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SUBJECT: Technical Highlights of Space and Terrestrial Systems Programs at Oak Ridge National Laboratory for March 1980

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FROM: A. C. Schaffhauser

This monthly report is intended to inform the Division of Advanced Nuclear Systems and Projects, DOE, and their contractors of significant technical highlights of technology and systems support programs in progress at Oak Ridge National Laboratory. A detailed technical presentation of information will be published in topical reports and open literature publications.

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CONTENTS

SOLAR-POLAR MISSION MATERIALS SUPPORT

(Activity AE 15 15 20 0, WPAS 02313)

R. L. Heestand

The purpose of this task is to provide direct materials support on work specific to the General Purpose Heat Source (GPHS) design. The scope of this work includes (1) determining the impact properties and optimum weld parameters for the DOP-26 iridium alloy fuel cladding material, (2) thermal conductivity and emissivity data on cladding and thermal insulation materials, (3) fabrication and characterization of test samples of carbon-bonded carbon-fiber (CBCF) insulation developed by ORNL and Y-12 which may be used for GPHS, and (4) determination of creep properties of the 2219 aluminum alloy housing. Data on the impact properties and welding of iridium are integrated with complementary work reported in the Materials Technology section of this report.

<u>CBCF-3 Insulation Fabrication</u> (G. W. Brassell and C. D. Reynolds)

The fabrication of ten sets of CBCF-3 insulation was completed for GPHS module impact tests to be conducted at Los Alamos Scientific Laboratory (LASL). Shipment was completed late in February. Several cylindrical test specimens were fabricated using the newly designed multiple nine tube mandrel. These specimens, which were fabricated as a part of the process feasibility study were within density specifications. Several 8-in.-diam discs were also fabricated and evaluated for density. The values for these discs ranged from 0.212 g/cc to 0.230 g/cc. Their respective compressive strengths were 99 psi and 139 psi.

Following evaluation of the initial nine tube mandrel runs, 27 tubes (3 runs) of CBCF-3 insulation were fabricated and are currently being machined and inspected. End caps for these tubes are being machined from an 8-in.-diam by 1-in.-thick molded disc. Density variations of both the tables fabricated using the multiple mandrel process and the flat discs were within the specified density tolerances.

Installation of the new CBCF-3 insulation facility at ORNL has been initiated. Laboratory space and the necessary equipment has been obtained and is presently being installed. It is anticipated the facility will be in operation by mid-April.

<u>Contact Conductance for GPHS</u> (G. C. Wei)

Contact conductances for material couples in GPHS were measured at low temperatures at Purdue University and at high temperatures at Battelle Columbus Laboratory. The results from Purdue are presented in Table 1. Calculations using 1/3, 1/2, and 2/3 rise time showed conductance values essentially the same as those listed in Table 1. The experimental data from Battelle are shown in Table 2. In this period, Fairchild also conducted contact conductance experiments on FWPF conical seats and GIS after simulated launch vibration. As reported in the previous monthly report, ORNL measured contact conductance of FWPF ($Z\parallel$) vs FWPF ($Z\perp$) at room temperature and the data were in agreement with Purdue's results considering the extrinsic nature of the surfaces involved in the measurements. Therefore, we have a total of four sets of contact conductance data. Figure 1 summarizes all the data on the FWPF contact which is the most important contact in the LMRE version of GPHS design.

Figure 1 shows that the contact conductance of FWPF vs FWPF obtained from Purdue, Battelle, and ORNL are in close agreement. The Fairchild data, however, were one to two orders of magnitude lower than the results obtained by the other three labs. Detailed examinations of Fairchild experiments indicated non-uniform heat fluxes, 40% heat loss, and uncontrolled geometry of contact. Corrections for these factors which could lead to erroneous results were not made in the calculation. Therefore, the Fairchild results should be treated with caution. In the meantime, high-temperature measurements of contact conductance of post-vibration FWPF conical seat and GIS were performed at Battelle. The results are not available as of this reporting period.

CBCF-3 Chemical Analysis (G. C. Wei)

Chemical analysis results of one outgassed (1500°C/36h/1.0E-5 torr) CBCF-3 sample along with MRC's draft specifications of trace impurities in outgassed CBCF-3 are shown in Table 3. It is obvious that all elements meet the specification except for the 300 ppm Si. As-made CBCF-3 samples typical of the CBCF-3 parts sent to LASL and Fairchild were analyzed chemically. The results are presented in Table 4.

Bonding of CBCF-3 with FWPF (G. C. Wei)

The purpose of developing a technique to bond CBCF-3 to FWPF is to facilitate GPHS module assembly work. To date, we have identified phenolic resin as the bonding agent for CBCF-3/FWPF. Metallographic examination revealed a $\sim$254 μm zone rich in the resin-derived carbon bond at the interface of CBCF-3 and FWPF. It should be noted that the amount of phenolic resin applied can be optimized depending on the strength and thermal conductance requirements. Dylon cement which was suitable for bonding grafoil with FWPF was analyzed chemically. The results shown in Table 5 indicated rather high concentrations of impurities in outgassed Dylon cement. Thus it is questionable that Dylon cement can be used in the GPHS system.

CBCF-3 QA Program (G. C. Wei)

Since CBCF-3 was formally selected to be used as thermal insulation in the GPHS module for the 1983 ISPM on March 1, we have initiated a formal quality assurance program for fabrication and testing of CBCF-3 parts. The basic philosophy used in the quality assurance program[1] of manufacturing of CBCF-3 parts for Galileo SIG program will be adopted in the present program.

The data base of single-mandrel fabricated CBCF-3 tubes was reviewed. The bulk density (0.20 ± 0.02 g/cc), compressive strength (>75 psi), and thermal conductivity data are listed in Tables 6 and 7.

To date, there are two problems with multi-mandrel fabricated CBCF-3 parts, (1) more recently fabricated CBCF-3 tubes using multi-mandrel molding facility had the same density as single-mandrel fabricated CBCF-3 tubes but displayed a larger variation in compressive strength with some samples in the order of 20 psi, and (2) CBCF-3 plate made of carbon fiber batch AEPC-2 had a density (0.21 ± 0.02 g/cc) slightly higher than CBCF-3 tubes.

Statistics-based experimental plans for obtaining a data base of multi-mandrel fabricated CBCF-3 parts were initiated. The cost estimate and schedule for these activities were also completed.

