66	11.40
23, 30	2.72	11.58	2.70	11.50
24, 29	2.75	11.58	2.73	11.50
25, 28	2.77	11.68	2.75	11.60
26, 27	2.80	11.68	2.78	11.60

Table 9. Burnups for Specimens
in Capsule HT-33

Capsule Position	Burnup, % FIMA		
	^{235}U	^{238}U	^{232}Th
1, 52	84.7	10.4	
2, 51			5.6
3, 50	84.7	11.7	
4, 49			6.2
5, 48			6.5
6, 47	84.7	13.7	
7, 46			7.1
8, 45			7.5
9, 44	84.7	15.4	
10, 43			8.1
11, 42			8.5
12, 41	84.7	17.0	
13, 40			9.1
14, 39	84.7	20.3	
15, 38			11.0
16, 37	84.7	21.0	
17, 36			11.5
18, 35			11.7
19, 34	84.7	21.9	
20, 33			12.1
21, 32			12.2
22, 31	84.7	22.5	
23, 30			12.5
24, 29			12.6
25, 28	84.7	23.0	
26, 27			12.8

THERMAL ANALYSIS

Time-temperature histories for the GA loose ThO_2 particles irradiated in magazines 1 through 4 are presented in Figs. 3 through 10, respectively. We calculated temperature histories by using the HTCAP1 thermal modeling computer code (modified version of the HTCAP computer code³). Each figure describes the maximum particle surface temperature, graphite holder annulus temperature, and power generated per particle as a function of time from the beginning of irradiation. The computer code calculations indicate that the irradiation temperatures were not constant; the discontinuities occurred between each cycle change. Only the last cycle had the most constant temperature.

We also calculated time-temperature histories for the ORNL fuel rods containing the LEU weak-acid resin (WAR) driver particles with the same computer code used for the loose particles. Time-averaged centerline and surface temperatures for each cycle are given in Table 10. The temperatures were high during the first cycle when the ^{235}U was being burned up. Later in irradiation the primary source of heat was from the fissioning of the "bred-in" ^{239}Pu .

Fig. 3. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 1 of HT-33 Capsule. (a) Holder 2, batch 39-0161-001, 821- μm -diam particle. (b) Holder 4, batch 40-0161-001, 828- μm -diam particle. (c) Holder 5, batch 40-0261-001, 832- μm -diam particle. (d) Holder 7, batch 40-0260-001, 829- μm -diam particle.

Fig. 4. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 1 of HT-33 Capsule. (a) Holder 8, batch 27-0161-001, 823- μm -diam particle. (b) Holder 10, batch 29-0261-001, 843- μm -diam particle. (c) Holder 11, batch 41-0161-001, 825- μm -diam particle. (d) Holder 13, batch 41-0161-001, 827- μm -diam particle.

Fig. 5. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 2 of HT-33 Capsule. (a) Holder 15, batch 29-0261-002, 833- μm -diam particle. (b) Holder 17, batch 27-0161-002, 829- μm -diam particle. (c) Holder 18, batch 41-0161-002, 823- μm -diam particle. (d) Holder 20, batch 41-0160-002, 817- μm -diam particle.

Fig. 6. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 2 of HT-33 Capsule. (a) Holder 21, batch 39-0161-002, 826- μm -diam particle. (b) Holder 23, batch 40-0161-002, 827- μm -diam particle. (c) Holder 24, batch 40-0261-002, 828- μm -diam particle. (d) Holder 26, batch 40-0260-002, 826- μm -diam particle.

Fig. 7. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 3 of HT-33 Capsule. (a) Holder 27, batch 08-0161-004, 836- μm -diam particle. (b) Holder 29, batch 10-0161-004, 818- μm -diam particle. (c) Holder 30, batch 05-0161-008, 870- μm -diam particle. (d) Holder 32, batch 06-0261-004, 816- μm -diam particle.

