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MASTER

CATEGORIES
C92A, C92B, C93B
M3679 (51st Ed.)
AEC RESEARCH AND
DEVELOPMENT REPORT

QUARTERLY PROGRESS REPORT No.2

for

COMPACT THERMOELECTRIC CONVERTER DEVELOPMENT PROGRAM - PHASE IIB (U)

RCA
**DIRECT ENERGY
CONVERSION**

CONTRACT AT(30-1)-3582

For the Period

April 1, 1967 to June 30, 1967

TR	U	4-12-82	Date
DECLASSIFICATION			Class: U

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Prepared for:
Sandia Area Operations Office
U.S. Atomic Energy Commission

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

CONTRACT AT(30-1)-3582

For the Period

April 1, 1967 to June 31, 1967

Prepared by:

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U.S. Atomic Energy Commission

Date Prepared:

Aug. 15, 1967

Classification cancelled (or changed to)
by authority of Dec
by W.C.C. TIC, date SEP 6 - 1973

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TABLE OF CONTENTS

	<u>Page</u>
I. Abstract	1
II. Module Process Development and Fabrication	2
III. Structure Evaluation Program	5
A. Two-Couple Test Structures - Linear Test Structure No. 4	5
B. Thermal Cycling Capability of the Phase IIA Elements - Linear Test Structure No. 7	5
C. Long Term Life Capability of the Phase IIA Elements	11
1. All-Brazed Structure - Partial Test Structure No. 13	11
2. All-Ductile Hot Stack Design - Partial Test Structure No. 14	11
D. Phase IIB Structures	11
1. Linear Test Structure No. 10	11
2. Linear Test Structure No. 11	16
3. Linear Test Structure No. 12	16
4. Linear Test Structure No. 13	16
5. Linear Test Structure No. 14	16
6. Linear Test Structure No. 15	19
E. Evaluation Test Conditions	19
IV. Thermoelement	20
A. Establish Bond Facilities	20
B. Process Development & Design Studies	20
1. Braze Penetration	20

TABLE OF CONTENTS (Cont.)

	<u>Page</u>
2. Voids in "P" SiGe-JTA Bonds	20
3. Alternate Brazes	24
a. Titanium-Zirconium	25
b. Titanium	25
4. Acceleration Factor	25
5. Effects of Processing on Element Performance	26
C. Materials and Bonds Evaluation	26
V. Hot Stack	29
A. Hot Stack Design Evaluation	29
1. Tungsten CM-50 Brazed to Nickel	29
2. Alternate Tungsten-Nickel Systems With Diffusion Inhibitors	29
B. Alternate Diffusion Barriers	30
1. Refractories and Noble Metals	30
2. Intermetallic Compounds	31
3. Ceramics	31
4. Graphite	31
VI. Cold Stack	32
VII. Hot and Cold Heat-Exchanger Plates	35
VIII. BeO Ceramics	40
IX. Flexible Connector	41
X. Summary	43

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LIST OF ILLUSTRATIONS

<u>Figure No.</u>	<u>Title</u>	<u>Page</u>
1	Linear Test Structure No. 14	3
2	Method of Joining Linear Module Unit Assemblies Using "Omega" Electrical Interconnects	4
3	Module LTS-4 Couple No. 1, Voltage, Resistance, Power, and Temperature vs. Time	7
4	Module LTS-4 Couple No. 2, Voltage, Resistance, Power, and Temperature vs. Time	8
5	Module LTS-7, Voltage, Resistance, Power, and Temperature vs. Time	9
6	Linear Test Structure of the LTS-7	10
7	Typical Partial Test Structure	12
8	PTS-13, Voltage, Resistance, Power, and Temperature vs. Time	13
9	PTS-14, Voltage, Resistance, Power, and Temperature vs. Time	14
10	Module LTS-12, Voltage, Resistance, Power, and Temperature vs. Time	17
11	Module LTS-13, Voltage, Resistance, Power, and Temperature vs. Time	18
12	Diffusion Bonded Slotted-Copper-BeO Subassembly	33

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LIST OF TABLES

<u>Table No.</u>	<u>Title</u>	<u>Page</u>
I	Test Structure Evaluation Summary	6
II	Phase IIB Test Structures	15
III	Summary of Average Resistance Values as a Function of Time at 800°C Room Temperature-Resistance in Milliohms ...	21
IV	Summary of Contact Resistance on Alternate Materials	23
V	Summary of Room Temperature Resistance on Tests to Deter- mine Acceleration Factor	27
VI	Summary of Average Resistance Values as a Function of Time at 900°C Room Temperature-Resistance in Milliohms ...	28
VII	Status of Life Tests	36
VIII	Heat-Exchanger Plate Development Thermal Storage Testing in Vacuum-Room Temperature Tensile Data	37
IX	Heat Exchanger Plate Development Thermal Storage Testing in Vacuum	39
X	Flexible Relief Connector Test & Evaluation Data	42

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I. ABSTRACT

Six additional test structures were assembled during this reporting period, with minor variations in components. These test devices have been placed on operational test in vacuum. One of these test structures was not mounted for test but is being examined metallurgically because of indications of higher than normal element-resistance.

Tests continue on thermoelement assemblies to evaluate braze penetration and void presence in some of the bonds. Additional work is being done on hot stack components to reduce interdiffusion of metals by means of metallurgical barriers.

Tests continue on heat-exchanger plate and flexible connector components for confirmation purposes. These components have essentially been firmed up but component life tests continue.