Table 1. Experimental Results of Contact Conductance at RT Using Laser Flash Diffusivity Method by Purdue University

Front Layer	Rear Layer	Atmosphere Conditions	Contact Pressure (psi)	Half-time (sec)	Contact Conductance (W cm^{-2}K^{-1}) Lee's Method	James Method [a]	James Method [b]
FWPF⊥[c]	FWPF∥[c]	0.1 atm	5	0.318	1.52	1.46	1.49
FWPF⊥	FWPF∥	0.1 atm	>75	0.182	32.	15.0	13.59
FWPF⊥	FWPF∥	0.1 atm	>150	0.175	690.	25.6	23.28
FWPF⊥	FWPF∥	0.1 atm	5	0.313	1.58	1.513	1.538
FWPF⊥	FWPF∥	0.1 atm	75	0.211	6.30	5.22	4.97
FWPF⊥	FWPF∥	0.1 atm	150	0.186	22.	11.97	10.94
FWPF⊥	FWPF∥	0	5	0.342	1.30	1.246	1.280
FWPF⊥	FWPF∥	0	75	0.218	5.29	4.50	4.31
FWPF⊥	FWPF∥	0	150	0.185	22.	12.6	11.50
PG[d]	FWPF∥	0.1 atm	5	0.263	0.85	0.774	0.776
PG	FWPF∥	0.1 atm	75	0.186	34.	∞	6.053
PG	FWPF∥	0.1 atm	175	0.179	large	∞	15.94
PG	FWPF∥	0	5	0.268	0.80	0.731	0.7336
PG	FWPF∥	0	75	0.189	32.	∞	4.769
PG	FWPF∥	0	175	0.172	large	∞	∞
PG	FWPF⊥	0.1 atm	5	0.393	0.45	0.4385	0.4369
PG	FWPF⊥	0.1 atm	75	0.286	1.53	1.36	1.289
PG	FWPF⊥	0.1 atm	175	0.264	2.91	2.36	2.238
PG	FWPF⊥	0		0.470	0.30	0.286	0.2921
PG	FWPF⊥	0	75	0.284	1.52	----	1.343
PG	FWPF⊥	0	175	0.258	3.0	----	2.785
PG	PG	0.1 atm	5	0.273	0.94	0.926	0.878
PG	PG	0.1 atm	75	0.220	large	----	∞
PG	PG	0.1 atm	150	0.205	large	----	∞
PG	PG	0	150	0.230	large	----	14.9
PG	PG	0	75	0.242	large	----	2.806
PG	PG	0	5	0.374	0.28	0.275	0.2776

Table 1. (Continued)

Front Layer	Rear Layer	Atmosphere Conditions	Contact Pressure (psi)	Half-time (sec)	Contact Conductance ($W\ cm^{-2}K^{-1}$)		
					Lee's Method	James Method[a]	James Method[b]
FWPF⊥	Iridium	0.1 atm	5	0.276	0.60	0.868	0.858
FWPF⊥	Iridium	0.1 atm	75	0.108	12.8	8.6	8.77
FWPF⊥	Iridium	0.1 atm	150	0.102	7.9	13.51	8.5
FWPF⊥	Iridium	0	150	0.103	11.0	----	----
FWPF⊥	Iridium	0	75	0.109	7.7	----	----
FWPF⊥	Iridium	0	5	0.376	0.38	0.622	0.621
Grafoil	FWPF⊥	0.1 atm	5	0.0980	0.59	0.582	0.5996
Grafoil	FWPF⊥	0.1 atm	75	0.0845	1.27	1.247	1.28
Grafoil	FWPF⊥	0.1 atm	150	0.0804	1.92	----	1.91
Grafoil	FWPF⊥	0	150	0.0807	1.85	----	1.84
Grafoil	FWPF⊥	0	75	0.0822	1.56	1.529	1.566
Grafoil	FWPF⊥	0	5	0.0900	0.84	0.859	0.874
Grafoil	Iridium	0.1 atm	5	0.0166	1.19	1.16	1.187
Grafoil	Iridium	0.1 atm	75	0.0066	∞	----	∞
Grafoil	Iridium	0.1 atm	150	0.0065	∞	----	∞
Grafoil	Iridium	0	150	0.0068	∞	----	∞
Grafoil	Iridium	0	75	0.0068	∞	----	∞
Grafoil	Iridium	0	5	0.0190	0.92	0.885	0.934
Grafoil	Iridium	0.1 atm	5	0.0170	1.14	----	----
Grafoil	Iridium	0.1 atm[e]	5[e]	0.0179[e]	1.85	----	----
CBCF3	FWPF⊥	0.1 atm	5	1.18	0.27	0.330	0.313
CBCF3	FWPF⊥	0.1 atm	75	1.12	1.7	0.6901	1.7
CBCF3	FWPF⊥	0	5	2.25	0.016	0.0202	0.0205
CBCF3	FWPF⊥	0	75	1.14	0.60	0.5316	0.76

[a]Uses a single leading term in expansion of the analysis.
[b]Uses additional terms in expansion of the analysis.
[c]Fine weave pierced fabric. "⊥," Z-axis perpendicular to flat face of disc; "∥," Z-axis parallel to flat face of disc.
[d]Pyrolytic graphite, heat flow is perpendicular to AB direction.
[e]300°C, used RT thermal conductivity values for iridium.

Table 2. Experimental Results of Contact Conductance at High Temperatures Using Longitudinal Heat Flow Method by Battelle Columbus Laboratory.

Material System	Temperature (°C)	Atmosphere Condition	Contact Pressure (psi)	Contact Conductance W $cm^{-2}K^{-1}$
FWPF vs FWPF	1150	Vacuum	5	0.6
FWPF vs FWPF	1150	Vacuum	37	1.0
FWPF vs FWPF	1150	Vacuum	75	2.1
FWPF vs PG	1150	Vacuum (5.0 E-7 torr)	37	0.23
FWPF vs PG	1100	10 μ P_{N_2}	37	0.17
FWPF vs PG	1100	100 μ P_{N_2}	37	0.10
FWPF vs PG	1100	500 μ P_{N_2}	37	0.10[a]
FWPF vs PG	1100	50 torr P_{N_2}	37	0.07
FWPF vs PG	1100	100 torr P_{N_2}	37	0.17

[a]System cycled to 760 torr P_{N_2}, pressure relieved then evacuated to 500 torr P_{N_2}.

Table 3. Chemical Analysis Results of A CBCF-3 Sample (Outgassed at 1500°C, 10^{-5} torr for 36 h) Randomly Selected from CBCF-3 Parts Made From Carbon Fiber Batch AEPC-2 Showed Trace Impurities.

Element	Concentration (ppm wt)	Element	Concentration (ppm wt)	Element	Concentration (ppm wt)
U	<10	Ru	<5	Cl	3
Th	<10	Mo	10	S	50
Bi	<3	Nb	3	P	10
Pb	<5	Zr	3	Si	300
Tl	<10	Y	<1	Al	3
Au	<10	Sr	1	Mg	≤10
Pt	<20	Rb	<1	Na	0.3
Ir	<30	Br	<2	F	<2
Os	<20	Se	<1	Lu	<3
Re	<20	As	<1	Yb	<10
Ta	≤20	Ge	<2	Tm	<3
Hf	<30	Ga	3	Er	<10
La	<2	Zn	<2	Ho	<3
Ba	20	Cu	<2	Dy	<10
Cs	<1	Ni	<2	Tb	<3
I	<2	Co	<1	Gd	<10
Te	<20	Fe	3	Eu	<5
Sb	<5	Mn	<1	Sm	<10
Sn	<10	Cr	1	Nd	<10
In	<2	V	1	Pr	<3
Cd	<10	Ti	≤3	Li	<50
Ag	<5	Sc	<1	B	∿50
Pd	<10	Ca	10	Be	<50
Rh	<2	K	1		

<u>MRC Draft Specification of Chemical Purity of Outgassed CBCF-3</u>

Element	Maximum Acceptable Limit
Cl, Na, K, P, S	50 μg/g each
Si, Al, Mg, Ni, Cr, Cu	100 μg/g each
B, Mn, Li, Cd, Mo, Pb, Zn	100 μg/g each
Fe, V, Ca, Ti	200 μg/g each

The sum total of all listed impurities shall not exceed 900 μg/g. The value of any single unlisted element shall not exceed 50 μg/g.