Fig. 8. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 3 of HT-33 Capsule. (a) Holder 33, batch 06-0161-004, 824- μm -diam particle. (b) Holder 35, batch 07-0261-004, 814- μm -diam particle. (c) Holder 36, batch 07-0161-004, 821- μm -diam particle. (d) Holder 38, batch 09-0161-004, 826- μm -diam particle.

Fig. 9. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 4 of HT-33 Capsule. (a) Holder 40, batch 09-0161-003, 824- μm -diam particle. (b) Holder 42, batch 07-0261-003, 813- μm -diam particle. (c) Holder 43, batch 07-0161-003, 826- μm -diam particle. (d) Holder 45, batch 05-0160-007, 872- μm -diam particle.

Fig. 10. Calculated Temperature and Power Histories for Loose ThO_2 Particles Irradiated in Magazine 4 of HT-33 Capsule. (a) Holder 46, batch 06-0261-003, 812- μm -diam particle. (b) Holder 48, batch 06-0161-003, 818- μm -diam particle. (c) Holder 49, batch 08-0161-003, 836- μm -diam particle. (d) Holder 51, batch 10-0161-003, 826- μm -diam particle.

Table 10. Time-Averaged Centerline and Surface Temperatures for ORNL Fuel Rods from HT-33^a

Fuel Rod	Time-Averaged Centerline Temperature, °C					Time-Averaged Surface Temperature, °C				
	Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5	Cycle 1	Cycle 2	Cycle 3	Cycle 4	Cycle 5
1	1265	1150	1122	1120	1114	1119	1048	1034	1036	1032
3	1312	1202	1188	1196	1190	1160	1097	1096	1110	1103
6	1278	1200	1198	1211	1203	1215	1155	1158	1172	1165
9	1290	1210	1210	1225	1215	1221	1163	1168	1183	1175
12	1391	1273	1270	1283	1270	1209	1150	1158	1174	1164
14	1588	1422	1407	1408	1392	1331	1250	1249	1255	1245
16	1510	1423	1433	1450	1434	1408	1353	1368	1387	1374
19	1542	1455	1466	1483	1465	1436	1383	1398	1417	1402
22	1684	1542	1545	1558	1536	1406	1354	1370	1390	1374
25	1712	1570	1574	1587	1564	1430	1378	1396	1415	1399
28	1585	1502	1514	1529	1513	1475	1426	1443	1460	1446
31	1558	1473	1485	1501	1484	1449	1399	1415	1433	1418
34	1689	1545	1548	1563	1541	1418	1362	1379	1399	1383
37	1651	1506	1505	1523	1504	1388	1330	1345	1366	1352
39	1443	1336	1330	1334	1321	1343	1268	1267	1273	1261
41	1411	1291	1286	1299	1286	1229	1168	1174	1191	1180
44	1295	1225	1226	1242	1233	1234	1181	1186	1203	1195
47	1261	1183	1179	1191	1184	1197	1139	1139	1152	1146
50	1314	1202	1187	1196	1186	1162	1097	1095	1107	1100
52	1201	1117	1099	1099	1093	1143	1077	1063	1065	1060

^aCycle 1 - 34 h, Cycle 2 - 547 h, Cycle 3 - 540 h, Cycle 4 - 564 h, Cycle 5 - 574 h. Total - 2759 h.

POSTIRRADIATION EXAMINATION

Disassembly

The capsule appeared to be in excellent condition when received at the hot cells. A gas sample from the capsule (the capsule was filled with argon at 1 atm and sealed before irradiation) was analyzed by gamma scanning and mass spectrometry. The results are presented in the Appendix. Although argon was the major constituent, significant quantities of helium, xenon, and krypton were measured. Helium was probably in the glovebox (with argon) and was introduced into the capsule just before it was sealed. The xenon and krypton result from fission gas release from the fuel. Calculations yield a release-to-birth ratio (R/B) of 0.13 for the total xenon and 0.35 for the total krypton. Such high R/B values may be caused by the poor performance of the SiC-coated driver particles (i.e., without outer LTI coatings), to be discussed in a later section.

