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QUARTERLY PROGRESS REPORT No.3

for

COMPACT THERMOELECTRIC CONVERTER DEVELOPMENT PROGRAM - PHASE IIB (U)

RCA

DIRECT ENERGY CONVERSION

CONTRACT AT(30-1)-3582

For the Period

July 1, 1967 to September 30, 1967

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**COMPACT THERMOELECTRIC CONVERTER
DEVELOPMENT PROGRAM** — PHASE II B (U)

CONTRACT AT(30-1)-3582

For the Period

July 1, 1967 to September 30, 1967

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U.S. Atomic Energy Commission

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ABSTRACT

Program effort was redirected toward more component studies and less assembly of full modules.

The principal increase of component effort was in the thermoelement assembly area where studies of alternate bonds and barrier layers were instituted. Effort in the beryllia ceramic area was increased to provide a higher quality product.

Test and evaluation was continued on the flexible connector and on the hot and cold heat exchanger plates with satisfactory results.

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I. INTRODUCTION AND SUMMARY

Program effort during this reporting period was subject to some redirection with more emphasis on components than had been previously planned. Assembly effort was continued on partial test devices, but scheduled fabrication of complete converter modules was deleted. As a result of this redirection effort, the work on element bonding was broadened to include other investigations of barrier layers and their effects. These additional bond systems are generally of the silicide and carbide types. Element work also included investigation of the use of the same grade of graphite in the "P"-element assembly as is used in the "N"-element assembly. This interchange seems feasible on the basis of experimental results.

Effort in the area of the beryllia insulator was also expanded with the prime objective of upgrading the quality of metallized ceramics and of establishing more meaningful tests and specifications.

Long term tests continued on diffusion barriers to prevent diffusion of gold through the hot and cold plate materials.

No new effort was instituted on either the flexible connector or on the hot and cold heat exchanger plates. Evaluation of tests in progress was continued, with satisfactory results.

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II. STRUCTURE EVALUATION PROGRAM

Table I presents a summary of the structures which were evaluated under simulated operational and environmental conditions during the reporting period. Comments on each structure, wherever appropriate, are presented below.

A. Two-Couple Test Structures - Linear Test Structure No. 4

The early linear test structures were designed to accomodate representative available parts which enabled the evaluation of the Compact Converter components to proceed. Round heat exchangers were employed. LTS-4 was tested to evaluate the long term performance capability of silicon-germanium-to-graphite hot shoe bonds at a hot shoe temperature corresponding to a reactor heat source application. LTS-4 contains: 1) a diffusion-bonded tungsten-gold-BeO insulator hot stack, 2) two pairs of rectangular, Phase I elements which have 30-mil graphite and tungsten shoes, copper pedestals, and tungsten compensators that are Niore-brazed to the hot stack, 3) a water-cooled, cold heat exchanger that is brazed to the structure, and 4) JTA and 8826 graphite for the "P"-and "N"-elements, respectively. LTS-4 was removed from test after accruing 3052 hours of test at an average hot shoe temperature of 700°C. LTS-4 successfully completed its test program as may be observed in Figures 1 and 2.

B. Long Term Life Capability of the Phase IIA Elements

All-Brazed Structure - Partial Test Structure No. 13

PTS-13 is a 12-couple structure having Phase IIA elements with 8826 graphite in the "N"-element and prefired JTA graphite in the "P"-element. PTS-13 was primarily established to evaluate the Phase IIA elements in an all-brazed structure under steady state conditions at a maximum hot-shoe temperature of 800°C. PTS-13 performed well until after 3300 hours of test when a severe thermal cycle was caused by a power failure. Subsequent operation revealed separation in four of the hot stacks at the nickel-to-tungsten interface. This type of separation is probably attributed to the Kirkendall-type void formation (see Stack Development of this report) which weakened the bond system to withstand the stresses of the severe thermal cycle. Operation has been stable in spite of the separation, as may be observed in the performance curves of Figure 3. PTS-13 has accrued more than 5600 hours of test.

C. Phase IIB Structures

1. Linear Test Structure No. 11

LTS-11 exhibited partial separations between the tungsten vapor plate and the 8826 graphite at the hot ends of two elements. This occurred during the initial operation of the structure at a hot shoe temperature of 800°C. Because electrical continuity was maintained, the test structure was remounted in another test station for continued evaluation of the "N"-elements using the tungsten vapor plate, and of the "P"-elements using a TiZr braze, under steady-state operation. Operation is stable after 350 hours of test as shown in Figure 4.

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TABLE I. EVALUATION OF COMPONENTS IN A STRUCTURE

STRUCTURE	HOT STACK		ELEMENTS		COLD STACK INSULATOR BOND SYSTEM ¹	HOT SHOE TEST TEMPERATURE	TEST HOURS CYCLES	REMARKS
	DIFFUSION BARRIER	OTHER COMPONENTS	'N' BOND SYSTEMS	'P' BOND SYSTEMS				
PTS-13	Ni-W	W-Au-BeO-Au-W	W-NiTi-8826	W-NiTi-JTA	Cu-BeO-Cu	800°C	5686 4	OPERATION STABLE WITH FOUR OPEN HOT STACKS WHICH SEPARATED DURING SEVERE THERMAL CYCLE AT 3344 HOURS
LTS-4	Au-W	Au-BeO-Au-W	W-NiTi 8826	W-NiTi-JTA (unfired)	Cu-BeO-Cu	700°C	2980 5	COMPLETED TEST PROGRAM
LTS-11	"	Au-BeO-Au	W-NiTi-W V P 8826	W-TiZr-JTA	Cu-BeO-Cu-W	800°C	367 4	OPERATION STABLE WITH PARTIAL SEPARATIONS IN TWO 'N' ELEMENTS WHICH APPEARED DURING INITIAL OPERATION
LTS-12	"	" "	W-NiTi-8826	" "	" "	"	2632 3	'P' RESISTANCE INCREASING
LTS-13	"	" "	" "	W-NiTi-8826 Al-SiGe	" "	"	2043 3	OPERATION SATISFACTORY
LTS-14	Ni-W	" "	" "	" "	" "	"	1734 0	" "
LTS-15	Au-W	" "	" "	" "	" "	"	1741 0	" "
LTS-16	"	" "	" "	W-TiZr-JTA	" "	"	—	BEING INSTRUMENTED AND MOUNTED FOR TEST
LTS-17	"	" "	" "	W-NiTi-JTA	Slotted Cu-BeO-Slotted Cu	"	—	" "
LTS-18	"	" "	" "	W-NiTi-8826-Al-SiGe	Cu-BeO-Cu-W	"	—	" "
LTS-19	"	" "	" "	W-NiTi-JTA	" "	850°C	—	IN FABRICATION
LTS-20	"	" "	" "	W-NiTi-8826-Al-SiGe	Slotted Cu-BeO-Slotted Cu	"	—	" "
LTS-21	"	" "	" "	" "	Cu-BeO-Cu-W	"	—	" "

NOTES 1 ALSO INCLUDED IN THE COLD STACK ARE A Cu PEDESTAL, W COMPENSATOR, Cu ELECTRICAL CONNECTOR, Cu FLEXIBLE CONNECTOR AND THE 316L SS COLD HEAT EXCHANGER.