Table 4. Chemical Analysis Results of As-Made CBCF-3 Parts for GPHS

	Concentration (ppm wt)				
Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Ag	<5	<5	<5	<5	<5
Al	30	10	30	100	50
As	10	1	10	<5	<1
Au	<10	<10	<10	<5	<10
Ba	5	<5	50	70	10
Bi	<3	<3	<3	<5	<5
Ca	1000	30	1000	1000	300
Cd	<10	<10	<10	<5	<10
Ce	<3	<3	<3	<5	<10
Cl	300	100	300	50	20
Co	1	<1	10	10	<1
Cr	3	1	10	70	20
Cs	<1	<1	<1	<5	<0.3
Cu	3	<3	3	5	<2
Dy	<20	<20	<20	<5	<10
Er	<30	<30	<30	<5	<10
Eu	<10	<10	<10	<5	<10
F	<5	<5	<5	<5	<2
Fe	400	30	100	500	300
Ga	3	<3	10	<5	<2
Gd	<10	<10	<10	<5	<10
Ge	<3	<3	<3	<5	<3
Hf	<50	<50	<50	<5	<10
Ho	<5	<5	<5	<5	<10
I	<5	<5	<5	<5	<3
In	<3	<3	<3	<5	<10
Ir	<30	<30	<30	<5	<10
K	1	3	5	40	4
La	<3	<3	<3	<5	<3
Lu	<5	<5	<5	<5	<10
Mg	<10	<10	<10	<5	<30
Mn	1	1	<1	7	2
Mo	<10	<10	10	<5	<6
Na	1	1	3	30	3
Nb	<3	<3	<3	<5	<3
Nd	<30	<30	<30	<5	<20
Ni	20	3	3	100	4
Os	<50	<50	<50	<5	<10
P	100	30	30	70	20
Pb	<10	<10	<10	<5	<5
Pd	<10	<10	<10	<5	<10
Pr	<3	<3	<3	<5	<3
Pt	<50	<50	<50	<5	<10
Rb	<1	<1	<1	<0.5	<1
Re	<30	<30	<30	<5	<10
Rh	<5	<5	<5	<5	<3
Ru	<10	<10	<10	<5	<10
S	200	200	70	100	50
Sb	<10	<10	<10	<5	<5
Sc	≤3	≤3	≤3	<5	<1
Se	<1	<1	<1	<5	<4
Si	1000	70	300	200	70
Sm	<30	<30	<30	<5	<10
Sn	<10	<10	<10	<5	<10
Sr	10	1	3	40	8
Ta	<20	<20	20	≤10	≤50
Tb	<5	<5	<5	<5	<10
Te	<30	<30	<30	<5	<10
Th	<30	<30	<30	<50	<10
Ti	10	3	7	<5	<20
Tl	<10	<10	<10	<5	<5
Tm	<5	<5	<5	<5	<10
U	200	20	<20	20	50
V	10	1	1	<5	<5
W	<50	<50	<50	<5	<40
Y	<3	<3	<3	<5	<2
Yb	<30	<30	<30	<5	<10
Zn	3	<3	3	50	5
Zr	3	3	3	2	<5

Table 5. Chemical Analysis Results of Outgassed Dylon Cement.

Element	Concentration (Wt ppm)	Element	Concentration (Wt ppm)
Si	>1000	Cr	20
Ti	500	Cd	<20
S	400	Mg	<20
Ca	300	Sn	<20
Cl	200	Pb	<10
Al	200	Sb	<7
Na	200	Zn	5
Fe	100	Bi	<5
Ba	100	Ni	3
Cu	50	Mn	<3
V	50	Nb	<2
Mo	50	As	<2
P	20		

Table 6. Variations in Density and Compressive Strength of As-Made CBCF-3 Cups Made of Batch AEPC-2 Carbon Fiber Using Single-Mandrel Facility.

Cup Number	Density (g/cm^3)	Compressive Strength at 5% Strain (psi)
	Shipment to LASL	
1	0.20	163
2	0.19	117
7781-65-2425	0.20	118
7781-65-2427	0.20	134
7781-65-7796	0.20	128
7781-65-2426	0.20	84
7781-54-2428	0.20	128
7781-66-2424	0.20	126
7781-54-2421	0.20	135
7781-54-2429	0.20	153
7781-65-7800	0.1927	157
	0.1959	153
	0.1947	130
	0.1956	170
7781-54-7813	0.2060	191
	0.2027	182
	0.2030	178
	0.2103	185
7781-54-7814	0.2117	186
	0.2094	173
7781-65-7815	0.2078	185
	0.2095	197
	0.2020	174
	Shipment to Fairchild	
17	0.1833	111
	0.1879	163
17A	0.2073	166
	0.2019	145
19	0.1961	113
	0.2026	150
7781-55-7764	0.2028	146
	0.1995	123
	0.2005	85
7785-18-4643	0.1963	173
	0.1886	182
	0.1982	85
	0.1953	125
7781-55-7776	0.1969	115
	0.1957	142
	0.1964	128
7785-18-4645	0.1997	111
	0.1980	76
7785-18-4646	0.1964	126
	0.1956	150
	0.1966	112
	0.1969	126
7785-18-4644	0.1956	131
	0.1966	159
	0.1969	89
7781-55-7763	0.2040	112
	0.1841	105
	0.2043	165
	0.1989	157
18	0.2173	87
	0.2159	150
27	0.1903	107
	0.1939	148
	0.1935	100
25	0.2222	92
	0.1992	105
	0.1984	126

Table 7. Thermal Conductivity of a Sample from Outgassed CBCF-3 Parts for GPHS

Temperature (°C)	Thermal Conductivity in Vacuum (W/m °K) of Outgassed CBCF-3 Cup (0.20 g/cm^3)
800	0.110
1000	0.122
1200	0.133
1400	0.144
1600	0.155
1800	0.167
2000	0.182