We had no difficulty opening the capsule and removing the four magazines. The end plugs, which contained the coated inert particles for permeability measurements, were removed, and the particles were retrieved. The low-temperature magazines, 1 and 4, yielded their fuel rods and graphite crucibles with no difficulty. However, we had considerable difficulty with the high-temperature magazines, 2 and 3; eventually, we had to slit magazine 2 to recover the specimens. This difficulty is not reflected in the dimensional data but must be attributable to debonding of the heater rods 14, 22, and 25, as discussed in the following section.

We removed the crucibles containing the GA fertile particles intact and later opened them in the presence of GA personnel. The particles were repackaged and shipped to GA for detailed examination.

Visual Examination

After removal from magazines the rods were visually examined with the stereomicroscope. The results are summarized in Table 11. The rods that remained intact are shown in Figs. 11 and 12. These indicate that the integrity of the warm-molded rods containing the Triso-coated particles

Table 11. Stereoexamination of Heater Rods from HT-33

Position ^a	Fabrication ^b Process	Type Shim	Shim Loading (vol %)	Appearance
1	SI	H-451	14.08	Good
3	SI	Speer	14.55	Few cracked particles
6	WM(Triso coated)	None		Some matrix cracking
9	WM(SiC coated)	None		Some debonding
12	SI	H-451	14.82	Good
14	SI	H-451	13.69	Complete debonding
16	WM(SiC coated)	None		Slight debonding
19	WM(Triso coated)	None		Good
22	SI	Speer	13.55	Complete debonding
25	SI	H-451	13.66	Complete debonding
28	WM(Triso coated)	None		Some debonding
31	WM(Triso coated)	None		Good
34	SI	Speer	13.91	Some debonding
37	SI	H-451	13.68	Good
39	WM(SiC coated)	None		Few cracked particles
41	SI	Speer	14.65	Some debonding
44	WM(Triso coated)	None		Good
47	WM(SiC coated)	None		Some debonding
50	SI	H-451	14.69	Good
52	WM(Triso coated)	None		Good

^aPositions 1 through 12 — magazine 1, 900°C; positions 14 through 25 — magazine 2, 1250°C; positions 28 through 39 — magazine 3, 1250°C; positions 41 through 52 — magazine 4, 900°C.

^bWM — warm-molded; SI — slug-injected.

Fig. 11. Fuel Rods from (a) Magazine 1, (b) Magazine 3, and (c) Magazine 4 in HT-33. SI - slug-injected; WM - warm-molded.

Fig. 12. Intact Fuel Rods from Magazine 2 of HT-33. (a) Rod 16.
(b) Rod 19.

was better than the warm-molded rods containing the SiC-coated particles (i.e., without an outer LTI coating). The difference must be caused by the bonding of the outer LTI coating and the matrix.

The slug-injected fuel rods performed adequately, except in magazine 2 where they experienced complete debonding. Less debonding occurred with the slug-injected rods that contained the H-451 shim particle than the Speer shim particle. We have not yet determined why the slug-injected rods from magazine 2 experienced complete debonding while similar rods in magazine 3 (irradiated under similar conditions) showed little debonding.

Dimensional Analysis

We inspected each intact fuel rod by measuring the diameters at 0° and 90° with a dial gage comparator and V-block assembly. We also determined a maximum length. The results are given in Table 12. Generally the length shrank, but some rods expanded slightly. However, it should be noted that the lengths are more difficult to measure and therefore more susceptible to errors.