2 TEST PURPOSE

- a THE 'N' & 'P' ELEMENT BOND SYSTEMS ARE BEING EVALUATED IN ALL STRUCTURES
- b THE DEVELOPMENT MODULE DESIGN HOT STACK IS BEING EVALUATED IN LTS-14
- c THE COLD STACK IS BEING EVALUATED IN LTS 17 AND LTS-20

3 ALL HOT HEAT EXCHANGES ARE HASTELLOY "B" EXCEPT THE LTS-4 EXCHANGER WHICH IS HASTELLOY X

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FIGURE 1. MODULE LTS-4 COUPLE NO. 1, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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FIGURE 2. MODULE LTS-4 COUPLE NO. 2, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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FIGURE 3 - PTS-13, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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FIGURE 4. MODULE LTS-11, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE vs. TIME.

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2. Linear Test Structure No. 12

LTS-12 is of the same design as LTS-11, except the "N"-elements are of the Phase IIA design without the tungsten vapor-plated graphite at the tungsten-to-graphite interface. As reported earlier, the tungsten layer on the graphite in the "N"-elements was considered to serve as a barrier to nickel-titanium braze penetration into the graphite to prevent possible subsequent interaction with the silicon-germanium. Review of the structure performance data of Phase IIA "N"-elements, which do not have the tungsten vapor-plated graphite, indicated that the use of a barrier may be unnecessary. This finding supported the withdrawal of a tungsten layer in elements from future structures. As shown in Figure 5, LTS-12 is performing satisfactorily after 2500 hours of steady-state operation at a maximum hot shoe temperature of 800°C, although the "P"-resistance is starting to increase.

3. Linear Test Structure No. 13

LTS-13 was placed on steady-state operation at a hot shoe temperature of 800°C to evaluate an alternate "P"-element design which uses 8826 graphite bonded to the silicon-germanium with an aluminum shim which acts as a dopant; see later discussions. LTS-13 is performing well after more than 2000 hours of operation as shown in Figure 6.

4. Linear Test Structure No. 14

LTS-14 was primarily established to evaluate the performance capabilities of the diffusion barrier system of the Development Module Design under steady-state operation at a hot shoe temperature of 800°C. LTS-14 is of the same construction as LTS-13 except for the diffusion barrier system of tungsten CM-50 brazed to nickel. LTS-14 is performing well after 1700 hours of operation as indicated in Figure 7.

5. Linear Test Structure No. 15

LTS-15 was established primarily to evaluate the performance of the alternate "P"-element under a steady-state test program at a maximum hot shoe temperature of 800°C. LTS-15 is of the same design as LTS-13. LTS-15 is performing satisfactorily after 1700 hours of test as shown in the performance curves of Figure 8.

D. Evaluation Test Conditions

All structures are tested in oil-diffusion pumped vacuum stations at pressures ranging from 10^{-5} to 10^{-6} Torr. All tests were conducted with electrical heaters. LTS-4, which is constructed on a tubular hot heat exchanger, used a graphite heater shaped to provide the desired temperature profile along the tube. The later LTS and PTS structures are being heated by planar graphite heaters which are machined so that both electrical connections can be made at one end of the structure. The heaters are insulated from the heat exchanger plate by thin strips of mica.

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FIGURE 5. MODULE LTS-12, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME.

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FIGURE 6. MODULE LTS-13, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME.

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FIGURE 7. MODULE LTS-14, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE vs. TIME.

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FIGURE 8. MODULE LTS-15, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE vs. TIME.

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The temperatures of all structures on life test are determined by the use of chromel-alumel thermocouples. Most thermocouples have been attached by spot welding, but in some cases, the thermocouple has been inserted into small holes drilled in the structure for this purpose. The thermocouples on the inside of the cold heat exchanger plate were brazed in-place during fabrication. All voltage and resistance measurements of individual elements and couples are made by using the thermocouple wires. Total voltage and resistance of each structure were measured through copper wires.

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III. THERMOELEMENT

A. Establish Bond Facilities

The initial thermoelements, fabricated in the additional facilities which were required to meet the increased demand for parts during Phase IIA of the program, have accrued 8500 hours in isothermal storage testing at 800°C. At the last downtime, 7000 hours, the samples were subjected to electrical and metallurgical examinations. The results of these examinations were covered in the previous quarterly report.

In addition to establishing this second facility, investigations were made and the results evaluated of equipment which had greater capacity but which used slightly different bonding techniques. These samples have accrued 8500 hours of isothermal storage at 800°C. They were last examined at 7000 hours and were also reported in the previous quarterly report. These samples have been scheduled for evaluation at downtime intervals of 3000 hours for the remainder of the test.

B. Process Development and Design Studies

1. Voids in "P" SiGe-JTA Bonds

An alternate approach to achieving thermoelements with low voids and low contact resistance is to use 8826-type graphite. To reduce the contact resistance which is normally above design limitations in a "P" SiGe - 8826 system, very thin aluminum shims are used at the graphite-silicon germanium interface. This effort has been initiated and samples are being evaluated in isothermal storage and operational life tests. The "as-bonded" contact resistance in the coupon configuration has averaged 0.12 milliohm compared to 0.60 milliohm in samples with no aluminum and 0.17 milliohm in samples with JTA graphite. Samples prepared for the feasibility study of this type element have accrued 9500 hours at 900°C in isothermal storage, and have exhibited an increase in resistance less than the standard "N"-thermoelement after comparable times at temperature. Samples have been subjected to the thermal cycling in the fabrication of a module and were subsequently placed in isothermal storage tests at 900°C. They were evaluated after the first downtime at 500 hours. There was no change evident in the metallurgical or electrical evaluations at this point when compared to the "acceptance aged" samples.

2. Acceleration Factor

This study was initiated to determine an acceleration factor which could be applied to test programs in which evaluation of the characteristics of the element as a function of time and temperature is a requirement. A series of thermoelements were prepared using JTA graphite in "P"-thermoelements and both 8826- and 950S-type graphites in "N"-thermoelements.