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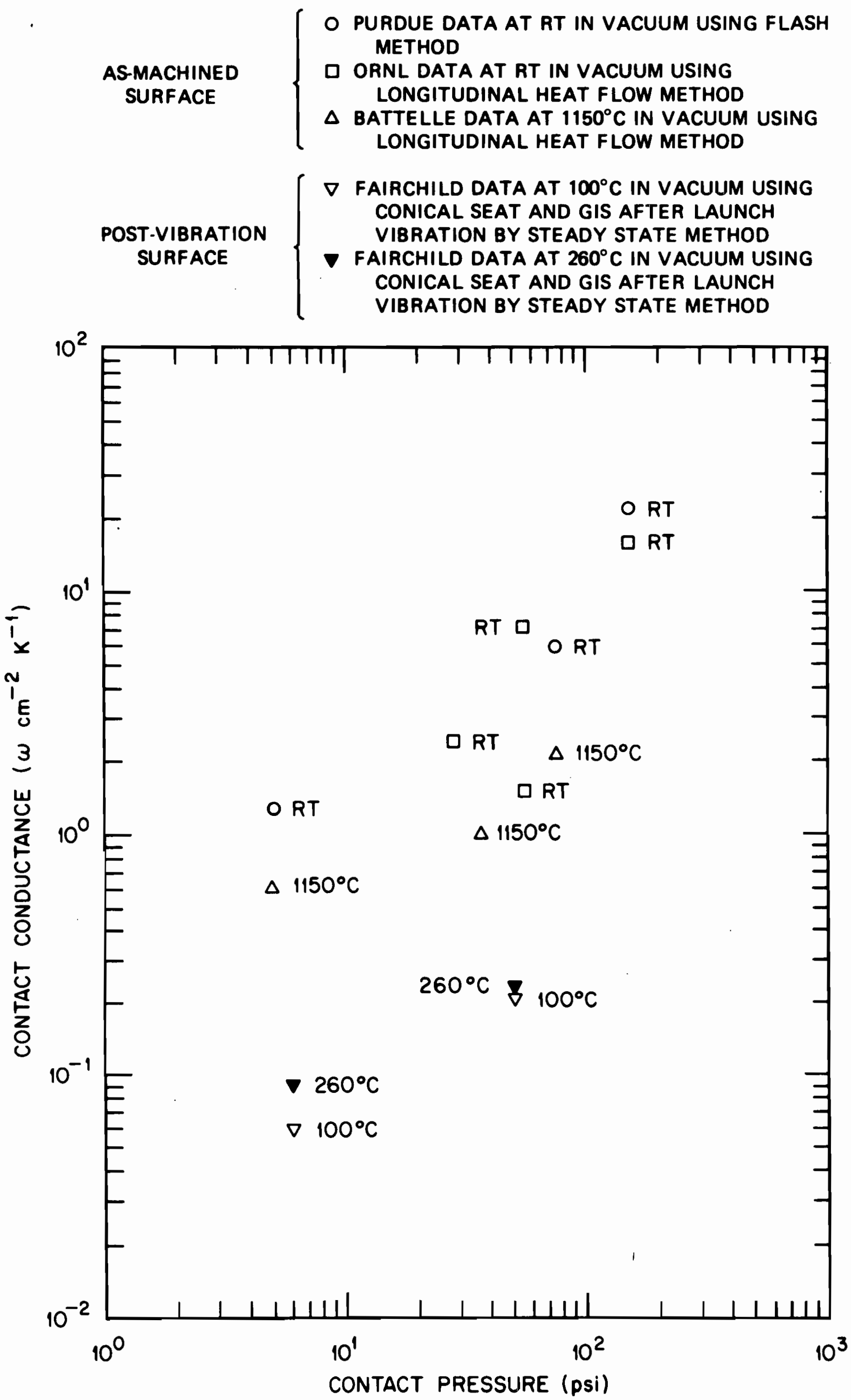

Fig. 1. A summary of four sets of contact conductance data for FWPF vs FWPF.

Creep Properties of 2219 Aluminum Housing Materials (J. P. Hammond)

The purpose of this task is to develop creep design curves on the 2219-T6 forging from which the shell of the RTG generator is to be fabricated. Log stress (σ), log time (t) relationships for 1% plastic strain (ε_p) at 300 and 500°F in the 1000- to 10,000-h time period are required. The following test matrix was developed from estimates and extrapolations made from 1000-h creep data obtained from Alcoa, New Kingsington Laboratory for establishing the foregoing information (Table 8).

Table 8. Creep Test Matrix on 2219 to Aluminum Forging Longitudinal Direction

Targeted t (h)	Loading σ for 1% ε_p (psi)	Duplicate Test, σ for 1% ε_p (psi)
(at 500°F)		
10,000	12,200[a]	repeat[b]
5,000	12,750[a]	
1,000	14,000	repeat[b]
(at 300°F)		
10,000	27,000[a]	repeat[b]
5,000	28,000[a]	
1,000	30,500	repeat[b]

[a]Selected values based on estimates and extrapolations from available 1,000 h rupture data.

[b]Adjust stress to better fit targeted time for 1%ε_p.

A section of the 2219-T6 aluminum shell forging was received from GE, Valley Forge, and 20 longitudinal creep specimens were fabricated. Six lever-arm creep machines were readied for initiating tests, which will be conducted in air.

Important Meetings

1. G. C. Wei and R. K. Williams presented results on contact conductance measurements at the GPHS Contact Conductance Conference at DOE HQ, Germantown, Maryland, March 5, 1980.

2. A meeting on Space Systems Hardware Status and QA Interface was held at ORNL on March 17–18. In attendance were personnel from MRC, DOE Headquarters, and ORNL.

References

1. "Quality Assurance Program for Fabrication of Carbon-Bonded Carbon-Fiber Insulation," G. C. Wei, December 1977.

FLIGHT SYSTEMS HARDWARE

(Activity AE 15 20 00 0, WPAS 01322)

D. E. Harasyn

The objective of this task is to supply Mound Facility with flight quality hardware components for use in the assembly of isotope heat sources. The major activity is on fabrication of iridium alloy forming blanks for isotope fuel capsules along with iridium foil for vents, decontamination covers, and weld shields. We have completed fabrication of iridium for the Galileo mission MHW heat source and have initiated fabrication of 1255 iridium blanks and associated foil for Solar-Polar Mission General Purpose Heat Source for delivery in FY 1980 and FY 1981. A new task on fabrication of carbon-bonded carbon-fiber (CBCF) thermal insulation for Light Weight Radioisotope Heating Units (LWRHU) has also been initiated.

Iridium Blank Fabrication (D. E. Harasyn)

Of the 316 blanks that were to have been shipped to MRC by the end of March, 204 have been shipped. Of the 81 blanks that MRC returned to ORNL for additional cleaning, 80 have been cleaned and returned to MRC. One of the 81 was rejected by ORNL after recleaning because of a penetrant indication that could not be worked out. Details of the cleaning procedure were given under this section in the February Monthly Highlights. Production data for the March shipment are shown in Table 9. Shipments of blanks to MRC are likely to remain behind schedule until July or August.

Ingots L264-269 were finish rolled to final thickness with new equipment this month. New WC rolls were used for the rolling. These rolls were installed last month in our Bliss 4-hi mill in the Metals Processing Lab. Previously, finish rolling had been done at Y-12 on borrowed equipment. A new Inconel muffle furnace was used during the finish rolling of L264-269. This new furnace will prevent any pickup of firebrick inclusions which had occurred in the past when furnaces with firebrick muffles were used. These changes have significantly reduced the amount of pitting and inclusions in the finish rolled sheet L264-269.