Table 12. Dimensional Changes in Heater Rods for HT-33

Position	Type Specimen ^a	Dimension Change, %		Temperature (°C)	Fast (>0.18 MeV) Neutron Fluence ^b (n/m ²)
		Diameter	Length		
1	SI(H-451 shim)	-1.44	+1.45	900	5.56 × 10 ²⁵
3	SI(Speer shim)	-1.07	-1.99	900	6.36
6	WM(Triso-coated particles)	-1.38	-3.90	900	7.25
9	WM(SiC-coated particles)	-0.57	-5.76	900	8.14
12	SI(H-451 shim)	-0.57		900	9.03
16	WM(SiC-coated particles)	-1.51	-6.03	1250	10.7
19	WM(Triso-coated particles)	-1.04	-0.59	1250	11.1
28	WM(Triso-coated particles)	-1.82	+0.45	1250	11.6
31	WM(Triso-coated particles)	-2.24	-2.97	1250	11.4
34	SI(Speer shim)	-1.23		1250	11.1
37	SI(H-451 shim)	-1.12	-1.92	1250	10.7
39	WM(SiC-coated particles)	-0.99	+0.16	1250	10.3
44	WM(Triso-coated particles)	-0.92	+0.33	900	8.14
47	WM(SiC-coated particles)	-0.10	+0.48	900	7.25
50	SI(H-451 shim)	-1.41	-3.30	900	6.36
52	WM(Triso-coated particles)	-1.85	-2.12	900	5.56

^aSI - slug-injected; WM - warm-molded.^bBased on operating history of capsule.

Pyrocarbon Permeability Measurements

We measured the neon-helium permeability of the inert particles (batch OR-2293-HT) from the top half of the capsule. We did not retrieve a sufficient number of particles from location 2-T during disassembly to obtain an accurate measurement. The results are presented in Table 13. The pyrocarbon permeability decreases with irradiation. Additional studies have also shown similar results.⁴ While the decrease in permeability of pyrocarbon coatings, which have inert kernels, does not prove that coatings with Th- or U-bearing kernels will behave similarly, it does demonstrate that the neutron damage does not disrupt the pyrocarbon structure to cause greater permeability.

Table 13. Measurements of Ne/He Permeability of Inert Particles

Sample	He (pmol)	Ne (pmol)	Approximate Irradiation Temperature (°C)	Fast (>0.18 MeV) Neutron Fluence (n/m ²)	Ne/He	
					As Measured	Normalized for Geometry
Unirradiated	107.0	20.5		0×10^{25}	0.19	0.24
1-T	120.0	11.3	900	5.6	0.09	0.13
1-B	98.7	11.8	900	9.3	0.12	0.13
2-B	59.0	3.3	1250	11.6	0.06	0.04

Metallography and Electron Microprobe Examination

Transverse sections from fuel rods 6, 9, 16, and 19 were metallographically polished. Rods 9 and 16 contained the SiC-coated particles (i.e., without an outer LTI coating), while rods 6 and 19 contained the Triso-coated particles. All were warm-molded.

Examination of rod 9 showed that the SiC-coated particles were in good condition (Fig. 13). The particles were obviously not bonded to the very dense surrounding matrix. The kernel appearance was typical of WAR particles, which show considerable densification of the kernel and buffer coating.⁵

Fig. 13. Typical SiC-Coated Particle from Rod 9 Located on Rod Outer Surface. (a) Bright field. (b) Polarized light. Rod outer surface located to the left of the micrographs. Time-averaged temperature — 1180°C; temperature gradient — 200°C/cm.

The SiC-Coated particles in rod 16 showed severe degradation of the SiC coating (Fig. 14). Degradation of the SiC also occurred with the inert particles (they had no outer LTI either) that were in close proximity to the driver particles (Fig. 15). The degradation is a result of sublimation of the SiC⁶⁻⁸ and was also observed with the SiC-coated driver particles in irradiation experiment HT-31.⁹ Particles located on the rod outer surface experienced nearly complete loss of the SiC (Fig. 16). Evidently, the sublimation rate was higher on the exposed surface. Examination with the electron microprobe showed that the uranium and neodymium remained in the particle (Fig. 17). Only trace amounts of cesium were detected in the particle, indicating that most was released during irradiation. It is believed that these particles were the source of high activity observed in the gas sample previously discussed.