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II. MODULE PROCESS DEVELOPMENT AND FABRICATION

The refinement and improvement of the developmental fixtures and tooling which are required to fabricate and assemble the linear test structures was continued. Six such structures were fabricated using these fixtures and tooling during this reporting period. One of these structures, LTS-14, is shown in Figure 1. The establishment of these developmental assembly fixtures and tooling has enabled nonengineering personnel to fabricate test structures, maintaining the quality of product at minimal cost consistent with other program considerations.

During this quarter, design work was initiated on the developmental fixtures and tooling required for the fabrication and assembly of a full module. Review of the Development Module Design, established at the end of Phase II-A, revealed an assembly problem in the design of the triple-width, cold heat-exchanger plates which is common to all three rows of thermocouples, viz., to assure proper bonding of the cold stack components of the middle row during the final assembly operation. The components of the middle row could not be visually inspected in the final assembly fixture before or after bonding. Moreover, a fixture designed to accommodate the middle row would be relatively complex and cumbersome, and it is doubtful that its use would assure proper bonding. Therefore a linear module unit-assembly concept was established to overcome these difficulties.

The linear module-unit-assembly concept now under study is basically an extended linear test structure with 14-over-14 thermocouples in a row. A full module of the present 84-couple design would be made by joining three linear module-unit assemblies at the ends by common manifolds. The series-parallel circuitry capability of the present design would be maintained by joining the copper electrical connectors of each couple with small copper "omega" electrical interconnects between the rows in a final assembly braze operation; see Figure 2. Each linear module-unit-assembly can be given 100 percent inspection prior to the final module assembly braze. Compared to the module concept employing the triple-width cold plates, the module design using the linear module-unit assembly is superior, being readily assembled by using a simple fixture and permitting the final braze to be made in a less critical area. The "omega" electrical interconnect is required only in the event of an electrical path failure; high joint mechanical strength is not required. Therefore, even partial contact at the bond interfaces would be acceptable.

In addition to the advantages of 100 percent inspection and the corresponding reduction in potential rejection rate of modules, the linear module-unit-assembly concept allows: 1) solid tubes to be used for the cold heat-exchanger in lieu of the welded tubes for the triple-width requirement, and 2) the use of multiples of units to more closely meet current and voltage requirements. As of this writing, most of the preliminary design work of the linear module-unit-assembly concept has been completed.

CONFIDENTIAL

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3

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FIGURE 1. LINEAR TEST STRUCTURE NO. 14

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FIG. 2 - METHOD OF JOINING LINEAR MODULE UNIT ASSEMBLIES USING
"OMEGA" ELECTRICAL INTERCONNECTS.

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III. STRUCTURE EVALUATION PROGRAM

Table I summarizes the structures which were evaluated under simulated operational and environmental conditions during this reporting period. A discussion on each structure follows:

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

The early linear test structures were designed to accommodate representative available parts which enabled the evaluation of the Compact Converter components to proceed. Round heat-exchangers were employed.

LTS-4 is being 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-insulator hot stack, 2) two pair of rectangular, Phase I elements which have 30-mil graphite and tungsten shoes, copper pedestals, and tungsten compensators that are Nicro-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. The performance curves in Figures 3 and 4 show that operation of LTS-4 continues satisfactorily after more than 3,000 hours at an average hot-shoe temperature of 700°C.

B. Thermal Cycling Capability of the Phase IIA Elements - Linear Test Structure No. 7

LTS-7 was removed from test for metallurgical examination after successfully completing its program of 134 cycles and 2,634 hours of steady-state operation at a hot-shoe temperature of 800°C as shown in Figure 5. Originally, LTS-7 was to be subjected only to a continuous thermal cycle program; the purpose of which was to determine the capability of the Phase IIA elements to withstand thermal stresses without the effects of time-at-temperature by testing to destruction. LTS-7 was a six-couple structure having a 1 x 3-over-1 x 3 array as shown in Figure 6. After 118 thermal cycles, LTS showed no indication of bond degradation based on a review of its thermal and electrical performance. It, therefore, appeared that thermal cycling to a maximum hot-shoe temperature of 800°C would not affect element bonds which have not been subjected to extended time-at-temperature. Having exceeded expectations, LTS-7 was placed on a test program of steady-state operation with one thermal cycle per week to continue to determine the elements' capabilities.

The metallurgical examination of LTS-7 disclosed the elements' bonds to be unchanged from those in as-bonded conditions. This finding coincides with the normal electrical performance before LTS-7 was removed from test. The testing of LTS-7 has clearly demonstrated the capability of the Phase IIA element bonds to withstand severe thermal stressing without the effects of extended time-at-temperature.

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TABLE I
TEST STRUCTURE EVALUATION SUMMARY

<u>Test Structure Number*</u>	<u>Number of Couples</u>	<u>Type of SiGe Couple</u>	<u>Hot Shoe Temperature</u>	<u>Status</u>
PTS-13	12	Rectangular Phase II	800 °C	Steady-State Test Operation - Satisfactory After 3300 Hours
PTS-14	12	Rectangular Phase II	800 °C	Steady-State Test - Removed After 2550 Hours for Metallurgical Evaluation
LTS-4	2	Rectangular Phase I	700 °C	Steady-State Test Operation - Satisfactory After 3000 Hours
LTS-7	6	Rectangular Phase II	800 °C	Steady-State - Thermal Cycle Test - Removed for Metallurgical Evaluation After 2634 Hours and 134 Cycles
LTS-10	6	Rectangular Phase II	800 °C	Dismounted for Metal- lurgical Evaluation
LTS-11	6	Rectangular Phase II	800 °C	Steady-State Test Operation Initiated
LTS-12	6	Rectangular Phase II	800 °C	Steady-State Test Operation - Satisfactory After 700 Hours
LTS-13	6	Rectangular Phase II	800 °C	Steady-State Test Operation - Satisfactory After 48 Hours
LTS-14	6	Rectangular Phase II	800 °C	Being Mounted for Steady-State Test Operation
LTS-15	6	Rectangular Phase II	800 °C	Being Fabricated - Steady-State Test Schedule