Although the amount of inclusions will be reduced, we expect that they will always be present to some degree in the finish rolled Ir sheet. This is simply because it is impossible to keep the rolls, furnace, and Ir absolutely clean. As a result of the above changes to the finish rolling process, we expect to minimize the size and number of inclusions to the point where only inspection at high magnification (>100×) can detect them. This high a level of inspection was not performed on MHW blanks. Inclusions visible only at 100× would have gone unnoticed because the visual inspection was limited to 30×. Neither the MRC nor the ORNL

Table 9. Solar Polar Forming Blank Production Data for March 1980

Ingot No.	Date Shipped	Blanks Machined	Blanks Shipped I.D. No.								Nonconformance[a] Blank I.D. No								Total Shipped lot/FY
			1	2	3	4	5	6	7	8	1	2	3	4	5	6	7	8	
L252[b]	3-27-80	8		1		1	1	1			v		u				p	p	
L253	3-27-80	8	1	1		1			1				p		p	p		u	
L254[b,c]	3-27-80	8	1	1	1	1	1	1	1									u	
L255[c]	3-27-80	8		1		1	1	1	1	1	p		u		v				
L256[c]	3-27-80	8		1	1		1	1		1	p			p			p		
L257[b]	3-27-80	7	1	1	1	1	1		1						v	u			
																			32/204

[a]V = visual, u = ultrasonic, p = liquid penetrant.
[b]Cracks observed in metallography sample.
[c]Hardness exceeded 550 DPH specification limit.

specification have set limits on surface inclusions in the past. The revised ORNL visual inspection procedure will specify no inclusions visible at 30x. Impact data show no effect on ductility for samples having a much larger quantity of inclusion and impurity 2 orders of magnitude lower than in the fuel (refer to data in next section).

Pits in the surface of finish rolled Ir sheet are expected to occur much more frequently than they did in surface ground MHW hardware. The pits can be caused by bits of foreign matter of flakes of Ir which stick to the rolls and are then pressed into the sheet. Small casting defects, the rough grainy surface of the Ir after cover rolling and sticking of the Ir to the rolls may all possibly result in pitting. With the use of the new furnace and WC rolls we will significantly reduce the number of pits in the Ir sheet, but we won't eliminate them. Based on discussions with Mound Facility, we have tentatively proposed a maximum pit size of 50 μ (0.001 in.) deep and 100 μ (0.004 in.) in diameter measured at the blank surface.

A discussion of the effect of inclusions and pits on the impact performance of DOP-26 Ir-0.3% W is given under the Materials Technology Support section.

Iridium Foil Fabrication (D. E. Harasyn)

Approximately 380 cm^2 of 0.13-mm-thick Ir-0.3% W foil was shipped to MRC in March (FS-3). Of the 5800 cm^2 that MRC has requested for FY 1980, 1800 cm^2 has been shipped.

Meetings

D. E. Harasyn, R. L. Heestand, A. C. Schaffhauser, and G. W. Brassell attended Space and Terrestrial Systems Hardware Status and QA Interface Review held at ORNL, March 17 and 18.

CBCF-3 Insulation Fabrication for LWRHU (G. W. Brassell and R. L. Heestand)

The six CBCF-3 sleeves fabricated previously were machined to cylindrical shapes for properties evaluation. Densities were found to fall within the current CBCF-3 requirements. Four of these sleeves were transmitted to LASL for vibration tests along with samples fabricated previously at a density of 0.3 g/cc. Further work has not been conducted pending acceptance of CBCF-3 for LWRHU or receipt of the part designs.

MATERIALS TECHNOLOGY SUPPORT

(Activity AE 15 35 00 0, WPAS 01495)

C. T. Liu

The primary objective of this task is to characterize and improve the metallurgical and mechanical properties of noble base alloys, mainly DOP-26 (Ir-0.3% W doped with 60 ppm Th and 50 ppm Al), to meet the requirements of cladding material in radioisotope heat resources for the Galileo and solar-Polar space missions. The current efforts are concentrated on four areas: (1) to characterize the impact properties of Ir alloys in the temperature range 800–1400°C, (2) to improve the low-temperature impact properties of welds in the DOP-26 alloy, (3) to identify the mechanism and sources that degrade the mechanical and metallurgical properties of doped Ir alloys under heat-source environments, and (4) to develop ductile high-temperature alloys acceptable for future flight missions.

Effect of Phosphorus Contamination on Impact Properties of DOP-26 Alloy (C. T. Liu and C. L. White)

Impact properties of DOP-26 alloy doped with phosphorus were determined at 1350°C and 85 m/s (200 fps). The P-doping procedure at LASL involved an anneal of 18 h at 1500°C in vacuum (to produce ∿14 grains per clad thickness) plus an exposure to phosphorus vapor for 1 h at 1500°C in a graphite crucible containing a small amount of P_2O_5. Two specimens of OLMF-8A were impacted at the as-doped condition. Specimens OLMF-8AB and -8AC were given a vacuum anneal of 1 h and 2 h at 1500°C, respectively, after P-doping. Table 10 summarizes the grain size, Ir/P peak-height ratio [P(120 eV)/Ir(229 eV)], and impact properties of these specimens.

The specimens in the as-doped condition showed brittle grain-boundary fracture with a 3.0–5.7% impact elongation. However, the p-doped specimens recovered all their ductility (>30% elongation and >90% reduction of area) after a vacuum-anneal at 1500°C. Auger analyses demonstrate that the phosphorus segregated at grain boundaries can be removed, at least partially, by vacuum annealing at 1500°C. The impact results combined with the Auger analyses indicate that DOP-26 alloy shows a sharp ductile-to-brittle transition at a P/Ir ratio of ∿0.5. Below the level, the impact ductility of DOP-26 appears to be unaffected when impacted at 1350°C and 85 m/s.

Effect of Oxidizing Environment on Metallurgical and Mechanical Properties of DOP-26 Alloy (C. T. Liu)

Previous studies indicate that long-term exposure of DOP-26 specimens to low-pressure oxygen (1.3×10^{-3} Pa) at 1400 and 1330°C causes external oxidation of thorium and thorium depletion at near surface grain boundaries. This thorium depletion unpins grain boundaries which in turn results in enhanced growth of near surface grains. To future characterize the oxygen-exposure effects, DOP-26 specimens were exposed to 1.3×10^{-3} Pa oxygen at 1280°C, the current designed fuel-clad temperature of GPHS heat sources.

