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DEVELOPMENT OF DURABLE LONG-TERM RADIOACTIVE
WASTE COMPOSITE MATERIALS

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**DEVELOPMENT OF DURABLE LONG-TERM RADIOACTIVE WASTE
COMPOSITE MATERIALS**

Progress Report No. 6

March-April 1974

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Previous Reports

Progress Report No. 5 October-December 1973 BNL 18652

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**The attached report encompasses the collection and evaluation
of data for this report period.**

Program Objectives

1. To immobilize or fix high, medium and low level radioactive wastes by incorporating them into monolithic solid blocks using techniques applicable to the formation of cement concrete, polymer impregnated concrete, and polymer-concrete composites.

2. To provide information on the formulations of composite materials using liquid and solid wastes and to evaluate the physical and durability properties of these composite formulations under anticipated radioactive storage conditions.

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3. To translate materials development and techniques resulting from this study to existing waste problems towards long term storage applications.

Nomenclature

1. Cement concrete (CC), radioactive waste materials, liquid or solid, are combined with cement and water to form a hard durable mortar or concrete composite.

2. Polymer-impregnated concrete (PIC), precast cement concrete composites containing liquid or solid radioactive waste materials impregnated with a monomer which is subsequently polymerized.

3. Polymer concrete (PC), a composite material formed by polymerizing a monomer and a solid radioactive waste.

SUMMARY

Leaching data for aqueous NaNO_3 cement concrete composites are reviewed. The leach rates for high alumina cement composites containing 29.8 wt % NaNO_3 are independent of specimen size over a volume factor of 55. Cement-concrete composites containing 5-30 wt % NaNO_3 were irradiated for total doses of 10^9 rads. The compressive strengths and leachability for irradiated specimens were the same as for the controls. Similar radiation experiments were performed using portland type II cement. The results showed no effect of radiation (10^9 rads) on strength and leachability properties. Addition of fly-ash and pumice to portland cement - NaNO_3 formulations did not have any appreciable effect in lowering the leach rates. These materials react with Ca(OH)_2 , a soluble by-product of portland cement hydration to form insoluble cementaceous materials.

Radiation stability studies were conducted for Al_2O_3 calcine composites containing $\approx 40\%$ wt % Al_2O_3 . The composites in the form of cement-concrete, polymer-impregnated concrete and polymer concrete were irradiated to 10^{10} rads. No apparent difference in strength and leachability properties were observed as compared to controls. Studies were conducted for gas evolution from

impregnated Al_2O_3 calcine composites subjected to ^{60}Co -gamma radiation. The gases evolved at 10^9 and 10^{10} rads were collected and analyzed. The yields, in terms of $G(\text{H}_2)$ and $G(\text{carbon})$ ranged from 0.005-0.43 and from 0.0017-0.0035 respectively. Weight losses based on the weight of the total composite were $\approx 0.1\%$.

Calculations to develop process flow sheets for the incorporation of intermediate-level liquid wastes into concrete have been started. Emphasis will be initially on BNL waste.

A computer program has been written to calculate the thermal stresses generated inside of solid radioactive concrete cylindrical forms of the type under study at BNL and for materials such as glasses, metals and bitumens.

I. Aqueous NaNO_3 -Cement Concrete Composites

1. Effect of Specimen Size on Leach Rate

Regardless of the mechanism by which a specific atomic specie is removed from the waste form and enters the surrounding environment, the leachability of the waste form should be independent of the specimen size. The leachability is defined by the equation

$$L_B = \frac{F_i \cdot d \cdot V}{t \cdot S}$$

where L_B is the bulk leachability in grams per cm^2 per day, F_i is the fraction of the i^{th} specie released in t days, d is the density of the waste form, V is the volume, and S the surface area of the specimen. By bulk leachability is meant the equivalent loss in weight of the waste form caused by the leachant. It is a term that originated with leach studies on glasses where the whole sample is slowly dissolved. The leachability, L_i , can also be conveniently used. This is the leach rate of the i^{th} specie and is given by the expression:

$$L_i = \frac{F_i \cdot d \cdot V \cdot C_i}{t \cdot S}$$

where C_i is the concentration of the i^{th} specie.