* PTS - Partial Test Structure
LTS - Linear Test Structure

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7

FIGURE 3. MODULE LTS-4 COUPLE NO. 1, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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

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6

FIGURE 5. MODULE LTS-7, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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FIGURE 6. LINEAR TEST STRUCTURE OF THE LTS-7

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C. Long Term Life Capability of the Phase IIA Elements

1. All-Brazed Structure - Partial Test Structure No. 13

PTS-13 is a 12-couple structure similar to that shown in Figure 7. The stacks are of the same design as that used in LTS-7, and include the Phase IIA elements with 8826 graphite in the "N"-element and pre-fired 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 continues to perform well after more than 3300 hours of operation as shown in Figure 8.

2. All-Ductile Hot Stack Design - Partial Test Structure No. 14

PTS-14 was established primarily to evaluate the performance of Phase IIA elements in an all-ductile hot stack under steady-state conditions at a maximum hot-shoe temperature of 800°C, and to compare the results with those of the PTS-13 performance, which is an all-brazed assembly discussed previously. As reported earlier, PTS-14 operated satisfactorily for more than 2000 hours at which time a severe thermal cycle occurred due to a heater-power failure. Subsequent operation revealed a lower total power output as well as a higher average hot heat-exchanger plate temperature as shown in Figure 9.

Visual examination of the structure revealed cracking in the hot stacks at the lower gold pedestal-to-BeO insulator joint; the stacks were fabricated by diffusion-bonding elements to the hot heat-exchanger plate through a gold-BeO-gold hot stack in a one-step operation. Metallurgical analysis disclosed that nickel from the Hastelloy "B" hot heat-exchanger diffused into the gold, rendering it relatively hard. Apparently, because of the resultant loss of ductility, the hot stacks cracked when the structure was cycled down. A diffusion barrier system which would have prevented the nickel diffusion was not used because it had not been developed when PTS-14 was fabricated. All bonds between the silicon-germanium and graphite showed no change from initial conditions. Similar to the findings in earlier structures, cracking was observed in the BeO insulator in the cold stacks. Refer to the cold stack section of this report for further discussion.

D. Phase IIB Structures

Table II describes the construction details of the structures fabricated to date in Phase IIB; other aspects of these structures are as follows:

1. Linear Test Structure No. 10

LTS-10 was removed from the test station before its test program was initiated; cold resistance measurements indicated relatively high

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FIGURE 7. TYPICAL PARTIAL TEST STRUCTURE

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13

FIGURE 8. PTS-13, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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17

FIGURE 9. PTS-14 VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME

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15

TABLE II
PHASE IIB TEST STRUCTURES

Test Structure	No. of Couples	"N" Element	"p" Element	Hot Stack		Cold Stack*	
				Diffusion Barrier	Other Parts	Cold End Pedestal	Heat Distributor Plate
LTS-10	6	W-NiTi W Vapor- Plated 8826	W-TiZr-JTA	W-Ni	Au-BeO-Au	Cu	W-Cu
LTS-11	6	W-NiTi W Vapor- Plated 8826	W-TiZr-JTA	W-Au	Au-BeO-Au	Au	W-Cu
LTS-12	6	W-NiTi-8826	W-TiZr-JTA	W-Au	Au-BeO-Au	Au	W-Cu
LTS-13	6	W-NiTi-8826	W-NiTi-8826 -Al-SiGe	W-Au	Au-BeO-Au	Au	W-Cu
LTS-14	6	W-NiTi-8826	W-NiTi-8826 -Al-SiGe	W-Ni	Au-BeO-Au	Au	W-Cu
LTS-15	6	W-NiTi-8826	W-NiTi-8826 -Al-SiGe	W-Au	Au-BeO-Au	Au	W-Cu

* All Cold Stacks Have W Compensators, Cu Electrical Connectors,
and Cu Flexible Connectors.

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readings in two of the elements. Visual examination disclosed that the tungsten vapor plate had separated from the 8826 graphite at the hot ends of these two elements. Metallurgical and storage life test evaluations are in progress to determine the cause of the separations.

2. Linear Test Structure No. 11

LTS-11, with the same "N"-elements as LTS-10, similarly exhibited partial separations between the tungsten vapor plate and the 8826 graphite at the hot ends of two elements. This defect occurred during the initial operation of the structure at a hot shoe temperature of 800°C. Because electrical continuity has been maintained, the test structure is being 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.

3. 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 previously, the tungsten layer on the graphite was considered in the "N"-elements to serve as a barrier to nickel-titanium braze penetration into the graphite, and thereby 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 judgment supported the discontinuance of the use of a tungsten layer in elements for future structures until further evaluation is completed.

As shown in Figure 10, LTS-12 is performing well after 700 hours of steady-state operation at a maximum hot shoe temperature of 800°C.

4. 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 previous discussions). Currently, LTS-13 has accrued 48 hours of operation as shown in Figure 11.

5. 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 barrier system of tungsten CM-50 brazed to nickel; see Figure 1. LTS-14 is presently being mounted in the test station.