Table 10. Effect of Phosphorus Contamination on Impact Properties of DOP-26 Alloy Tested at 1350°C and 85 m/s (280 fps)

Specimen No.	P-Contamination Condition	Heat Treatment	P(120 eV)[a] / Ir(229 eV)	Grain Size[b]	Impact Ductility: Elongation	RA[c] (%)	Fracture Model[d]
OLMF-8A	P-doped	18 h/1500°C/vac + 1 h/1500°C/P	0.57	14.1	3.0-5.7	5-7	GBS
OLMF-8AB	P-doped + Vacuum annealed	18 h/1500°C/vac + 1 h/1500°C/P + 1 h/1500°C/vac	0.39	----[e]	35.2	94	DR
OLMF-8AC	P-doped + Vacuum annealed	18 h/1500°C/vac + 1 h/1500°C/P + 2 h/1500°C/vac	0.21	11.8	31.6	94	DR
	Uncontaminated	19 h/1500°C/vac	0	14	34-40[f]	94-97[f]	DR

[a] Average Auger intensity ratio at grain boundaries.

[b] Number of grains per clad thickness.

[c] Reduction of area.

[d] DR = ductile rupture, GBS = grain-boundary separation.

[e] Not measured.

[f] Results of 6 impacts.

Table 11 shows the impact properties of the DOP-26 specimens exposed for 1000 and 3000 h at 1280°C. The oxygen exposed specimens are very ductile at 1350°C, with impact elongation not different from the specimens annealed 19 h at 1500°C in vacuum. This comparison suggests that exposure to low-pressure oxygen up to 3000 h at 1280°C does not degrade the impact ductility of DOP-26 alloy. Metallographic examination of the grain structure of these specimens is in progress.

Table 11. Impact Properties of Oxygen-Exposed DOP-26 Specimens Tested at 1350°C and 85 m/s (280 fps)

Exposure Condition	Impact Elongation (%)	Reduction of Area (%)	Fracture Mode
1000 h/1280°C/1.3 x 10^{-3} Pa O_2	40.4	96	Ductile Rupture
3000 h/1280°C/1.3 x 10^{-3} Pa O_2	39.8	96	Ductile Rupture
19 h/1500°C/1.3 x 10^{-3} vacuum	34-40	94-97	Ductile Rupture

Grain Growth in Ir—0.3% W Alloys (D. E. Harasyn)

MHFT-67 was recently impacted at LASL. The Ir cladding was exposed to 100 h at 1330°C plus a reentry heat pulse equivalent to 2 min at 1550°C. The cladding had been outgassed 1 h at 1500°C at MF prior to fueling. The hemishells used in MHFT-67 were J9-2 and J7-2. LASL reported that the grain size was 14.5 grains across the thickness.

We simulated the above thermal history by vacuum annealing archive samples from the sheet J7 and J9. Temperature was measured with a W-3% Re vs W-25% Re thermocouple. The grain size of sample J7 after 1 h at 1500°C plus 100 h at 1330°C plus 2 min at 1550°C was 21.8 grains across the thickness while J9 was 22.3. These grain sizes are in fair agreement with an estimated 20 grains/thickness based on the equations and plots of grain growth in DOP-26 Ir-0.3% W given in July 1979, Technical Highlights ORNL/CF-79/286.

The grain size of the cladding in MHFT-67 is significantly coarser than expected based upon the above simulation and estimate. If the PICS were overheated, it most likely occurred during the reentry heat pulse because in this simulation the PICS temperature is calculated rather than measured, and we understand that no calibration was run to verify the calculations. Overheating to about 1600°C would result in such a grain size. This degree of overheating would be inconsequential with respect to grain growth for MHW tests run one month or longer.

Another possible explanation is that the FSA environment causes accelerated the grain growth in MHFT-67. Notably LASL has shown that exposure of DOP-26 Ir-0.3% W to P_2O_5 enhances grain growth. LASL has verbally reported that the phospnorus to iridium peak height ratio on the Ir grain boundaries of MHFT-67 was 0.08 as determined by AES. Unfortunately, we do not know how much phosphorus is needed to produce the observed enhanced grain growth in MHFT-67, nor do we know if MHFT-67 remained at temperature long enough for phosphorus to produce such an effect.

Characterization of DOP-26 Ir-0.3% W Production Sheet (D. E. Harasyn, C. T. Liu)

The purpose of this subtask is to characterize production lots of Ir-0.3% W sheet. Primarily this will involve reporting the results of uniaxial impact tests performed on each production lot. (A production lot is defined as a group of ingots/sheet fabricated as a group at the same time. Six ingots/sheet normally comprise a lot from which about 48 forming blanks are machined.)

This month a review meeting was held at ORNL concerning the status of Solar Polar Hardware and the QA Interface between the Labs and GE. Among the topics discussed was the nature and influence of pits and inclusions in the surface of Solar Polar forming blanks. A discussion of the probable source (firebrick), and actions taken to remove the inclusions and reduce the pitting is given under the Flight Systems Hardware section.

To test the influence of an inclusion on the impact performance of DOP-26 we impacted a tensile sample from WKR-16. This sheet was DOP-26 Ir-0.3% W used to make hardware for GPHS development. This sample contained two large pits and an inclusion within the gage section. The surface inclusion was approximately 0.15 mm long and 0.07 mm across and lay at the bottom of a pit 0.5 mm long, 0.25 mm across and about 0.05 mm deep. An inclusion of this size is likely to have been removed during the visual inspection and cleaning used on production blanks made after L224. The size of the pit is typical of the largest pits occasionally found in production blanks after L224. The tensile sample was annealed 19 h at 1500°C in vacuum. All visual evidence of the inclusion vanished after the heat treatment. If the inclusion was aluminosilicate, we would expect it to have been reduced and react with the Ir under these conditions. The impact was run at 1350°C and 85 m/sec where most of our data base is on DOP-26. The sample fractured away from the pitted area with greater than 90% reduction of area and more than 30% elongation which is expected of DOP-26 Ir-0.3% W. Next month we intend to impact test four more samples with inclusions. We expect to use the SEM to study the chemistry of the inclusions before and after heat treatment. We will also use impact conditions more appropriate to GPHS. This successful impact is not a surprising result because the DOP-26 Ir-0.3% W is rather insensitive to surface defects. This has allowed us to make tensile samples with a punch and die without the need for grinding or other surface conditioning prior to testing.

There have been pits and microscopic inclusions in varying amounts since the finish rolling process was initiated. This material has been used extensively in the GPHS development tests. Over 60% of the uniaxial impact tests conducted at ORNL to characterize DOP-26 for GPHS have used finish rolled Ir sheet. About half of the GTA butt welds made during investigations by the GPHS Welding Task Force in early 1979, were done with finish-rolled Ir sheet. And at present, an informal check of the capsules in the LASL "IRG" series of impacts shows that about 75% are made from finish rolled sheet. There have been no reports that any of these tests were influenced by the surface condition of the Ir.

There may be some concern that the aluminosilicate inclusions will increase the vent plugging problem. However, a simple estimate of the maximum amount of Al and Si present in the inclusions shows that the fuel containing about 100 ppm of each of Al and Si can contribute about 10^4 that amount. It seems at this time that the most significant problem with the inclusions will be with welding of the vent components. Much of this welding has already been done. Those blanks yet to be formed and welded have been recleaned to remove the inclusions.