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The 950S graphite samples were used as controls in the test for comparison with similar samples prepared during Phase IIA. The tungsten shoe was 40 mils thick and the JTA graphite was pre-fired for bonding. These are standard system samples using siliconized graphite in the "N"-thermoelement, a nickel-titanium braze system, and a one-step bonding operation. The acceleration factor is to be determined by a comparison of electrical resistance and metallurgical evaluations of the following conditions as a function of time at temperature:

1. Void levels in the tungsten graphite bond
2. Void level in the silicon-germanium-graphite bond
3. Changes in the graphite shoe material.

Current data is summarized in Table II. Comparison of the data at 400 hours for 800°C and for 900°C indicates that some accelerated deterioration is occurring and is evident from the higher electrical resistance in the 900°C samples. The evaluation will continue to determine if this is a trend toward acceleration or if it is a function of the silicon-germanium bulk material.

3. Effects of Processing on Element Performance

A major difference between isothermal storage testing and operational tests (use in modules) is that in the latter evaluations, samples are subjected to thermal cycling involved in the further processing required for the addition of the hot and cold stacks to the elements. These fabrication steps involve variations in pressure, temperature, and environment for brazing or diffusion bonding techniques.

Samples were prepared and subjected to the processing encountered in module fabrication. They were placed in isothermal storage tests and have accrued 500 hours at temperature. The electrical resistance values have shown no significant increase and were still well below the design limitations.

4. NiTi Control

During Phase IIA, the importance of controlling the composition of the nickel-titanium braze was investigated. Samples which had a high nickel content had a high rejection rate for contact resistance. The conclusion was that this deficiency resulted from the formation of a nickel-rich system which moved by mass flow or capillary action through the porous graphite. Limits were specified for the nickel-plate thickness based on the control capability of the plating process. However, these limits are not sufficiently stringent to assure a reasonable yield.

Consequently, a feasibility study was initiated, using nickel shim material as a replacement for the plated nickel on the tungsten shoe. Samples are being prepared with various compositions in the NiTi braze to determine its mechanical and metallurgical properties.

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TABLE II

SUMMARY OF ROOM TEMPERATURE RESISTANCE ON TESTS TO DETERMINE ACCELERATION FACTOR

Test	Hours at 900 °C	Hours at 800 °C	Resistance in Milliohms				
			R _t	R _g	R ₁	R ₂	R ₃
JTA Graphite	0		2.65	2.58	0.04	0.15	0.19
	16		2.71	2.61	0.04	0.14	0.19
Phase IIB	500		2.80	2.71	0.04	0.14	0.18
	2500		2.71	2.63	0.03	0.18	0.21
	4000		2.73	2.68	0.02	0.22	0.24
		500	2.71	2.64	0.04	0.14	0.20
		2000	2.83	2.72	0.05	0.17	0.22
8826 Graphite	0		2.59	2.55	0.02	0.12	0.14
	16		2.62	2.58	0.03	0.13	0.16
Phase IIB	300		2.73	2.62	0.01	0.17	0.18
		500	2.76	2.72	0.01	0.16	0.17
	1000		2.58	2.50	0.01	0.15	0.16
	2500		2.83	2.72	0.01	0.17	0.18
	4000		2.95	2.92	0.01	0.16	0.17
		2000	2.65	2.63	0.01	0.13	0.14
		4000	2.70	2.68	0.01	0.14	0.15
950S Graphite Phase IIB	0		2.14	2.03	0.06	0.10	0.16
	16		2.24	2.15	0.06	0.11	0.17
	500		2.63	2.48	0.06	0.12	0.18
		500	2.63	2.49	0.08	0.13	0.21
	2500		2.30	2.11	0.05	0.13	0.18
		2000	2.53	2.38	0.07	0.12	0.19
	4000		2.61	2.57	0.05	0.15	0.21
		4000	2.65	2.47	0.07	0.13	0.19
950S Graphite Phase IIA	0		2.14	2.04	0.06	0.11	0.17
	16		2.20	2.17	0.06	0.11	0.17
	500		2.36	2.24	0.06	0.12	0.18
		2000	3.32	2.18	0.06	0.12	0.18
	2500		2.30	2.11	0.04	0.13	0.17
	4000		2.41	2.31	0.05	0.14	0.19
		4000	2.51	2.34	0.06	0.14	0.20
		6000	2.61	2.53	0.04	0.14	0.19

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5. Hot Resistance

One of the objectives of the isothermal life tests is to determine the effect of time at temperature on the total resistance at operating conditions. Accordingly, provisions have been made to remove samples at regular intervals for hot resistance measurements.

An apparatus has been completed for the measurement of total element resistance at elevated temperatures up to 900°C. The accuracy of the equipment was evaluated, and the result was that for a molybdenum test piece, the resistance at 775°C agreed within $\pm 1.5\%$ of the literature values. Measurements to determine repeatability over a range of temperature from 400°C to 850°C were found to repeat within $\pm 1.0\%$. The precision of the equipment was determined and found to be $\pm 2.5\%$.

With the equipment completed and in operating condition, evaluation has been initiated of the accumulated samples. Initially, while developing a technique and training personnel in the operation of the equipment, samples for measurement were randomly selected. Both "P"-and "N"-thermo-elements were measured over a range of temperatures up to 850°C. All 10 "P"-samples followed the same pattern on a total resistance-versus-temperature plot and indicated a straight line increase in resistance up to 850°C. All the "N"-elements which were measured showed an increase up to 700°C. Between 700°C and 850°C, the resistance remained steady or decreased slightly. Among the 20 samples measured, were a series of "N"-coupons from one test which had run up to 1000 hours at 900°C, and "P"-thermoelements in the coupon configuration from another test which had run up to 300 hours in isothermal storage at 900°C.

The initial observation was that the data lacked reliability in that the behavior was contrary to established trends for silicon-germanium. Also noted was that the hot resistance values were directly related to the cold resistance values which are highly sensitive to test cooling conditions. Accordingly, a screening test was initiated to determine the procedure and to establish the methods necessary to overcome these effects. A new program was developed in an attempt to determine the effect of cooling rate on hot resistance values. Samples are being fabricated for this program.

6. Siliconizing

Under an RCA Applied Research Program, examination determined that prewetting the graphite surface prior to bonding is definitely advantageous in establishing long, high-temperature life. Investigation of various surface treatments conducted under this Program indicated that firing the coating of silicon assured uniform wetting during the bonding procedure. The disadvantage of the siliconizing, however, was that the wafers became slightly domed because of the different thermal expansion coefficients. The amount of doming can be minimized by controlling the amount of silicon applied to the graphite shoe. The initial method utilized a spray technique which produces a range in weight of silicon applied.

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To achieve a better control and a uniform silicon weight on a large number of shoes, the feasibility is being studied whereby the silicon is applied by silk screen techniques. The coated shoes are being evaluated for uniformity of coating and range of coating weights. Thermoelements which use these graphite shoes will be subjected to isothermal storage testing and compared with the standard coupon on which the silicon is applied by spray-coating techniques.