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FIGURE 10. MODULE LTS-12, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME.

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18

FIGURE 11. MODULE LTS-13, VOLTAGE, RESISTANCE, POWER, AND TEMPERATURE VS. TIME.

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6. Linear Test Structure No. 15

LTS-15 was established primarily to evaluate the performance of the alternate "P"-element under a combination steady-state-thermal cycle test program at a maximum hot shoe temperature of 800°C, LTS-15 is of the same design as LTS-13 and is presently in fabrication.

E. 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 are conducted with electrical heaters. LTS-4, which is constructed on a tubular hot heat-exchanger, uses a graphite heater shaped to provide the desired temperature profile along the tube. The later LTS structures and the 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. 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, it has been possible to insert the thermocouple into small holes drilled in the structure for this purpose. The thermocouples on the inside of the cold heat-exchanger plate are 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 are measured through copper wires.

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IV. THERMOELEMENT

A. Establish Bond Facilities

During Phase IIA, the element fabrication capabilities were expanded by establishing a second facility to provide thermoelements to meet the increased requirements of the program. The thermoelements fabricated in this second facility were placed on isothermal storage and operational life testing. In addition to establishing this second facility, investigations were conducted of equipment with greater capacity but which used slightly different bonding techniques. The products from these latter facilities met the acceptance criteria for contact resistance in the silicon-germanium-graphite bond. Samples from these facilities were also placed on steady-state isothermal storage test at 800°C. Thermoelements from Phase I were used for control purposes with these tests.

The evaluation of these tests is based on a study of electrical resistance as a function of time-at-temperature; the data is presented in Table III. There has been no unusual change in any contact resistance, but the increase in total resistance is attributed to the aging characteristics of silicon-germanium.

B. Process Development & Design Studies

1. Braze Penetration

Isothermal storage life test evaluation continued of the JTO-type graphite - "N" silicon-germanium element system. This system was originally being evaluated as a possible improvement over the "N"-elements using 950S graphite where braze penetration had been encountered which led to bonds of high contact resistance and low strength. The use of JTO, a grade similar to JTA except without boron, did reduce the braze penetration, but it exhibited a sharp rise of contact resistance with time-at-temperature. The system was deemed unacceptable after 150 hours at 800°C in vacuum, but it is being continued on test to determine the extent of resistance increase before mechanical failure occurs. The summary of test results is shown in Table IV.

Additional work is underway to resolve braze penetration problems. See section on Alternate Brazes, B.3.

2. Voids in "P" SiGe-JTA Bonds

Evaluation of a 950S-"P" silicon-germanium element system continued. This component was originally considered as a replacement for the "P" SiGe-JTA element which possesses a variable void level in the bond area. The use of 950S graphite with the "P" silicon-germanium

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TABLE III

SUMMARY OF AVERAGE RESISTANCE VALUES AS A FUNCTION OF TIME AT 800°C
ROOM TEMPERATURE-RESISTANCE IN MILLIOHMS

Sample Description	Test No.	Type	Hours at 800	R _t	R _g	R ₁	R ₂	R ₃	SiGe*	Predicted for SiGe	Avg.% Voids in SiGe Graphite Interface
Phase I Control	1	N	0**	2.35	2.23	0.03	0.16	0.19	1.97		1
			150	2.55	2.43	0.03	0.17	0.20	2.15		
			500	2.70	2.60	0.04	0.15	0.19	2.32		
			1000	2.70	2.62	0.02	0.21	0.23	2.24		1
			1500	2.65	2.51	0.03	0.18	0.21	2.23	2.36	
			3000	2.65	2.50	0.03	0.21	0.24	2.17		1
			5700	2.54	2.45	0.03	0.14	0.17	2.20	2.41	
			7000	2.61	2.53	0.02	0.13	0.15	2.27		
Phase II	2	N	0**	2.06	2.00	0.03	0.13	0.16	1.68		1
			150	2.26	2.21	0.04	0.13	0.12	2.02		
			500	2.36	2.30	0.03	0.11	0.14	2.08		
			1000	2.36	2.29	0.03	0.12	0.14	2.08		1
			1500	2.31	2.27	0.04	0.13	0.14	2.03	2.02	
			3000	2.21	2.20	0.04	0.10	0.14	1.93		
			5700	2.40	2.32	0.03	0.11	0.14	2.12	2.15	
			7000	2.47	2.42	0.04	0.11	0.15	2.20		
Phase I Control	3	P	0**	2.55	2.50	0.03	0.16	0.19	2.17		20.0
			150	2.60	2.54	0.03	0.08	0.11	2.38		
			500	2.65	2.62	0.03	0.10	0.13	2.39		
			1000	2.67	2.62	0.04	0.11	0.15	2.37		24.5
			1500	2.69	2.64	0.03	0.14	0.17	2.35	2.43	
			3000	2.98	2.90	0.03	0.14	0.17	2.64		10.6
			5700	3.00	2.93	0.03	0.14	0.17	2.70	2.65	
			7000	3.20	3.16	0.03	0.22	0.24	2.77		
Phase II	4	P	0**	2.51	2.49	0.02	0.15	0.17	2.21		1
			150	2.60	2.52	0.02	0.13	0.15	2.30		
			500	2.65	2.58	0.02	0.13	0.15	2.35		
			1000	2.67	2.60	0.02	0.15	0.17	2.33		1.2
			1500	2.67	2.59	0.03	0.16	0.19	2.29	2.48	
			3000	2.70	2.61	0.03	0.16	0.19	2.52		1
			5700	3.10	3.01	0.04	0.11	0.15	2.80	2.70	
			7000	3.32	3.24	0.04	0.12	0.17	3.00		

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21

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TABLE III (Cont.)