Figure I shows leach curves for three NaNO_3 -cement concrete composites of identical composition but ranging in volume over a factor of 55. The composites contain 29.8 wt % NaNO_3 . The specimen sizes, volumes, surface areas, fractional release rates and the leachabilities (L_1 and L_B) are tabulated in Table I.

The fractional release rates scale in proportion to the surface to volume ratio. The completed leach rates, given in Table I are in good agreement and indicate that they are independent of size over the range studied.

2. Radiation Stability

A series of aqueous NaNO_3 -cement concrete composites were fabricated to conduct radiation stability studies. The test specimens were prepared by adding high alumina cement (Lumnite) to varying concentrations of aqueous NaNO_3 mixtures. High alumina cement is being considered as a primary cement candidate for this work because of its fast cure time (~ 24 hrs) and its chemical stability. The amount of cement which can be added to a given aqueous NaNO_3 mixture depends on the amount of water in the mixture. The formulations given in Table II have been optimized to satisfy the water requirements for the cement hydration reactions and to give good workability.

FIGURE I

LEACH DATA FOR AQUEOUS NaNO_3 CEMENT CONCRETE
COMPOSITES (29.8 wt.% NaNO_3)

Table I

EFFECT OF SPECIMEN SIZE ON THE LEACH RATE OF NaNO_3
FROM H.A.C. (a) COMPOSITES

Specimen (b) size dia. x l (cm)	Specimen weight (g)	Volume (cc)	Surface area (cm^2)	Initial NaNO_3 (g)	Leach time (days)	Fraction release	Leach rate $\frac{\text{gm/cm}^2/\text{day}}$	
							L_i	L_B
2.62 x 5.80	68.4	31.3	58.5	20.38	7	0.48	2.4×10^{-2}	8.0×10^{-2}
5.28 x 10.40	498.8	227.7	216.3	148.6	7	0.25	2.4×10^{-2}	8.2×10^{-2}
10.40 x 20.25	3,783.8	1,720.2	831.5	1,127.7	7	0.12	2.3×10^{-2}	7.8×10^{-2}

(a) High alumina cement

(b) Cement-concrete composites containing 29.8 wt % NaNO_3

Workability is defined as the ease with which a given set of materials can be mixed into concrete and subsequently handled, transported and placed with minimum loss of homogeneity.

The water-cement ratios (w/c) for the formulations in Table II are independent of NaNO_3 concentration and are calculated at 0.22. This indicates that NaNO_3 is an inert additive to water-cement paste.

Test specimens were exposed to ^{60}Co gamma-rays for a total integrated dose of 10^9 rads at a radiation intensity of 4.4×10^6 rad/hr. Irradiations were conducted in air at 68°C .

Subsequent to radiation, specimens were tested for compressive strength and leachability. Table II gives the results of these tests as compared to the control specimens. The compressive strength for irradiated specimens, over the entire range of NaNO_3 concentration shows little or no change as compared to the controls. The data also indicates that addition of NaNO_3 , up to 30 wt % to a water-cement paste does not affect its strength. This is surprising since the amount of cement or binder in the composite containing 30 wt % NaNO_3 is considerably lower than that in the composites containing lesser amounts of NaNO_3 .

Table II
EFFECT OF ^{60}Co GAMMA RADIATION ON THE STRENGTH AND LEACHABILITY OF
AQUEOUS NaNO_3 -H.A.C. COMPOSITES

Composite NaNO_3	formulations (wt %)		Compressive Strength (psi)		NaNO_3 Leachability (L_i) ^(c) g/cm ² /day	
	Cement (a)	H_2O	Control	Irradiated ^(b)	Control	Irradiated
0.0	81.8	18.2	13,200	12,500	—	—
5.0	77.5	17.5	13,000	12,400	1.3×10^{-3}	1.4×10^{-3}
7.2	75.8	17.0	13,000	12,200	2.6×10^{-3}	2.6×10^{-3}
10.0	73.5	16.5	13,100	12,500	5.0×10^{-3}	6.1×10^{-3}
15.0	69.3	15.7	13,000	12,800	1.0×10^{-2}	1.6×10^{-2}
20.0	65.5	14.5	13,300	13,300	1.5×10^{-2}	1.4×10^{-2}
25.0	61.3	13.7	13,600	13,000	1.8×10^{-2}	1.9×10^{-2}
30.0	57.3	12.7	12,700	12,000	2.3×10^{-2}	1.9×10^{-2}

(a) High alumina cement (H.A.C.)