7. Higher Si-Content SiGe

Two of the objectives are to refine and upgrade the graphite-bonded thermoelement to take full advantage of the high temperature capability of silicon-germanium. The operating temperature of the module may be raised by increasing the silicon content of the silicon-germanium and by developing or revising the bonding technique to achieve uniform, low resistance, high mechanical strength thermoelements. A program to develop this higher temperature capability has been initiated and the first samples have been prepared and are being evaluated. The silicon content in these samples is 70 atomic weight percent. The proposed plan is to evaluate up to 85 percent silicon at an operating temperature of between 950°C and 975°C.

C. Materials and Bonds Evaluation

The storage life test evaluation of the Phase IIA elements continued during the reporting period. The test samples have variations in thickness of both the tungsten and the graphite shoes. The samples for these tests are in the coupon configuration, and the controls are in the Phase IIA design with 40-mil tungsten shoes and 30-mil graphite shoes. Electrical, physical, and metallurgical examinations are performed at each downtime. The average values for total and contact resistances are shown in Table III. There has been no significant change in any of the values and all are within the present design objectives.

D. Alternate Bonds

1. Titanium-Chromium Silicide Process Development

Titanium-chromium silicide is being evaluated for high temperature conductive bonds for thermoelements between the tungsten shoe and the silicon-germanium. The preparation and application of the (Ti, Cr) Si_x can be achieved by pack cementation to deposit chromium on the tungsten substrate, by the fused salt bath process to deposit titanium on the chromium precoated tungsten substrate, and finally by low pressure pack deposition of silicon. The experimental progress of the three processes is discussed below:

a. Deposition of Chromium

Variables involved in the pack cementation process have been investigated to a limited extent and reproducible results have been achieved. The average chromium-diffusion coating on tungsten is 0.2 mil thick.

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TABLE III

SUMMARY OF AVERAGE RESISTANCE VALUES AS A FUNCTION OF TIME AT 900°C
ROOM TEMPERATURE RESISTANCE IN MILLIOHMS

Sample Description	Test No.	Type	Hours at 900°C	R _t	R _g	R ₁	R ₂	R ₃	SiGe*	Predicted for SiGe	% Voids in SiGe Graphite Interface
Control	1	N	0	2.14	2.04	0.06	0.11	0.17	1.80		1
40-mil Tungsten			16	2.20	2.12	0.06	0.11	0.17	1.86		1
30-mil Graphite 950S			425	2.36	2.24	0.06	0.12	0.18	2.00		11
			1050	2.32	2.18	0.06	0.12	0.18	1.96	1.97	1
			2546	2.30	2.11	0.04	0.13	0.17	1.96		1
			4000	2.35	2.23	0.06	0.12	0.18	1.99		
			6000	2.38	3.34	0.04	0.13	0.16			
20-mil Tungsten	2	N	0	2.18	2.11	0.04	0.15	0.19	1.83		1
30-mil Graphite 8826			16	2.27	2.22	0.04	0.13	0.17	1.93		1
			425	2.38	2.34	0.04	0.15	0.19	2.00		1
			1050	2.33	2.28	0.06	0.13	0.19	1.95	1.99	1
			2546	2.27	2.22	0.04	0.15	0.18	1.91		1
			4000	2.30	2.20	0.05	0.14	0.19	1.92		
			6000	2.34	2.26	0.04	0.14	0.18			
Control	3	P	0	2.65	2.58	0.04	0.15	0.19	2.27		50.2
40-mil Tungsten			16	2.71	2.61	0.04	0.14	0.19	2.33		52.0
30-mil Graphite JTA			425	2.80	2.71	0.04	0.14	0.18	2.44		77.8
			1050	3.09	2.98	0.02	0.17	0.19	2.71	2.44	74.7
			2546	3.19	3.16	0.03	0.20	0.22	2.75		54.0
			4000	3.22	3.14	0.05	0.15	0.19	2.84		
			6000	3.31	3.21	0.05	0.12	0.17			
40-mil Tungsten	4	P	0	2.38	2.36	0.02	0.12	0.14	2.10		46
20-mil Graphite JTA			16	2.41	2.39	0.02	0.12	0.14	2.13		82
			425	2.64	2.63	0.02	0.14	0.16	2.32		88
			1050	2.75	2.72	0.02	0.18	0.20	2.35	2.23	84
			2546	2.84	2.92	0.03	0.17	0.19	2.46		61
			4000	2.01	2.93	0.04	0.16	0.20	2.61		
			6000	2.46	2.44	0.02	0.17	0.18			
Phase I	5	P	0	2.64	2.61	0.02	0.13	0.15	2.34		1
20-mil Tungsten			16	2.68	2.63	0.03	0.14	0.17	2.34		5.8
20-mil Graphite JTA			425	2.81	2.78	0.02	0.14	0.16	2.49		5.1
			1050	3.00	2.92	0.04	0.17	0.21	2.48	2.48	7.0
			2546	3.16	3.04	0.04	0.17	0.21	2.74		6.0
			4000	3.42	3.32	0.05	0.17	0.22	2.98		
			6000	3.63	3.55	0.03	0.18	0.22			

* Total Resistance R_t less the contact resistances (2 x R₃).

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It is difficult to achieve a thicker chromium diffusion coating on tungsten because tungsten is the element with the highest melting point, and chromium and tungsten form a complete series of solid solutions. Therefore, tremendous energy is required to create vacancies in tungsten and to mobilize it to undergo substitutional diffusion with chromium.

b. Deposition of Titanium

Process variables involved in the fused salt bath deposition of titanium were investigated. Reproducible results were achieved and a process has been established. The average titanium coating thickness is 1.0 mil.

c. Deposition of Silicon

Process variables involved in the low pressure diffusion process for deposition of silicon are being investigated. No reproducible results have been achieved to date.

2. Development of Improved Processes for Preparation of Tungsten Carbide-Silicon Carbide as a High Temperature Conductive Bond

In the past, this bond material was obtained by imbedding tungsten shoes in active carbon powder contained in a graphite boat, and heating the contents at a relatively high temperature for one hour followed by quick cooling. Siliconizing is also performed at a relatively high temperature. At such high temperatures, the recrystallization of tungsten is rapid and so severe that it weakens the element. The quick cooling procedure usually causes cracks in the carburized zone which sometimes extend into the silicon carbide layer.

A new approach now under examination is performed at temperatures a few hundred degrees lower than those used in present methods. Preliminary results are very promising; the tungsten carbide shows no cracks, and a layer of extremely adherent pyrolytic graphite always forms on the carburized surface. This layer of pyrolytic graphite can remain on the surface and be utilized as the source of carbon for the formation of silicon carbide as the top layer. Experimental results are being evaluated.