Sample Description	Test No.	Type	Hours at 800	R _t	R _g	R ₁	R ₂	R ₃	SiGe *	Predicted for SiGe	Avg. % Voids in SiGe Graphite Interface
Backup Facility #1	5	P	0**	2.57	2.51	0.03	0.14	0.17	2.23		10.1
			150	2.62	2.58	0.03	0.08	0.11	2.40		
			500	2.67	2.57	0.02	0.09	0.11	2.45		
			1000	2.67	2.59	0.03	0.12	0.15	2.35		7.2
			1500	2.72	2.64	0.03	0.14	0.17	2.38	2.50	
			3000	2.97	2.84	0.02	0.15	0.17	2.63		9.1
			5700	3.12	3.01	0.04	0.11	0.15	2.82	2.72	
			7000	3.32	3.24	0.04	0.14	0.18	2.96		
Backup Facility #2	6	P	0**	2.55	2.49	0.03	0.16	0.19	2.17		1
			150	2.50	2.36	0.03	0.10	0.13	2.24		
			500	2.50	2.35	0.03	0.14	0.17	2.16		
			1000	2.50	2.37	0.02	0.15	0.17	2.16		2.1
			1500	2.50	2.40	0.03	0.14	0.17	2.16	2.43	
			3000	2.64	2.52	0.04	0.15	0.19	2.26		1
			5700	3.02	2.94	0.03	0.12	0.15	2.72	2.65	
			7000	3.20	3.16	0.03	0.12	0.15	2.92		

* Calculated from total resistance less contact resistances

** After 16 hours at 900°C

TABLE IV

SUMMARY OF CONTACT RESISTANCE ON ALTERNATE MATERIALS

<u>Sample Description</u>	<u>Test No.</u>	<u>Element Type</u>	<u>Hours</u>	<u>R_t</u>	<u>R_g</u>	<u>R₁</u>	<u>R₂</u>	<u>R₃</u>	<u>SiGe*</u>	<u>Predicted for SiGe</u>	<u>Average Percent Void</u>
JTO Graphite	1	N	0**	2.26	2.20	0.03	0.12	0.15	1.96		26
			150	2.46	2.41	0.03	0.32	0.35	1.76		
			500	2.61	2.55	0.03	0.40	0.43	1.75		
			1000	2.61	2.54	0.04	0.30	0.34	1.93		
			1500	2.56	2.48	0.04	0.35	0.39	1.78	2.35	
			3000	3.06	3.00	0.03	0.43	0.46	2.14		39
			5700	3.39	3.19	0.07	0.37	0.45	2.39	2.41	
			7000	3.30	3.27	0.04	0.31	0.35	2.65		
950S Graphite	2	P	0**	3.65	3.57	0.10	0.53	0.64	2.37		21
			150	4.75	4.64	0.12	0.74	0.86	3.03		
			500	5.22	5.15	0.11	0.81	0.92	3.41		
			1000	5.25	5.18	0.14	0.80	0.92	3.41		
			1500	5.15	5.02	0.14	0.80	0.94	3.27	2.66	
			3000	6.92	6.74	0.14	0.87	1.01	4.92		21
			5700	4.28	4.02	0.10	0.74	0.84	2.60	2.71	
			7000	5.91	5.83	.13	1.10	1.26	3.63		

* Calculated from total resistance less control resistance.

** After 16 hours at 900°C.

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did result in low void levels, but a significantly higher contact resistance was realized. As in the JTO-"N" SiGe system, the contact resistance exceeded the maximum design limits after 150 hours at 800°C in vacuum, but the test is continuing to determine the extent of resistance increase before mechanical failure occurs. The summary of test results is also shown in Table IV.

An alternate approach to obtaining low-void, low-contact resistance element bonds is under investigation with the use of an 8826-type graphite. High contact resistance is a characteristic of the bond between 8826-type graphite and "P"-type silicon-germanium. This result has been attributed to the formation of a high-resistance reaction product during the bonding operation. The bond, however, does have the advantage of a low-void area in the silicon-germanium graphite bond and a high mechanical strength. Experience with the type of graphite in the "N"-type thermoelement has proven it to be reliable and highly reproducible. Early investigations revealed that the use of this type of graphite in the "P"-thermoelement, with a low resistance dopant, reduced the contact resistance without appreciably affecting the thermoelectric properties of the bulk silicon-germanium.

Good results have been achieved with the use of aluminum shims. The "as-bonded" contact resistance in the silicon-germanium-graphite interface in a thermoelement in the coupon configuration has averaged 0.12 milliohm compared to 0.60 milliohm in samples without the aluminum and 0.17 milliohm in samples using JTA graphite. Data on samples after 8000 hours at 900°C in isothermal storage have exhibited an increase in resistance less than the standard "P"-element after a comparable period. Evaluation of the mechanical properties in the "as-bonded" and "acceptance-tested" stages has shown no difference in the strength of the bond compared with the "N"-or JTA "P"-type thermoelement. Samples have been prepared for evaluations to determine the effects of thermal cycling and fabrication steps required in the assembly of a module. Isothermal storage tests at 900°C in vacuum are in progress. Those elements which have been exposed to the thermal cycling and simulated processing involved in the fabrication of a module have exhibited no changes in electrical, mechanical, or metallurgical characteristics.

Additional work is underway to reduce the "P" SiGe-JTA bond void level; see section on Alternate Brazes, B.3.