(b) Total integrated radiation dose, 10^9 rads at a radiation intensity
of 4.4×10^6 rads/hr

(c) $L_i = \frac{\text{Fraction release} \times \text{density} \times \text{volume} \times \text{NaNO}_3 \text{ conc.}}{\text{time (days)} \times \text{surface area}}$

There does not appear to be any significant effect of radiation on the release rate of NaNO_3 . This is shown in Figure II where the leach rates for irradiated specimens were compared to the control specimens.

The data also shows that the leach rate for NaNO_3 increases rapidly with increasing NaNO_3 concentration up to ≈ 15 wt % NaNO_3 . Above 15 wt %, the increase in leach rate is much less. From 5 to 15 wt % NaNO_3 the leach rate increases by a factor of 10 whereas from 15-30 wt % the leach rate only increases by a factor of 2. This effect has been previously observed and is described in Report No. 5.

3. Portland Cement

Previous reported NaNO_3 -cement composites have been for high alumina cement. The use of portland cement for immobilization of aqueous NaNO_3 waste is also being considered. Portland type II cement has been selected for conducting initial studies. Other portland types will eventually be investigated.

Test specimens consisting of aqueous NaNO_3 mixtures and portland cement were made to determine leachability, strength and radiation stability and to compare the results with those containing high alumina cement (H.A.C.). The formulations

Figure II

EFFECT OF ^{60}Co - GAMMA RADIATION ON LEACH RATE
OF HIGH ALUMINA CEMENT - NaNO_3 COMPOSITES

for portland cement, given in Table III are identical to those for H.A.C. The same w/c ratio, 0.22, was used to obtain good workability. A noted difference between portland type II cement and H.A.C. is that portland cement required a 28 day cure as compared to 24 hrs for H.A.C.

Table III also gives compressive strengths of cured composites containing varying concentrations of NaNO_3 before and after irradiation. The radiation conditions were the same as those reported in Table II. The results are essentially the same as those observed for H.A.C. in that no apparent difference can be seen between the irradiated and the control specimens for a given NaNO_3 concentration. An effect which does appear noticeable is that the strength of the composites decrease slowly with increasing NaNO_3 concentration. This is not significant since the composites have high integrity even at the higher NaNO_3 concentrations.

Figure III gives the leach rates for NaNO_3 -portland type II cement composites. The leach data is plotted as fractional leach rate vs. NaNO_3 concentration. For composites up to 15 wt % NaNO_3 , the compositional dependence is very strong. Above 15 wt % there is essentially no dependence of fractional leach rate on composition.

Table III
EFFECT OF ^{60}Co GAMMA RADIATION ON THE STRENGTH OF
AQUEOUS NaNO_3 -PORTLAND CEMENT COMPOSITES

Composite <u>NaNO_3</u>	Formulations ^(a) <u>cement (b)</u>	(wt %) <u>H_2O</u>	Compressive strength (psi) 28 day cure	
			<u>control</u>	<u>irradiated</u> ^(c)
0.0	81.8	18.2	12,300	12,400
5.0	77.5	17.5	12,600	12,900
7.2	75.8	17.0	12,000	13,000
10.0	73.5	16.5	11,000	10,200
15.0	69.3	15.7	8,700	8,600
20.00	65.5	14.5	9,000	8,600
25.0	61.3	13.7	9,000	8,500
30.0	57.3	12.7	8,000	7,400

(a) cured for 28 days

(b) Portland type II cement

(c) Total integrated dose, 10^9 rads at a radiation intensity
of 4.4×10^6 rads/hr

Figure III

FRACTIONAL LEACH RATE VS. COMPOSITION
FOR PORTLAND CEMENT COMPOSITES

This effect can be related to the NaNO_3 -water mix composition which is added to cement. For composites containing less than 15 wt % NaNO_3 there is sufficient water to dissolve all the salt. Above 15 wt %, the mix water is saturated and there is always excess NaNO_3 . The same behavior was observed with high alumina cement and is described in the previous report.

A comparison was made between the leach rates for high alumina cement composites and for portland type II cement composites. It has been observed that leach rates are slightly lower for portland type II cement. This is illustrated in Figure IV. For low NaNO_3 concentrations leach rates from the portland cement composites are about 20% those of the high alumina cement composites. This is not a significant difference in leachability but it has indicated that the difference may result from the pore size and pore size distribution of the two composite materials.