3. Bonding Development

a. (Ti-Cr) Si on Tungsten

Coated pieces of tungsten were further siliconized by the silk-screen method. As mentioned previously, the silicon is initially applied to form the (TiCr) Si coating, using a low pressure pack process. The purpose of the additional siliconizing is to assure that an excess of silicon is available on the bonding surface.

Bonds were made, using the silk-screened pieces for comparison with joints to the pieces which were only pack-siliconized. The SiGe bonded

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to the silk-screened surfaces, but did not bond to the others. Acceptance testing, involving exposure of the bonds to 1000°C for 16 hours, resulted in separation of all bonds. This failure was apparently caused in part by lack of wetting at the interfaces.

Work is continuing on the development of the silicon layer. Besides the effort in pack deposition, epitaxially-grown silicon layers are being investigated. The use of alternate substrates in conjunction with these coatings is also under consideration.

b. SiC on Carburized Tungsten

Tungsten discs were carburized by packing them in active carbon and fixing them at relatively high temperatures. A silicon-carbide layer was then vapor-deposited on the carburized surface after which the elements were assembled and bonded. They were then exposed to acceptance testing for 16 hours at 1000°C and, after inspection, were exposed to similar conditions for an additional 88 hours. Metallographic examination of these life-tested bonds indicated that the SiC was successful in preventing reaction between the tungsten and the silicon of the pellet except in the isolated areas where small separations existed in the SiC layer. Pieces of similar structure but without the SiC barrier layer showed deep penetration of the silicon into the tungsten where silicides were formed. Extensive germanium-rich areas resulted at this bond interface.

Additional life testing of the SiC-coated carburized tungsten for a total of 287 hours at 1000°C in vacuum was conducted. At that time, inspection indicated that reaction had occurred. These pieces are in process of evaluation.

c. WC on 8826 Graphite

A series of tests were conducted to determine the effectiveness of WC as a means of achieving an element capable of higher temperature performance. Those materials were prepared by vapor-plating 8826-graphite pieces with tungsten. The plated pieces were carburized using a high temperature carburizing process which produced two layers of tungsten carbide separated in sections by a thin layer of tungsten. Bonds were made to SiGe.

Metallographic examination of the interfaces in the as-bonded condition showed that a germanium-rich layer existed and that silicides had formed. The reaction extended through approximately one third of the WC layer. Acceptance test exposure at 1000°C for 16 hours in vacuum resulted in complete separations at most of the interfaces in the germanium-rich zone. The reacted carbide and silicide zone remained bonded to the graphite but with extensive peeling evident in some samples at the extremities.

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d. SiC on 8826 Graphite

SiGe bonds were made to SiC-coated 8826-graphite shoes. The elements were acceptance-tested for 16 hours at 1000°C and after inspection were returned for an additional 88 hours at 1000°C. Metallographic examination of the bonds showed no reactions or Ge enrichment of the interfaces caused by the thermal exposure. Additional testing is scheduled for this system, and includes fabrication of additional undoped SiC elements and the development of doped SiC layers to bring the electrical resistance level into a more desirable range.

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IV. BeO CERAMICS

A. Specifications for Metallized BeO

During the report period, effort continued in upgrading the quality of metallized BeO ceramic insulators by establishing more rigid specifications for commercial suppliers of BeO. Vendor E has accepted the new specification for his product; acceptance by Vendors A and C remain to be made.

B. BeO Metallizing Adherence Tests

Results of brazed peel tests conducted by RCA disagreed with those performed by Vendor C. This variance has prompted a thorough review of all the facets of this test at both RCA and Vendor C's. Preliminary indications are that the brazing schedule for this test is very critical. The standard metallizing adherence test consists of a Nicro-brazed, ceramic-moly strip assembly in which the moly strips are bent and peeled away from the ceramic. Vendor C reported a Nicro-brazing schedule of 975°C for 2-1/2 minutes whereas RCA employs a brazing schedule of 1025°C for the same time. Microscopic examination of polished sections of samples which were brazed by Vendor C and which passed metallizing adherence tests (failure in the moly strip) indicated many voids in the Nicro-braze area. This lack of wetting of the moly strips with subsequent void formation is characteristic of cold brazing conditions. Examination of polished sections of samples from this same lot of ceramics which were brazed by RCA and which failed metallizing adherence tests (failure in metallizing layer) showed hardly any voids in the Nicro-braze area. These two structures are illustrated in Figure 9. Figure 9A shows the large voids in the Nicro braze and the moly-strip failure characteristic of Vendor C's tests. Figure 9B shows the lack of such voids in the samples brazed by RCA and the failure in the metallizing layer.

Additional microscopic examination of recent metallizing adherence-test samples seemed to confirm the fact that, in general, samples which pass metallizing adherence tests tend to be associated with cold Nicro-brazing conditions. Engineering tests are now under way to determine optimum time-temperature schedules for brazed metallizing adherence test samples. Brazes other than Nicro will also be investigated.

The expected difficulties with the Nicro-brazed peel test led to the development early in 1967 of new types of metallizing adherence tests. Under investigation are a diffusion-bonded tensile test, a diffusion-bonded shear test, and a diffusion-bonded peel test. Preliminary results indicate that all three of these tests are capable of distinguishing between good and bad lots of metallized BeO. Additional tests will be initiated next quarter to secure correlation between these preliminary results and the test results of simulated thermoelectric devices on structures.

C. Metallizing Process Parameter Investigation

Results of metallizing adherence tests of ceramics metallized by RCA in Vendor B's plant indicated poor metallizing adherence values when tested by

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← BeO
← Moly MET.
← Nioro
← Moly Strip

MAG 145X

NIORO-BRAZED AT 975°C FOR 2-1/2 MINUTES BY VENDOR C. SAMPLE PASSED METALLIZING ADHERENCE TEST. FAILURE WAS IN THE MOLY STRIP. COLD NIORO BRAZE - NOTE THE VOIDS BETWEEN THE METALLIZING LAYER AND THE MOLY STRIP.

A

← BeO
← Moly MET.
← Nioro
← Moly Strip

MAG 145X

NIORO-BRAZED AT 1025°C FOR 2-1/2 MINUTES BY RCA. SAMPLE FAILED METALLIZING ADHERENCE TEST. FAILURE WAS MOSTLY IN THE METALLIZING LAYER. NOTE THE LACK OF VOIDS IN THE NIORO BRAZE AND THE COMPLETE WETTING OF THE MOLY STRIP BY THE NIORO BRAZE.