3. Alternate Brazes

During Phase IIA, investigations of alternate brazes for the tungsten-to-graphite bond were initiated. One objective of this work was to achieve an improvement over the nickel-titanium braze and to lessen the difficulties encountered with it which have been reported previously. A second objective was to reduce the void level realized in the "P" SiGe-JTA bonds.

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Work has been directed to those braze systems which melt above the bonding temperature for the silicon-germanium-graphite bond. This effort necessitates a two-step element assembly procedure involving the pre-bonding of the tungsten shoe and graphite barrier as a subassembly prior to bonding the thermoelement. As reported previously, this procedure produces bonds with lower void areas in the "P"-type silicon-germanium-to-JTA graphite interface. A second advantage of the two-step assembly approach is that the subassemblies of tungsten and graphite shoes can be inspected to detect poor bonds or braze penetration prior to their use in the thermoelement. Discussions of the braze systems investigated during this reporting period, follow:

a. Titanium-Zirconium

One of the major advantages of this system is its high melting temperature, 1630°C. Subassemblies which consisted of the tungsten shoe and the graphite barrier are brazed initially and inspected visually for the surface characteristics and physical alignment. The mechanical integrity is determined by tensile testing a representative sample. Metallographic examination is also performed on a representative sample.

Thermoelements using the titanium-zirconium braze system with prefired JTA graphite, tungsten-coated JTA, or tungsten-coated 8826 and "P"-type silicon-germanium were subjected to metallographic and tensile examination. The initial evaluations indicated that all three systems are acceptable for further consideration. Samples are being prepared for isothermal storage, operational, and thermal cycling testing.

b. Titanium

The investigations of the use of titanium as a braze between the tungsten shoe and both the 8826 and JTA graphite with or without tungsten coating have revealed a high void, low strength bond. Work on this system has been shifted from a brazing operation to diffusion bonds between the tungsten shoe and tungsten coated 8826 and JTA graphites. Samples are being prepared for evaluation in isothermal storage and thermal cycling tests.

4. Acceleration Factor

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

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The 950S graphite samples are being used as control samples in this test and will be compared with similar samples initiated during Phase IIA of the program. The other samples represent the thermoelement as it was specified at the end of Phase IIA. The tungsten shoe is 40 mils thick and the graphite has been prefired prior to bonding; the 8826 has a coating of silicon; the nickel-titanium braze system was used in one-step bonding operation. The acceleration factor is to be determined by a comparison of the electrical resistance and the metallurgical evaluation of the following as a function of time-at-temperature: (1) void levels in the tungsten-graphite bond; (2) void level in the silicon-germanium-graphite bond, and (3) changes in the graphite shoe. As of this report, no acceleration factor can be determined; see Table V.

5. Effects of Processing on Element Performance

A major difference between isothermal life test samples and thermoelements which are used in modules is that the latter are subjected to further processing required for completion of the test structure. This processing involves the temperatures, pressures, and atmosphere required for hydrogen brazing and vacuum diffusion bonding.

A test was initiated to determine the effect on an element performance of the fabrication techniques. The test samples were comprised of "P" and "N" thermoelements in both the Phase IIA and coupon configurations, with and without tungsten shoes, and also with the nickel-titanium and titanium-zirconium braze systems. One half of the samples were subjected to the thermal cycling involved in braze processing; the remaining samples were subjected to diffusion bonding cycling. All the samples were put into isothermal storage life testing at 900°C. After 264 hours of testing, a malfunction in the thermocouple control system permitted an over-ride in temperature up to 1200°C and resulted in a catastrophic failure of the samples.

New samples are being prepared for a rerun of the program.

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 graphite shoes. The samples for these tests are in the coupon configuration, and the controls are of 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 resistance are shown in Table VI. There has been no significant increase in any of the values, and all are within the present design objectives.

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27

TABLE V

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 II B	500		2.80	2.71	0.04	0.14	0.18
	2500		2.71	2.63	0.03	0.18	0.21
		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
	300		2.73	2.62	0.01	0.17	0.18
Phase II B		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
		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 II B	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	2.65	2.47	0.07	0.13	0.19
950S Graphite Phase II A	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	2.32	2.18	0.06	0.12	0.18
	2500		2.30	2.11	0.04	0.13	0.17
		4000	2.51	2.34	0.06	0.14	0.20

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TABLE VI

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		
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		
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		
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		
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		

* Total Resistance R_t less the contact resistances ($2 \times R_3$).

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V. HOT STACK

During this reporting period, work continued in evaluating, optimizing, and improving the hot stack as defined in the Development Module Design specified in Phase IIA. The hot-stack design included a diffusion barrier of tungsten bonded to the Hastelloy "B" hot heat-exchanger plate through a nickel connector plate. The prime method of joining these materials was by brazing a high nickel-content material with CM-50. The remainder of the hot stack, gold-BeO-gold, was diffusion-bonded to the tungsten barrier in subsequent processing. The alternate methods of joining the tungsten-nickel-Hastelloy "B" were a combination of diffusion-bonding and brazing.

A. Hot Stack Design Evaluation

1. Tungsten CM-50 Brazed to Nickel

Isothermal storage life testing of CM-50-brazed, simulated lower hot stacks is continuing. As previously reported, 85 percent Kirkendall voids were observed between the tungsten and nickel after approximately 1600 hours at 900°C. Samples, tested to 1000 hours at 835°C and 865°C, respectively, showed no significant increase in void count from as-bonded conditions. Additional samples have been placed on storage life testing at these two temperatures for longer time periods. These added evaluations will be examined at the end of the next quarter. The samples tested for 2000 hours at 800°C exhibited no change from the as-bonded condition. A sample removed at the next downtime, 3300 hours, showed approximately 20 percent Kirkendall voids. The next scheduled downtime at 800°C is due at 4000 hours.