High alumina cement is coarser grained than the portland cement. Measurements of the pore size of high alumina and portland cement composites, from which the salt has been completely extracted indicate an average pore diameter of 0.5μ for the high alumina cement and 0.095μ for the portland cement.

Figure IV

LEACH RATE VS. COMPOSITION FOR
HIGH ALUMINA AND PORTLAND CEMENT COMPOSITES
(AVERAGED OVER FIRST TEN DAYS)

The measurements were made by mercury porosimetry. The effects of cement particle size on leachability and on other composite properties will be pursued further.

Cement Additives

Aqueous NaNO_3 -portland cement specimens containing fly-ash and pumice were prepared and leach tested. Fly-ash and pumice react with $\text{Ca}(\text{OH})_2$, a soluble by-product of portland cement hydration to form insoluble cementaceous materials. The results of these tests are given in Table IV.

The additives in Table IV do not appear to have any appreciable effect in lowering the leach rate, especially at the lower salt concentrations. There may be some effect at the higher concentrations of salt. Further studies on the use of cement additives to decrease NaNO_3 leachability are being initiated. These will include clays, binders and other cementaceous materials.

Table IV

EFFECT OF FLY-ASH AND PUMICE ON NaNO_3 LEACH RATES

<u>Specimen Composition (wt %)</u>				<u>Leach rate (NaNO_3)</u> <u>$\text{g/cm}^2/\text{day}$</u>
<u>Portland type II</u> <u>cement</u>	<u>water</u>	<u>NaNO_3</u>	<u>additive</u>	
61.9	15.3	7.4	15.4 (fly-ash)	7.9×10^{-4}
55.3	15.7	15.1	13.9 (fly-ash)	5.8×10^{-3}
49.0	13.7	25.0	12.3 (fly-ash)	6.7×10^{-3}
45.9	12.8	29.8	11.5 pumice	8.2×10^{-3}
40.4	12.8	29.8	17.0 pumice	8.2×10^{-3}
77.5	17.5	5.0	—————	2.5×10^{-4}
61.3	13.7	25.0	—————	9.8×10^{-3}

II. Al₂O₃ Caline Waste Composites

Simulated Al₂O₃ calcine (ICPP) was incorporated into a water-cement (H.A.C.) mixture to form solid cylindrical cement concrete forms for radiation stability studies. Specimens containing 38.9 wt % Al₂O₃ calcine were exposed to ⁶⁰Co gamma rays for a total integrated dose of 10¹⁰ rads at a radiation intensity of 4.2 x 10⁶ rads/hr. The composite formulation and the results of these tests are given in Table V. Following irradiation the compressive strength decreased by only 16% as compared to the control specimen. No change was observed for NaNO₃ leachability as indicated in Table V. The results of the above tests clearly show Al₂O₃ calcine-cement composites to be highly resistant to radiation damage.

Cement-concrete composites of the same composition as those described above were impregnated with styrene monomer (14.5 wt % of the composite) and subjected to the same radiation treatment as the cement-concrete specimens. Impregnation increased the strength of the cement-concrete composite from ≈6,000 to 12,000 psi. It also reduced the leachability by a factor of ≈2. The irradiated specimen maintained the same strength value as the control but showed a twofold decrease in leachability as compared to the control specimen. This data is also given in Table V.

Table V

RADIATION STABILITY OF Al_2O_3 CALCINE COMPOSITES

<u>Composite</u>	<u>Formulation (wt %)</u>				<u>Compressive strength (psi)</u>		<u>NaNO_3 Leachability (L_1) g/cm²/d</u>	
	<u>Al_2O_3 calcine</u>	<u>cement (a)</u>	<u>water</u>	<u>Polymer loading</u>	<u>control</u>	<u>irradiated</u>	<u>control</u>	<u>irradiated</u>
Cement concrete	38.9	38.9	22.2	—	6,200	5,200	7.0×10^{-4}	6.8×10^{-4}
Polymer-impregnated concrete	38.9	38.9	22.2	14.5 (b)	12,100	12,000	4.5×10^{-4}	2.6×10^{-4}
Polymer concrete	46.9	—	—	53.1	6,400	6,200	6.2×10^{-5}	1.6×10^{-5}

(a) high alumina cement

(b) wt. increase resulting from impregnation of a preformed cement-concrete specimen

(c) irradiated for a total dose of 10^{10} rads at a radiation intensity of 4.2×10^6 rads/hr at 68°C

Experiments were conducted to determine the gases evolved during irradiation of polymer-impregnated calcine cement composites.