B

FIGURE 9. VENDOR C BeO CERAMICS METALLIZING ADHERENCE TEST SAMPLES

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means of Niore-brazed peel samples, and fair to good values when tested with diffusion-bonded tensile samples. These results agree with those noted for Vendor C's product. Consequently, the metallizing test will be repeated, using these samples, particularly the marginal lots, to develop more realistic and reliable metallizing adherence tests for incoming products.

D. Materials and Bonds Development and Evaluation

A coupon program was initiated to study the effects of bonding to various materials and of processing on the physical and electrical properties of the BeO insulator as a function of time and temperature. The following variables are being evaluated:

1. The various suppliers of BeO
2. Platings on the metallized ceramics
 - a. Electroless vs. Electrolytic Nickel
 - b. Iron vs. Nickel
3. The effects of diffusion-bonding copper and gold to the insulator.

The samples are being bonded into sandwiches which simulate a portion of a structure. The samples bonded with nickel-copper are to be storage life tested at 400°C and 650°C, while those bonded with nickel-gold will be aged at 800°C and 900°C. In addition to the sandwiches, metallized ceramics and metallized and plated insulators will be subjected to the same times at temperatures to establish a reference plane of measurement. Samples will be removed from storage life testing at periodic intervals and will be evaluated by metallographic examination, tensile testing, and measurement of thermal conductivity and electrical resistivity at the testing temperatures. These properties (strength, electrical resistance, and thermal conductance) will be plotted as a function of time at temperature for the variables under consideration.

E. Electrical and Thermal Properties of BeO

Work was initiated during this quarter on the final assembly and de-bugging of equipment to measure both the thermal and electrical properties of BeO at elevated temperatures. Preliminary measurements are expected to be made at the beginning of the next quarter.

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V. DIFFUSION BARRIERS

A. Coatings

Chromium silicide is being evaluated as a diffusion barrier for the gold-nickel (Hastelloy B) couple. Efforts were directed toward the development of processes for preparing chromium silicide and applying it to Hastelloy B. The fused salt bath process was chosen for the study of codeposition of chromium and silicon to form the chromium silicide. The results of an examination of process variables and of reproducibility were that the bath composition, temperature, and time are the most influential variables, and that experimental results were found to be partially reproducible.

A fused salt bath composition has been tentatively optimized. The effects of processing temperature and time on deposition are illustrated in Figures 10 through 12. These specimens of chromium silicide were treated in a salt bath of the tentative optimum composition at various temperatures and times which were proportional to the rate of deposition. Further investigation of process variables and reproducibility will be undertaken in the near future.

A thermal storage life test program was designed for evaluating the barrier properties of the chromium silicide as an interlayer between gold and nickel.

Pieces of Hastelloy B 1/2 inch square and 0.025 inch thick were coated with Cr_xSi_y . The resultant weight-gain was noted after suitable washing removed salt contamination from the pieces. Pure gold squares, measuring 0.300 x 0.300 x 0.010 inch were centrally located on, and diffusion-bonded to the coated Hastelloy pieces. Figure 13 shows the cross section of such a bond. The bonded pieces were cut in halves. One half was selected for life test; the mating half was to be used as a control during the life test. The cross sections were examined at 20X magnification to verify the continuity of the bond across the interfaces.

The life-test program including the downtimes, temperatures of exposure, and testing media is shown in Table IV. Most of the study will be conducted at 900°C. Pieces will be tested at 800°C and 950°C to establish a family of curves which represent the diffusion rate as a function of temperature for varying amounts of time. The effects of the thermal exposure on bond strength will also be determined as a function of time.

The measurement of the diffusion of gold into nickel will be determined by several methods. First, metallographically-prepared samples will be examined at 500X magnification and the penetration depth measured. By comparing this value with that obtained from the mating half, which is the control piece, the net frontal shift may be determined. Second, the electron beam probe will be used to determine the penetration depths of the nickel and gold. In this case, a series of profiles will be made parallel to the bond interface and at increasing distances from it. Thus the gold concentration change may be determined as a function of position relative to a fixed reference plane. The electron beam probe is also usable in identifying compounds formed as a result of the processing or of the thermal exposure of the specimens. Test

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FIGURE 10. CHROMIUM SILICIDE DEPOSIT ON HASTELLOY B FROM A SALT BATH OF TENTATIVE OPTIMUM COMPOSITION AT 950°C FOR 4 HOURS.297X.

FIGURE 11. CHROMIUM SILICIDE DEPOSIT ON HASTELLOY B FROM A SALT BATH OF TENTATIVE OPTIMUM COMPOSITION AT 1000°C FOR 1 HOUR.297X.

FIGURE 12. CHROMIUM SILICIDE DEPOSIT ON HASTELLOY B FROM A SALT BATH OF TENTATIVE OPTIMUM COMPOSITION AT 1100°C FOR 5 HOURS.297X.

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FIGURE 13. GOLD - Cr_xSi_y - HASTELLOY B
DIFFUSION BOND.

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TABLE IV
Cr-Si₂ DIFFUSION BARRIER LIFE TEST

<u>Downtimes</u>	<u>Hours</u>	No. of Pieces Tested				<u>Total Pieces Life Tested</u>
		<u>Exposure:</u> <u>Temperature:</u>	<u>800°C</u>	<u>Vacuum</u> <u>900°C</u>	<u>Air</u> <u>900°C</u>	
As bonded	1	All Pieces Bonded 800°C 1 Hour				
	25			2		2
	50			4		4
	75			2		2
	100	1	4	1	1	7
	250		2			2
	500	1	4	1	1	7
	750		2			2
	1,000	1	4	1	1	7
	2,000		2			2
	3,000		4			4
	4,000		2			2
	5,000	1	4	1	1	7
	7,000		2			2
	8,500		4			4
	10,000	1	4	1	1	7
	11,000		2			2
	12,000		4			4
	13,000		2			2
	14,000		4			4
	15,000	1	4	1	1	7
	17,500		2			2
	20,000	1	4	1	1	7
	25,000	1	4	1	1	7
	30,000	1	4	1	1	7
	35,000	1	4	1	1	7
	40,000	1	4	1	1	7
	45,000	1	4	1	1	7

Total -- 124

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specimens are in varying stages of preparation and will include representative samples of the improved, thicker Cr_xSi_y .

B. Refractories and Ceramics

The evaluation program of Fosterite ceramic as a diffusion barrier, continued in storage life testing at 800°C and 900°C. The samples examined after 1000 hours of life at both temperatures remain unchanged. There is no evidence of deterioration, cracking, penetration, or diffusion-zone growth. The Fosterite ceramic appears to be a most promising barrier based on its chemical stability, but its relatively high thermal impedance makes it less desirable and would only be considered as an interim system.