A test was completed in which tungsten rectangles of a new vendor were compared to the existing tungsten parts. The new tungsten appeared superior in several respects, viz., it was stronger when subjected to the incoming inspection break tests and it had a more desirable microstructure when compared metallographically to the existing product. When brazed into simulated hot stacks, five of the 16 stacks which contained existing tungsten had vertical cracks; no cracks were visible in the sandwiches made with the new product. At the completion of the test, 1000 hours at 900°C with 10 thermal cycles, only 2 stacks of the new vendor had a few small cracks. After 1000 hours at 900°C, Kirkendall void formation in CM-50 braze systems, using the new tungsten, was half as much as that which occurred with the existing product. Additional evaluation effort is planned to determine whether this superiority is due to a minor constituent, some doping agents, or a difference in vendor processing.

2. Alternate Tungsten-Nickel Systems With Diffusion Inhibitors

The limited data gathered to date on the tungsten-nickel CM-50 braze system, indicated that its life potential is limited. Accordingly,

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effort has been expended on modifying the system to improve its life capabilities. As reported previously, the introduction of certain materials into the bond at the tungsten-nickel interface has significantly retarded the inter-diffusion process, thereby reducing the rate of Kirkendall void formation. A pure tungsten-nickel diffusion bond was found to have 85 percent Kirkendall voids after 485 hours at 800°C, whereas the same percentage was noticed in a tungsten-nickel CM-50-brazed sample after 1600 hours at 900°C.

A literature search revealed that rhodium plating and thoriated tungsten might behave in a similar manner to retard diffusion. Accordingly, the following variations have been placed on storage life testing at 800°C and 900°C with and without thermal cycling:

a. Diffusion-Bonded Systems

Rhodium-Plated Tungsten - Nickel Pure

Tungsten - CM-50 Bond Shim - Nickel Pure

Rhodium-Plated Tungsten - CM-50 Bond Shim - Nickel Pure

Thoriated Tungsten (1% and 2%) - CM-50 Bond Shim - Nickel Pure

Rhodium-Plated Thoriated Tungsten (1% and 2%) - CM-50 Bond Shim - Nickel Pure

b. Brazed Systems

Rhodium-Plated Tungsten - CM-50 Braze Shim - Nickel Pure

Thoriated Tungsten (1% and 2%) - CM-50 Braze Shim - Nickel Pure

B. Alternate Diffusion Barriers

During this reporting period, work continued in developing alternate diffusion barriers using refractories and noble metals, intermetallics, ceramics, and a graphite which are discussed as follows:

1. Refractories and Noble Metals

Effort continued in developing the process for achieving the vapor-deposition of tungsten on nickel and Poco Graphite. A thin, vapor-deposited layer of tungsten will be evaluated and compared to the performance of bulk tungsten when used on a nickel stress isolation pad. The use of the vapor-deposited tungsten is also being evaluated on a graphite stress isolation system. Tungsten which is vapor-deposited directly to Hastelloy "B" is being evaluated in a storage life test program.

Work continued in optimizing the processes of bonding Rhenium, Rhodium, and Iridium to both Hastelloy "B" and gold. Emphasis is being concentrated on achieving the desired thickness without cracking the barrier. Based on preliminary data, Rhenium appears to be an effective barrier.

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2. Intermetallic Compounds

During this reporting period, effort continued in the optimization of the Cr_xSi_y coating process. The deposition of Cr_xSi_y by the fused salt-bath process has been most successful among the methods under investigation. Preliminary studies of the effectiveness of Cr_xSi_y as a diffusion barrier showed that Cr_xSi_y -coated Hastelloy "B" reduced gold diffusion into the Hastelloy "B" by 75 percent at 800°C for 1000 hours and 50 percent at 900°C for 1000 hours. A more methodical approach is being taken to develop the desired CrSi_2 coating on Hastelloy "B" substrates because it is anticipated that this coating will be a definite improvement over the Au- Cr_xSi_y barrier - Hastelloy "B" system.

3. Ceramics

Preliminary evaluations were conducted on utilizing Al_2O_3 , BeO, and Fosterite ceramics as barriers bonded to a Hastelloy "B" substrate. Acceptable bonds could not be achieved with the first two because of the large thermal expansion mismatch they exhibited with Hastelloy "B". Horizontal shear cracks formed and the integrity of the system could not be maintained.

An excellent bond was achieved between the Hastelloy "B" and Fosterite, using a nickel-titanium braze; the gold was diffusion-bonded to the ceramic in a subsequent operation. Sandwiches of this system are on storage life testing at 800°C and 900°C isothermally and with thermal cycling. Samples removed after 500 hours at both temperatures and subsequently evaluated metallographically showed no evidence of any deterioration of the bonds or component. Testing is continuing; the next downtime is scheduled for 1000 hours. Although it shows some promise, Fosterite has a relatively high thermal impedance and is not considered a prime material as a diffusion barrier.

4. Graphite

Graphite which possesses desirable thermal properties is also being investigated as a diffusion barrier. It was not possible to obtain integral bonds to the Hastelloy "B" using the graphites presently incorporated in the element assemblies because of their relatively low coefficient of thermal expansion. A Poco graphite of much higher thermal expansion was successfully bonded to the Hastelloy "B". Initial attempts to diffusion-bond gold to siliconized Poco graphite have been unsuccessful. No further effort is planned for this concept. The use of vapor-deposited tungsten on Poco graphite, discussed earlier, will continue to be evaluated.