The prepared specimens were placed into a container and evacuated prior to irradiation. The evolved gases were removed for analysis at a radiation dose of 10^9 rads. The same specimens were then re-evacuated and irradiated until they had received a total integrated dose of 10^{10} rads. The gases evolved at doses of 10^9 and 10^{10} rads were analyzed and are given in Table VI. It can be seen from Table VI that various reactions are involved during irradiation of a multicomponent system such as polymer-impregnated concrete to produce volatile gases. These gases result predominately from the radiolysis of polystyrene, an organic material, and from radiolysis of water and atmospheric gases entrapped in the composite during preparation.

An attempt will be made, however, to calculate the gas yields in terms of G values. The symbol G is used for expressing radiation-chemical yields and can be defined as the number of molecules produced per 100 eV of absorbed energy. In calculating $G(H_2)$ it was assumed that all of the hydrogen evolved from the radiolysis of polystyrene. It was also

Table VI

GAS EVOLUTION FROM IMPREGNATED Al_2O_3 CALCINE COMPOSITES
SUBJECTED TO ^{60}Co -GAMMA RADIATION (a)

Evolved Gas (b)	Polymer Impregnated Concrete (c)				Polymer Concrete (d)			
	10^9 rads		10^{10} rads		10^9 rads		10^{10} rads	
	mole %	g. $\times 10^{-3}$	mole %	g. $\times 10^{-3}$	mole %	g. $\times 10^{-3}$	mole %	g. $\times 10^{-3}$
H_2	31.20	5.06	58.5	8.68	43.00	5.62	40.9	5.66
N_2	63.20	143.6	34.3	71.40	49.32	90.19	41.2	79.80
O_2	0.02	0.05	0.04	0.096	0.005	0.0096	0.03	0.067
CO_2	0.20	0.88	—	—	2.08	5.97	10.6	32.12
CO	2.36	5.32	3.0	6.16	1.42	2.60	2.1	4.20
H_2O	0.46	0.72	1.1	1.48	0.79	0.93	0.4	1.23
CH_4	1.40	1.76	2.3	2.72	2.18	2.28	3.1	3.36
Argon	0.11	0.40	0.2	0.60	0.09	0.24	0.5	1.40
Hydrocarbons	1.05	6.24	0.24	0.85	1.11	5.86	0.07	0.22
TOTAL	100.00	164.07	99.88	92.65	100.00	113.69	100.00	129.49

(a) Radiation intensity, 4.6×10^6 rads/hr at 68°C

(b) Gases were removed and analyzed at a total dose of 10^9 rads and again at 10^{10} rads

(c) Formulation: 38.9 wt % Al_2O_3 calcine waste; 22.2 wt % H_2O ; 38.9 wt % high alumina cement; 14.5 wt % (composite) styrene

(d) Formulation: 46.9 wt % Al_2O_3 calcine; 53.1 % styrene

assumed that the formation of CO , CO_2 and CH_4 derive from C-C bonds broken during irradiation from the styrene molecules. In an over-simplified manner, the yields for the above gases were calculated in terms of $G(\text{carbon})$. The calculated G values for hydrogen and carbon are given in Table VII. G values are also given for pure polystyrene. These values were calculated from gas evolutions studies reported in Progress Report No. 2, January-March 1973. There appears to be some discrepancy between $G(\text{H}_2) = 0.043$ for pure polystyrene and that for polystyrene-impregnated concrete, $G(\text{H}_2) = 0.016$. Although the values are comparable in magnitude, the noticeable lower hydrogen yield for polymer-impregnated concrete may be due to hydrogen absorption on surfaces of the porous calcine material in the composite. Hydrogen can also recombine to form hydrocarbons and H_2O from entrapped oxygen in the composite. On the other hand, the $G(\text{C})$ values for pure polystyrene and for polymer-impregnated concrete were in good agreement. These values, however, are extremely low and are reflected in the very small calculated weight losses given in Table VII. The percent weight loss for impregnated concrete (based on weight of polystyrene in the composite) was 0.09 at a radiation dose of 10^{10} rads. This indicates that polystyrene is a very