Rhodium, Rhenium, and Iridium are the noble refractory metals under consideration as barriers. The three are being evaluated as 1 mil shims directly adjacent to the Hastelloy B to which the gold ductile member is diffusion-bonded. Rhodium is also being evaluated as a plating on the Hastelloy. Samples that have accumulated 1000 hours at 900°C and 2000 hours at 800°C have been examined. The Iridium appears to be the most promising of the three metals. Although there is some indication of gold penetrating into the Iridium, there is no metallographic evidence of gold penetrating into the Hastelloy B or of nickel penetrating into the gold. In all three cases, the shims had multiple vertical cracks which occurred during initial bonding. Where cracks exist, the interdiffusion of metals is rapid and appears similar to bonds made without barriers. If Iridium proves to be an acceptable barrier, process development will be required to optimize the bonding conditions to prevent the cracking of the shim.

Tungsten vapor deposited on Hastelloy B does not seem to be acceptable as a barrier. Vertical cracks occur with time at temperature and the anticipated barrier effect is lost. Where no cracks exist, nickel diffuses completely through the tungsten within 1000 hours at 900°C, and then interdiffusion readily occurs between the gold and the Hastelloy.

NOTE: The discussion of the tungsten-diffusion barrier used as a bulk material is presented under the Hot Stack section of this report.

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VI. STACK DEVELOPMENT

A. Hot Stack

The program to optimize the Development Module Hot Stack Design specified at the end of Phase IIA continued. Sufficient data has been accumulated to date to establish some tentative conclusions regarding the materials and bonds, although more data is required before final conclusions may be drawn.

A complete discussion of all the data will be presented in the Phase IIB final report. The tentative conclusions are:

1. The optimum thickness of the tungsten diffusion barrier is 20 mils. Thicker members were found to crack under initial cycling conditions. Parts thinner than 20 mils developed cracks with time at temperature. The thinner the part, the more extensive the cracking, whereas the 20-mil part showed no evidence of cracking after 1100 hours at 900°C.
2. Diffusion-bonding of the hot stack where CM50 is utilized as a bonding shim is superior to brazing the members using the same CM50 material with respect to deterrence of Kirkendall-type void formation. Although the data is limited, a three-fold increase in life would be expected of the diffusion-bonded system over the brazed system.
3. Diffusion-bonding of tungsten direct to nickel, without intermediate shims, results in a much shorter life than in the CM50 brazed system; 100 percent separation occurs within 500 hours at 900°C. The diffusion rates appear to be faster when pure metals are in contact. Introduction of CM50 with its addition of Si and B as either a brazing or a diffusion-bonding shim, inhibits diffusion and, therefore, increases the life expectancy of the bond.
4. The introduction of small amounts of other high temperature materials seems to inhibit the interdiffusion rates and thus increases life capability. However, it is emphasized that this conclusion is based on extremely limited data. Rhodium plating and thoriated tungsten are two materials that act in this manner. Further time at temperature will be required to verify these preliminary results and to determine which of the various materials is most beneficial in retarding interdiffusion.

B. Cold Stack

Effort is being directed toward developing a suitable method for incorporating the beryllia insulator into the cold stack. The most promising method utilizes an integral copper pedestal which is diffusion-bonded to the beryllia insulator. (See Figure 14) A successful process for diffusion-bonding Cu to BeO has been developed. However, further effort is required to optimize the bonding pressure to prevent cracking of the ceramic. Previous data from screening tests revealed that a structurally sound Cu-BeO bond can be assured by using the pedestal concept in conjunction with diffusion-bonding techniques.

FIGURE 14. DIFFUSION-BONDED COPPER INTEGRAL PEDESTAL BeO
INSULATOR CONCEPT-CROSS SECTIONAL VIEWS.

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Two methods have been investigated. The first shown in Figure 15 utilizes a plain pedestal. Successful diffusion bonds were achieved, but some difficulty was encountered in subsequent attaching operations. Brazing this subassembly into a structure creates fillets around the copper thereby eliminating the desired pedestal effect. Figure 14 shows the more recently developed integral pedestal which, while retaining the pedestal effect, provides a shield against braze runover and the subsequent formation of fillets. In this concept, the copper pedestal retains its configuration during diffusion-bonding and in subsequent handling operations which allows proper contact to the flexible connector.

A group of coupons utilizing the pedestal configuration shown in Figure 15 is being subjected to long term storage life at 450°C to determine the metallurgical characteristics of the diffusion-bonded system. Evaluation downtimes are scheduled at 3,000 and 6,000 hours, and at every 2,000 hours thereafter up to and including 20,000 hours.

A second group incorporated the integral pedestal shown in Figure 14. These coupons were subjected to 1, 5, 10, 20, and 50 rapid thermal cycles at 405°C maximum temperature. Samples were tensile-tested and metallographically analyzed. No bond fractures were incurred during tensile testing. Loads ranging from 740 to 1140 pounds resulted in solder failures and in separation of the ceramic body.

A third group also utilizing the integral pedestal is presently being fabricated and is scheduled for environmental storage life at 650°C, including thermal cycling.

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FIGURE 15. DIFFUSION-BONDED COPPER PEDESTAL BeO INSULATOR CONCEPT.

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VII. FLEXIBLE CONNECTOR

Work continued in determining the capabilities of the flexible connector to prevent stresses from being generated because of the difference in thermal expansion between the cold and the hot heat exchanger plates. Special test samples are being evaluated in storage life and environmental testing at design and accelerated temperatures. A detailed description of the test program and evaluation procedures were described in the Phase IIA Final Report.

During the reporting period, no significant changes were observed in the physical characteristics and the metallurgical properties of the flexible connector samples. Tensile strengths and the 25-mil constant deflection force after 40 cycles, continue to remain well within the acceptable limits described in previous reports. All bonds are metallurgically sound.

Tables V and VI show current summaries of metallographic, tensile, and constant deflection data accumulated on samples having the indicated life and environmental histories.