Fig. 14. Typical SiC-Coated Particle from Rod 16 Under Bright Field Illumination. Rod outer surface located 3600 μm to the left. Note severe degradation of SiC coating. Time-averaged temperature - 1445°C; temperature gradient - 75°C/cm.

Fig. 15. SiC-Coated Inert Particle from Rod 16 with SiC Degradation in Proximity to a Fissile Particle that is Located in the Lower Left Corner.

The Triso-coated particles in rods 6 and 19 showed no SiC degradation (Figs. 18 and 19, respectively). These rods operated at comparable temperatures to the rods that contained the SiC-coated particles. Thus it can be concluded that SiC sublimation can be stopped by the addition of the outer LTI coating. However, the results on rod 9 indicate that particles without outer LTI coatings may be operated up to temperatures of about 1400°C (see Table 10) for less than 2 d without serious SiC degradation.

Recent developments in governmental policy, relative to the adoption of proliferation-resistant nuclear fuel cycles, prompted a change in the reference HTGR fuel cycle from one using high-enriched uranium (HEU) to one using enrichments of less than 20% ^{235}U or ^{233}U . These new cycles have been referred to as medium-enriched uranium or MEU (10–20% ^{235}U or ^{233}U) and low-enriched uranium or LEU (less than 10% ^{235}U or ^{233}U). Thus

Fig. 16. SiC-Coated Inert Particle from Rod 16 Located on Rod Outer Surface Showing Nearly Complete Loss of SiC on Outer Surface. Time-averaged temperature — 1380°C ; temperature gradient — $300^{\circ}\text{C}/\text{cm}$.

the examination of the Triso-coated driver particles became of interest since they represented the only irradiated LEU (or MEU) particles available. As mentioned before, these Triso-coated particles suffered no SiC degradation from sublimation.

The Triso-coated particles from rods 6 and 19 (Figs. 18 and 19, respectively) showed densification of the kernel and buffer coating, dispersion of the kernel phases in the densified buffer, and excess carbon from the kernel, which are all typical of WAR particles.^{5,10}

Close examination of the SiC coatings on the particles from fuel rod 19 showed corrosion and penetration into the SiC (Fig. 20). Fission product palladium was identified by the electron microprobe as the cause of the corrosion (Fig. 21). It has been hypothesized that the corrosion takes place along grain boundaries where free silicon may be present and low-melting ($\geq 720^{\circ}\text{C}$) palladium-silicon eutectics are formed. Particles in this rod had time-averaged temperatures greater than 1400°C .

Fig. 17. Electron Microprobe X-Ray Displays of SiC-Coated Particle (Also Shown in Fig. 14) From Rod 16. (a) Backscattered electron image, (b) U Ma, (c) Si Ka, and (d) Nd La.

Fig. 18. Typical Triso-Coated Particle from Rod 6. (a) Bright field. (b) Polarized light. Rod outer surface located 800 μm to the left. Time-averaged temperature - 1185°C; temperature gradient - 155°C/cm.

Fig. 19. Typical Triso-Coated Particle from Rod 19. (a) Bright field. (b) Polarized light. Rod outer surface located 400 μm to the left. Time-averaged temperature - 1420°C; temperature gradient - 285°C/cm.

Fig. 20. Triso-Coated Particle from Rod 19 Showing Corrosion of the SiC. (a) Bright field. (b) Polarized light. Particle located 2200 μm from rod outer surface. Time-averaged temperature - 1460°C; temperature gradient - 170°C/cm.

Fig. 21. Electron Microprobe Identified SiC Corrosion in Rod 19 Caused by Palladium. (a) Back-scattered electron image. (b) Si $L\alpha$ x-ray display, and (c) Pd $L\alpha$ x-ray display. Particle located 2200 μm from outer surface. Time-averaged temperature — 1460°C; temperature gradient — 170°C/cm.