Weld Development (S. A. David)

A series of successful DOP-26 butt welds were made using 6 kW laser power at a welding speed of 76 cm/min for impact property evaluations. The welds were made by operating the laser in an annular beam mode with a F-18 telescope for beam focusing. The welds were narrow and with minimum distortions.

During an earlier investigation the laser was operated in a cresent beam (highly focused) mode. Autogenous bead on plate welds were made on coupons of DOP-26 alloy at a welding speed of 76 cm/min and laser power ranging from 4–6 kW. The results were identical to electron beam welding at or near sharp beam focus conditions. The weld bead length contained a series of holes punched in the sheet. Efforts to defocus the beam resulted in limited penetration.

Hence, the best beam condition for laser beam welding of Ir alloys has been found to be an annular beam.

Characterization of Tensile Properties of Pt-30 Rh (wt %) (J. R. Keiser and J. F. Newsome)

The metal clad for the Light Weight Radioisotopic Heating Unit (LWRHU) must have good impact properties at low temperature (<200°C) and adequate strength at operating temperatures. The alloy Pt-30 Rh-8 W (Pt-3008) has more than adequate strength at elevated temperature but may not have sufficient ductility at low temperatures. Since Pt-30 Rh is an alternate to Pt-3008, the tensile properties of Pt-30 Rh have been measured and are given in Table 12. In terms of low temperature ductility, Pt-30 Rh is superior to Pt-3008; however, Pt-30 Rh is considerably weaker at elevated temperatures.

Table 12. Tensile Properties of Pt-30 Rh

Test Temperature (°C)	Ultimate Tensile Strength		0.2% Yield Strength		Total Elongation (%)
	MPa	ksi	MPa	ksi	
-196	775.7	112.5	171.3	24.8	54.9
-80	561.9	81.5	147.6	21.4	52.8
24	473.7	68.7	112.6	16.3	54.4
150	426.2	61.8	134.3	19.5	45.2
300	393.3	57.0	114.5	16.6	41.4
530	338.0	49.0	130.6	18.9	32.6
760	277.7	40.3	105.6	15.3	38.7
925	229.7	33.3	99.7	14.5	48.8
1093	168.0	24.4	83.3	12.1	63.6
1175	139.1	20.2	60.9	8.8	61.4
1316	86.7	12.6	47.7	6.9	39.2

ISOTOPE FUEL DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 02314)

R. S. Crouse

$^{90}SrF_2$ Compatibility (R. S. Crouse)

Disassembly of the 30,000 h capsules from PNL has begun at the Fission Product Development Lab (FPDL). According to H. T. Fullam the following samples were shipped to ORNL:

A-44 and -45	TZM at 600°C
B-43 and -44	Hastelloy C-275 at 600°C
C-37 and -38	Haynes 25 at 600°C
A-59 and -60	TZM at 800°C
B-50 and -51	Hastelloy C-276 at 800°C
C-46 and -47	Haynes 25 at 800°C

The Operations' people at FPDL have so far only found nine of twelve. Fullam checked back through the records at PNL and could find nothing to indicate that less than twelve were shipped. We will expand our search here in hopes of finding the missing three. In any event, examination of all we have will continue.

Samples of the fluoride from the TZM capsules will be sent to Analytical Chemistry for spectrographic analysis. This is by request from Fullam.

TERRESTRIAL RADIOISOTOPE APPLICATION DEVELOPMENT

(Activity AE 15 35 00 0, WPAS 01367)

F. N. Case, K. W. Haff, and F. J. Schultz

Cesium-137 Low Solubility Compounds

The thermostat unit for the calorimeter constant temperature bath was received and installed. The heat outputs of four ^{137}Cs pollucite pellets were measured using the calorimeter. This data is given in Table 13 below.

Table 13. Heat Output of Cesium-137 Pollucite Pellets Measured by Calorimetry

Pellet No.	Weight (g)	Heat Output (watts)	Curie Content
C1	41.99	2.18	450
C2	41.15	2.15	444
C3	41.10	2.15	444
C4	39.00	2.12	437

The curie content of each pellet falls within the expected range of 420 to 450 curies.

The saw, which will be used to section the pollucite pellets, was modified after discussions with the hot cell operators. Upon completion of the modifications the saw and its accompanying off-gas scrubber system will be transferred into the hot cell and sectioning of the ^{137}Cs pollucite pellets will begin.

Examination of WESF Cesium Capsule

A WESF ^{137}Cs capsule, fully loaded with ^{137}Cs chloride, was placed on test at 380°C for 289 days. The capsule (C-72) was originally loaded on September 12, 1975 and contained 69,000 Ci on April 18, 1978. The capsule was sectioned on each end and in the center and examined. The specimens indicate pitting up to 10 mils and a general corrosion of 1 to 3 mils depth. No intergranular attack was observed. It is not known if this minor pitting and corrosion was due to the elevated temperature test or resulted from normal loading and storage.

Figure 2 is a typical section of the capsule as polished. Magnification of the section is 500X.