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TABLE V. FLEXIBLE CONNECTOR TEST & EVALUATION DATA

Group No.	TESTING				POST TEST EVALUATION													
	Hours Storage Life w 270°C	Environmental Tests		Hours Storage Life a 270°C	METALLOGRAPHIC			TENSILE				CONSTANT DEFLECTION						
		Thermal Cycling 270°C Max	Shock & Vibration Incl'd Random Vib		% Voids in Reaction Zone		REMARKS	Before Constant Defl.		After Constant Defl.		'X" Direction			'Z" Direction			
					Cu Slug Side	Stain. Steel Side		Fracture		Fracture		Defl. Force (Lb) ⁽²⁾			Defl. Force (Lb) ⁽¹⁾			
								Strength PSI	Location	Strength PSI	Location	1 ~	10 ~	40 ~	1 ~	10 ~	40 ~	
2	1000		-		6-15	NIL	All Bonds look metallurgically & structurally sound	17,800	At Random in Web	22,800 21,200	At Random in Web	2 2	7 0	8 7	17 5	18 4	17 2	
3	3000			-	9-15	NIL	Same as above	18,200	At Random in Web	21,800 20,400	At Random in Web	4 5	6 9	8 5	22 0	22 0	22 5	
4	6000	-	-		14-22	NIL	Same as above	21,400	At Random in Web	20,600 19,200	At Random in Web	6 3	8 4	10 0	27 5	23 0	23 0	
6	1000	10		-	16	NIL	Same as above	17,900	At Random in Web	19,700 19,700	At Random in Web	9 8	11 5	12 7	34 0	30 5	30 0	
7	1000	10		2000			Not complete											
37	-	10	-		7-15	NIL	Same as above	22,200	At Random in Web	19,500 22,200	At Random in Web	4 2	5 9	6 9	25 9	22 9	20 5	
38		10	-	1000	7-23	NIL	Same as above	19 7	At Random in Web	22,000 22,000	At Random in Web	7 0	9 0	10 2	28 5	25 5	25 0	

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TABLE VI - FLEXIBLE CONNECTOR TEST & EVALUATION DATA

Group No.	TESTING				POST TEST EVALUATION													
	Hours Storage Life in 340°C	Environmental Tests		Hours Storage Life in 340°C	METALLOGRAPHIC			TENSILE				CONSTANT DEFLECTION						
		Thermal Cycling 340°C Max	Shock & Vibration Incl'd Random Vib		% Voids in Reaction Zone		REMARKS	Before Constant Defl.		After Constant Defl.		X" Direction			"Z" Direction			
					Cu Slug Side	Stain. Steel Side		Fracture		Fracture		Defl. Force (Lb) ^a			Defl. Force (Lb)			
								Strength PSI	Location	Strength PSI	Location	1 ~	10 ~	40 ~	1 ~	10 ~	40 ~	
11	-	-	-	-	10-12	NIL	All Bonds and Webs look Metallurgically & Structurally Sound	18,600	At Random in Web	16,900 & 17,700	At Random in Web	7.4	10.8	11.7	39	50.5	53	
44	100	-	-	-	15	NIL	Same as above	14,900	At Random in Web	19,600 & 20,000	At Random in Web	5.7	7.9	9.1	34	46	47.5	
45	576	-	-	-	8-12	NIL	Same as above	16,900	At Random in Web	17,600 & 16,100	At Random in Web	5.2	7.5	8.6	26.5	30.5	31.0	
46	900	-	-	-	4-13	NIL	Same as above	16,000	At Random in Web	20,300 & 22,500	At Random in Web	7.0	10.2	13.0	31.5	31.0	30.0	
67	-	20	-	-	4-18	NIL	Same as above	18,100	At Random in Web	19,300 & 21,200	At Random in Web	7.5	10.3	11.3	28.5	25.5	25.0	
68	-	20	-	139			Not completed	13,550	At Random in Web	14,820 13,920	At Random in Web	6.9	7.6	8.3	18.5	17.5	16.5	
50	100	20	-	-	4-17	NIL	Same as above	20,800	At Random in Web	20,500 & 21,200	At Random in Web	8.0 lb	9.5	10.5	35	32.2	32.0	
69	-	20	-	619	12-17	NIL	Same as above	11,260	At Random in Web	12,340 14,050	At Random in Web	5.3	6.2	6.9	22.0	21.0	20.0	
53	100	20	40 G ^s	-	19-29	NIL	Fractures in the Webs were observed both before and after constant deflection	10,500	In Web near Brace Fillet on Crown	20,000 & 20,000	At Random in Web	12.2	13.0	13.0	61	58.6	57.0	
53	100	20	40 G ^s	-	-	-	The % of Fractured Webs for a given side ranged from 0 - 50% of total	22,000	At Random in Web	21,100	At Random in Web							
70	-	20	-	900	2-10	NIL	Same as above	15,420	At Random in Web	13,550 15,950	At Random in Web	8.9	10.4	11.7	25.0	23.0	21.3	
52	100	20	-	600			Not completed	13,100	At Random in Web	13,320 14,800	At Random in Web	4.6	5.7	6.8	23.0	22.5	22.5	
51	100	20	-	900	15-17	NIL	Same as above	13,570	At Random in Web	16,100 15,200	At Random in Web	7.1	8.1	9.2	22.0	20.5	20.0	
54	100	20	40 G ^s	600			Not completed	13,650	At Random in Web	9,370 14,170	At Random in Web	8.1	8.7	9.1	23	22.5	20.8	
55	100		40 G ^s	900	8-23	NIL	One broken Web near Fillet, otherwise bonds look good	12,490	At Random in Web	-	-	-	-	-	-	-	-	
55	100	20	40 G ^s	900			Not completed	13,850	At Random in Web	12,570 15,550	At Random in Web	8.8	9.5	9.5	21.5	20.0	20.0	
47	100	10	-	-	12-20	NIL	All Bonds & Webs look metallurgically & structurally sound	20,300	At Random in Web	19,100 17,200	At Random in Web	11.1	12.8	13.7	41	39	39	
48	100	10	-	600	18-20	NIL	Same as above	18,600	At Random in Web	18,600 18,300	At Random in Web	8.7	11	12.4	40	36	34.8	

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VIII. HOT AND COLD HEAT EXCHANGER PLATES

During this reporting period, the steady-state life testing of materials and bonds relating to the hot heat exchanger plate was continued.

The test specimens consist of pieces of Hastelloy B and Hastelloy X which were machined suitable for tensile testing. Unwelded pieces were used as controls; electron beam and tungsten inert gas-welded specimens represented the joining techniques proposed for the fabrication of the heat exchanger plates in the Developmental Module Design.

Brazed specimens which simulated the plate-to-stack joint and were suitable for tensile test were also made. These samples consisted of cylindrical pieces of Hastelloy bonded at either end to larger pieces of nickel suitable for tensile testing. The brazed materials used were:

- 1) a nickel-gold eutectoid alloy
- 2) a nickel - titanium eutectic
- 3) a nickel-silicon-boron alloy.

The life test series included similar structures evaluating the cold heat exchanger plates, i.e. 316L stainless steel and 200 nickel tensile specimens together with brazed pieces in which both nickel-gold eutectoid alloy and a germanium-copper alloy were evaluated. This phase of the program was completed and reported in the previous quarterly report.

No downtimes occurred during this reporting period and the scheduled downtimes of 8000 and 1600 hours have been extended to 20,000 and 45,000 hours, respectively, in order to obtain performance data after greatly extended periods of isothermal exposure.

The data previously collected and evaluated up to 4000 hours supported the selection of Hastelloy B and 316L stainless steel as the hot and cold heat exchanger plate materials, respectively.

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