The particles in rod 6 irradiated at lower temperatures (time-averaged temperatures $<1215^{\circ}\text{C}$) than those in rod 19 showed only slight penetration of palladium into the SiC (Fig. 22). Examination of the SiC coatings from all the particles in rods 6 and 19 revealed the relationship between palladium penetration rate and temperature shown in Fig. 23. Comparison with the rate for the lanthanides and SiC^{11,12} shows the palladium to be more of a problem at temperatures less than 1300°C , while the rates are comparable at higher temperatures. Correlating the SiC-Pd reaction rate plotted in Fig. 23 to time in reactor, a particle operating at 1200°C would experience palladium penetration halfway through a $35\text{-}\mu\text{m}$ SiC coating in approximately 879 d of irradiation. It should be emphasized that the irradiation temperatures used for Fig. 23 are calculated and some discrepancy with the actual temperatures may occur. Recent unpublished data¹³ indicate that the SiC-Pd reaction rates are somewhat lower and approximate the SiC-Nd reaction rate.

We also determined the behavior of the other fission products and actinides. In the particles from rod 19 the uranium and plutonium were dispersed throughout the densified buffer (Fig. 24). Chlorine was detected with these concentrations, although the counting rate was very low. The particles in rod 6 did not show as much uranium and plutonium dispersion (Fig. 25); however, some was in the densified buffer. Chlorine was present but the counting rate was too low to obtain an acceptable x-ray display. The greater dispersion in rod 19 results from the higher irradiation temperatures. Other studies¹⁴ have shown that volatilization of UC_2 occurs in WAR particles at easily measurable rates at temperatures greater than 1650°C , which some of the kernels in the high-temperature region reached during irradiation.

The fission product zirconium distributions in both rods closely followed the actinide distributions just discussed (Figs. 23 and 24). Similar behavior has been observed in HEU WAR particles.¹⁰ Cesium was in the densified buffer and inner LTI coatings, as shown in Fig. 24 (the counting rate was too low to produce an acceptable x-ray display for the particle examined in rod 19). Barium and strontium were detected in the particles, but again their counting rates were too low.

Fig. 22. Electron Microprobe Showed Slight Penetration of SiC by Palladium in Particle in Rod 6 (also Shown in Fig. 18). (a) Optical, (b) backscattered electron, and (c) Pd L α x-ray display.

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Fig. 23. SiC-Fission-Product Palladium Reaction Rate from HT-33 Data.

Fig. 24. Electron Microprobe X-Ray Displays of Particle from Rod 19 (Also Shown in Fig. 19). (a) Backscattered electron image, (b) U M α , (c) Pu M α , (d) Si K α , (e) Cl K α , and (f) Zr L α .

Fig. 25. Electron Microprobe X-Ray Displays of Particle from Rod 6 (Also Shown in Fig. 18). (a) Backscattered electron image, (b) U M α , (c) Pu M β , (d) Cs L α , and (e) Zr L α .

Though palladium was involved with SiC corrosion, the other noble metal fission products behaved differently. The molybdenum, ruthenium, rhodium, and technetium were with the actinide distributions, and noble metal inclusions were in the residual kernel material (Figs. 26 and 27). Similar results have been observed with HEU WAR particles with comparable oxygen contents.¹⁰

The rare earth fission products — neodymium, cerium, praseodymium, and lanthanum — were located with the actinide distributions (Figs. 28 and 29). These results are identical to HEU WAR particles with comparable oxygen contents.¹⁰

An initial concern with using partially converted LEU WAR particles was the migration of plutonium in the particle. The plutonium migrated extensively in particles from rod 19, aggravated by the high irradiation temperatures. Plutonium migrated to the SiC coating in one particle where the inner LTI had separated from the SiC. Chlorine was identified with the plutonium distribution (Fig. 30). The particles from rod 6 indicate that plutonium migration is minor in LEU WAR particles when the irradiation temperatures are less than 1200°C.

CONCLUSIONS

Irradiation capsule HT-33 achieved its objectives while utilizing space that had not been used in previous HT capsules.