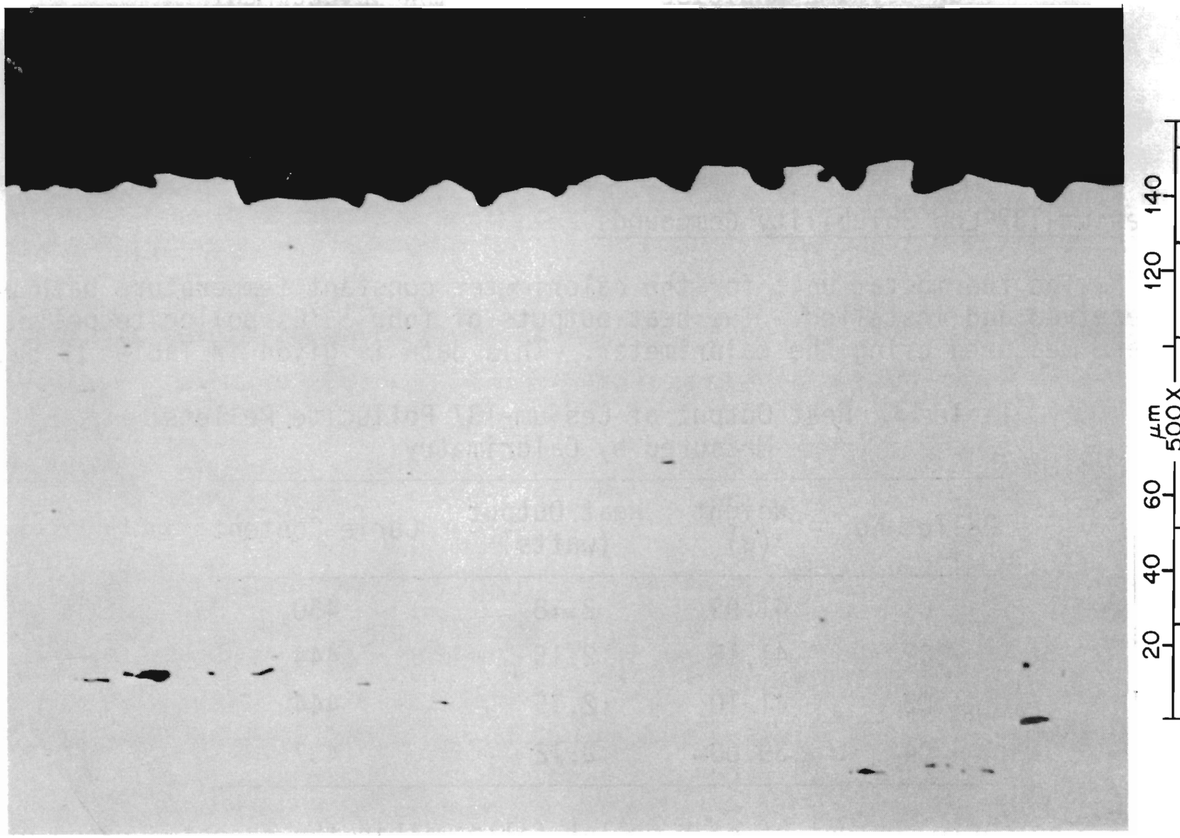

Figure 2. As polished section of WESF ^{137}Cs chloride capsule.
(Loaded September 1975 and examined in March 1980.)
Capsule was heated to 380°C for 289 days
prior to sectioning.

Krypton-85 Light Source Development

Modifications of the light pipe used to conduct light from the quartz light source provided essentially uniform light output in every direction. The modifications consisted of making the top of the pipe cone shaped instead of a hemispherical dome.

A new light source containing red phosphor was fabricated to replace one that was fabricated with non-radiation resistant quartz.

A meeting was held at ORNL with DOE Headquarters personnel (Bill Remini, Gary Bennett), the Air Force Contract Officer Captain Holden, and a DOE consultant, Frank Quinn, to review the program progress and to obtain input from the Air Force concerning program requirements. The first phase of the program, prototype fabrication, is essentially completed. Improvement in the prototype with respect to increasing light output and reduction of radiation will continue. Safety analysis for accident conditions will be started after credible accident criteria are supplied by the Air Force. Testing to determine structural durability relative to vibration and impact will also be carried out. Shipping container design and licensing requirements will be established.

BENEFICIAL APPLICATIONS OF RADIATION TECHNOLOGY

(Activity AE 15 35 00 0, WPAS BART001)

C. S. Sims

On March 11, the BART team's preliminary assessment of the Cs-137 irradiation technology program was presented to DOE, Sandia, and CH_2MHill. Several action items and directives for extensive review of the assessment resulted from that presentation and the BART program schedule has been modified accordingly.

The preliminary draft assessment was modified to reflect comments made at the March 11 meeting and was submitted on March 18 to all members of the BART assessment team for review and comment. Team members were continuing to interact with the appropriate DOE contractors relative to the review as the report period closed.

Discussions were held with Jerry Foess (CH_2MHill-Milwaukee) relative to techniques for extraction of heavy metals from sludge. Mr. Foess furnished the BART team with summary papers on the subject.

The reasoning used to set the 1 Mrad dose criterion for sludge irradiation (40 CFR 257) was discussed on March 26 in Cincinnati with Dr. Farrell and Mr. Stern of the Environmental Protection Agency. In short, the 1 Mrad value is based on the dose calculated to reduce poliovirus concentrations by two to three orders of magnitude when electron beam D_{10} inactivation data determined by A. J. Sinskey are used along with a factor for conservatism. Concerns which remain at this time are related to:

1. effectiveness of electrons versus gammas,

2. current Sandia D_{10} values for poliovirus are about double those reported by Sinskey,

3. Sinskey's data were for liquid sludges, the irradiation will be done on solids, and

4. possible misapplication of the conservatism factor.

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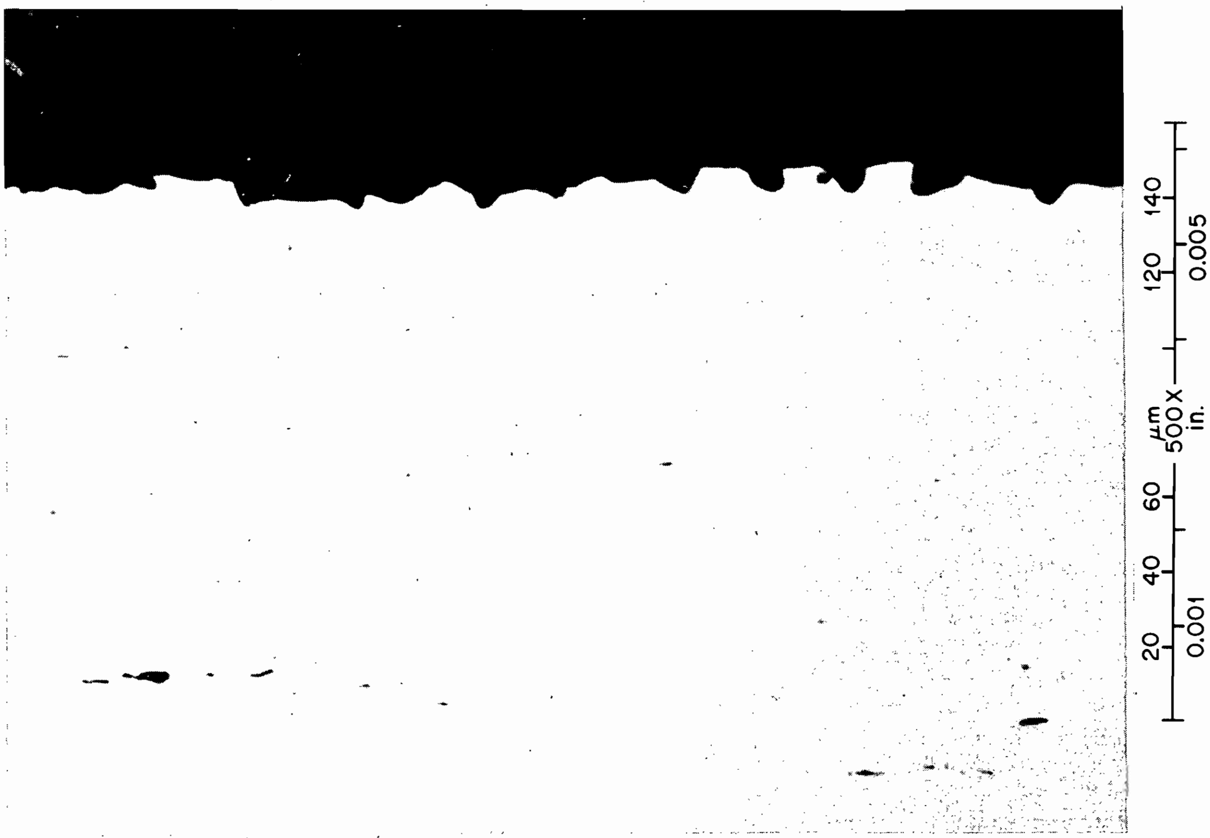

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