Table VII

G VALUES FOR GASES EVOLVED FROM IRRADIATED CALCINE COMPOSITES CONTAINING POLYSTYRENE

<u>Irradiated Material (a)</u>	<u>Weight of composite (g) (b)</u>	<u>Weight of polystyrene in composite (g)</u>	<u>G(H₂)</u>	<u>G(C) (c)</u>	<u>% weight loss composite polystyrene</u>	
Pure polystyrene	—	0.56	0.043	0.0035	—	0.22
Polymer impregnated concrete	280.7	40.7	0.016	0.0031	0.09	0.63
Polymer cement	206.0	107.1	0.005	0.0017	0.12	0.23

- (a) Total integrated ⁶⁰Co radiation dose, 10¹⁰ rads at 4.6 x 10⁶ rads/hr
 (b) Al₂O₃ calcine waste composites
 (c) Based on CO, CO₂, and CH₄ evolved during radiation

stable polymer with respect to radiation and very large doses are required to produce any noticeable change.

The nitrogen evolved during irradiation, Table VI, is attributed to the radiolysis of the chemical catalyst, α -(t-butylazo) isobutyronitrile, used to cure the styrene monomer.

Polymer-concrete composites were made by combining Al_2O_3 calcine and styrene monomer in a mixer. The mixture was poured into molds and cured in an oven for 4 hrs at 80°C. A chemical catalyst was added to styrene monomer prior to mixing to initiate polymerization at elevated temperature. Polymer-concrete specimens were irradiated similar to the polymer-impregnated concrete specimens described above. Results of these tests are shown in Tables V, VI, and VII.

The strength of the irradiated polymer concrete specimen was the same as the control specimen. As a result of radiation, leachability decreased by a factor of 4. This is not understood but previous work has indicated that the leach rates for irradiated composites remain the same or decrease as compared to control specimens.

The different gases evolved during irradiation of polymer-concrete composites are the same as for polymer-impregnated

concrete except that the quantities vary. The gas yields, $G(H_2) = 0.005$ and $G(\text{carbon}) = 0.0017$ was calculated for polymer-concrete composites. These values are lower than those obtained for the other materials described above. Again, the only explanation that can be given at this time is that a large portion of the gases formed during irradiation are absorbed on the calcine surfaces or react to form non-volatile materials at the conditions of the experiment.

III. Process Development

Calculations to develop process flow sheets for the incorporation of intermediate-level liquid wastes into concrete have been started. Initial work has been with wastes of the type to be produced by Allied-Gulf Nuclear Services (AGNS) at their Barnwell, South Carolina facility.

As a result of information received from AGNS, a waste volume of 70 gal/MTU of a 3M HNO_3 solution containing $\approx 11\%$ salts was assumed as a basis for calculation. After neutralization of the waste, two processes were considered. The first involved incorporation of the $\approx 75\%$ water - 25% salt solution directly into a concrete. Using a water/cement ratio of 0.22, a concrete containing 5.8% salts would be

produced. Approximately 1 yd³ of concrete would be produced per MTU processed.

If the neutralized waste could be concentrated to ~70% salt, subsequent incorporation into concrete would result in composites containing 29.8% salt. A concrete volume of 0.2 yd³/MTU would be expected.

During the next report period similar calculations will be made for BNL waste.

IV. Thermal Stress Studies

A computer program has been written and used to calculate the thermal stresses generated inside of solid radioactive concrete cylindrical forms of the type under study at BNL. The information obtained from these calculations is presently being assembled into curves which will serve as "handbook" or "look-up" data.

These curves will permit the quick determination of the temperature generated and the resulting thermal stresses built up in concrete and similar materials, over a suitable range of material properties. Listed below are the ranges of material properties used:

cylindrical radius	0.5 to 3.0 ft
cylindrical height	infinite
thermal conductivity	0.1 to 1.0 BTU/hr ft °F
power density	0.004 to 1.0 watts/liter
modulus of elasticity	1.0 to 13.0 10^6 psi
coefficient of thermal expansion	1.0 to 15.0 $\times 10^{-6}$ in/in/°C
Poissons ratio	1.10 to 0.23