Fuel rods with slug-injected matrices (using a pitch binder) and warm-molded matrices (using a thermo-setting resin binder) both showed good irradiation behavior. The matrices did not cause any problems with fuel rod integrity.

The slug-injected rods containing the Speer shim particles had more debonding problems compared to similar rods containing GLCC H-451 shim particles; however, the difference was not great.

Pyrocarbon permeability measurements demonstrated that the disorder created by the neutron flux did not cause an increase in the inert gas permeability.

The Triso-coated particles irradiated without an outer LTI coating (referred to as SiC-coated in our report) showed extensive degradation of

Fig. 26. Electron Microprobe X-Ray Displays of Particle from Rod 16 (Also Shown in Fig. 19). (a) Backscattered electron image, (b) Pd L α , (c) Ru L α , (d) Mo L α , (e) Rh L α , and (f) Tc L α .

Fig. 27. Electron Microprobe X-Ray Displays of Particle from Rod 6 (Also Shown in Fig. 18). (a) Backscattered electron image, (b) Pd $L\alpha$, (c) Mo $L\alpha$, (d) Ru $L\alpha$, and (e) Tc $L\alpha$.

Fig. 28. Electron Microprobe X-Ray Displays of Particle from Rod 19 (Also Shown in Fig. 19). (a) Backscattered electron image, (b) Nd L α , (c) Ce L α , (d) Pr L α , and (e) La L α .

Fig. 29. Electron Microprobe X-Ray Displays of Particle from Rod 6 (Also Shown in Fig. 18).
(a) Backscattered electron image, (b) Nd L α , (c) Ce L α , and (d) Pr L α .

Fig. 30. Electron Microprobe X-Ray Displays of Particle from Rod 19 Showing Uranium and Plutonium Migration to SiC. (a) Backscattered electron image, (b) U M α , (c) Pd L α , and (d) Pu M β . Maximum fuel temperature - 1430°C.

the SiC at temperatures above approximately 1375°C. Triso-coated particles (with outer LTI coatings) showed no SiC degradation at similar temperatures. The rods containing the SiC-coated particles also showed less integrity than the rods with Triso-coated particles. We had hoped that the elimination of the outer LTI coating would make it possible to increase the fuel loading per rod (by decreasing the particle size) and stop matrix-particle interaction; but evidently the outer LTI coating is necessary for optimum irradiation performance.

The behavior of the fission products in these LEU WAR particles appeared to be similar to studies on HEU WAR particles with comparable oxygen contents.^{5,10} Calculations¹⁵ have been made that indicate that the only significant fission products (i.e., those with fission yields >0.1%) with inventories higher in a 20%-enriched particle at 20% FIMA (300- μ m kernel) than a 93%-enriched particle at 70% FIMA (200- μ m kernel) were palladium and silver (3.67 and 12.28 times higher, respectively). No data on silver behavior was available from the electron microprobe in our test. Palladium caused corrosion of the SiC coating, especially at temperatures above 1400°C.

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APPENDIX

RESULTS OF CAPSULE HT-33 GAS SAMPLING

APPENDIX

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1. The void volume of the capsule was 35.528 cm^3 . The amount of gas release from the capsule was 36.294 cm^3 at standard temperature and pressure.

2. The composition of gas as determined by mass spectrometry is as follows:

<u>Compound</u>	<u>Percentage (%)</u>
H ₂	0.51
He	8.63
CH ₄	0.05
H ₂ O	0.23
N ₂ + CO	0.33
O ₂	0.06
Ar	88.08
CO ₂	0.04
Kr	0.54*
Xe	1.53†

*Kr composed of 5% ^{83}Kr , 39% ^{84}Kr , 8% ^{85}Kr , and 48% ^{86}Kr .

†Xe composed of 0.2% ^{129}Xe , 0.8% ^{130}Xe , 5.9% ^{131}Xe , 22.8% ^{132}Xe , 24.5% ^{134}Xe , and 45.8% ^{136}Xe .