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VI. COLD STACK

Development effort continued during this reporting period to establish a suitable method of incorporating the BeO insulator into the cold stack design.

The thermal cycle program was completed and the effects of varying the copper heat-distributor plate thickness on the incidence of BeO cracking were evaluated. Although cracking was observed in all test specimens, the samples which had the thinner heat-distributor plates exhibited the least amount of cracking in the BeO insulator.

Following these results, evaluation was made of the performance of this bond system with pedestalling as well as by varying the heat-distributor plate thicknesses. The samples, which were subjected to a thermal cycle program, were fabricated in a two-step operation. The copper heat-distributor plate was initially brazed to the copper pedestal using Nicro material. This subassembly was then brazed to the BeO insulator using Cusil material. This procedure assured proper alignment of the parts which could not be readily achieved if it were accomplished in a one-step Cusil braze operation. Evaluation of five-, 10-, and 15-mil copper parts in a total of 9 combinations revealed that excessive braze runout, observed on all samples, caused large fillets which negated the pedestalling effect and caused horizontal cracks in all samples before the completion of 20 thermal cycles. The degree and the extent of crack formation seemed to verify that the pedestalling would minimize this problem if braze fillets could be kept to a minimum.

The prime design to achieve the above-mentioned goal was considered to be a brazed system using a diffusion-bonded copper pedestal - BeO subassembly. Simulated cold stack structures, including the thermoelement, were fabricated, utilizing this subassembly of diffusion-bonded copper to the BeO. The diffusion-bonding of the subassemblies was accomplished at low pressure and high temperatures; some vertical cracking occurred in the BeO. The process was optimized at a later date to eliminate this vertical cracking. The cold side of the BeO successfully survived thermal cycling with no deleterious effects to the BeO insulator due to the copper pedestal and the heat-distributor parts. Because of the diffusion-bond, no fillets were formed next to the BeO, and the full pedestalling effect was obtained.

However, cracking had been observed on the element side of the BeO. The braze material was not kept to a minimum in the copper pedestal-to-copper electrical connector joint and heavy braze runout occurred. The resultant formation of fillets at the BeO interface negated the pedestalling effect achieved with the diffusion-bond.

A diffusion-bonded slotted-copper-BeO subassembly (Figure 12) is being developed to overcome the aforementioned difficulties. This approach enables the incorporation of the BeO into the design without the problem of braze fillets, while maintaining the pedestal effect. Parts are being fabricated for a storage life test evaluation.

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FIGURE 12. DIFFUSION BONDED SLOTTED-COPPER-BeO SUBASSEMBLY

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Until the improved cold stack design has been developed, the cold stacks of test structures will be fabricated from an interim design using a tungsten heat-distributor copper pedestal combination. The combination is similar to the configuration of the element side of the BeO which has proven to be highly successful in structures fabricated and tested in Phase IIA. This concept, is not desirable, however, because it increases the weight, thermal impedance, and cost of a structure, and will, therefore, only be used until the improved design is developed.

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VII. HOT AND COLD HEAT-EXCHANGER PLATES

During this quarter, the thermal storage life testing of materials and bonds initiated in Phase IIA continued. In Phase IIA, Hastelloy "B" and "X" were selected as hot heat-exchanger-plate materials with the former as the primary system. Tensile specimens were machined from both materials and used in the evaluation of the materials as a function of time-at-temperature in vacuum. These specimens were compared with similarly machined materials which had been electron-beam welded or T.I.G. welded. The electron-beam weld was selected as the primary method of joining machined-plate halves and associated piping; the T.I.G. weld was a backup system. The purpose of these tests was to evaluate the effects of time at elevated temperatures in vacuum on the tensile strength and metallurgical properties of the materials and bonds. Evaluation of plate materials and bonds has been completed up to 4000 hours. Table VII shows the status of life tests for the various systems.

Table VIII shows room temperature tensile data for the plate materials and bonds. These results indicate that no significant change in strength has occurred even after exposure for 4000 hours at the respective design temperatures of 800°C and 340°C for the hot and cold heat-exchanger plates. Variations in results are considered to be due to the normal variation expected within a material as well as to a reflection of the bead size, depth of penetration, and other variables of the welds involved. Metallographic examination of the specimens shows that the weld metal is continuing to develop equi-axed grains, thus breaking up the weld structure in the case of the Hastelloy. The 316L and the 200 nickel specimens show no appreciable change as a result of vacuum exposure at 340°C for 4000 hours.

The Phase IIA Final Report describes in detail the tensile specimen construction of brazed assemblies representing plate-to-stack bonds. In the case of the hot heat-exchanger plate, these samples represent the brazing of a nickel disc, which is the first component in the hot insulator stack, to the Hastelloy "B" or Hastelloy "X" plate materials. The braze materials evaluated are as follows:

1. A nickel-gold eutectoid alloy braze
2. Titanium shim used to form a NiTi braze
3. A high nickel-silicon-boron alloy braze.

Similarly, in the case of the cold heat-exchanger plates, the samples represent the brazing of OFHC copper discs to the plate materials. The braze materials evaluated in conjunction with the cold heat-exchanger plate are as follows:

1. A nickel-gold eutectoid alloy braze
2. A germanium-copper alloy braze.

The metallurgical analysis of brazed structures listed in Table IX are discussed in other sections of the report.

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TABLE VII
STATUS OF LIFE TESTS

<u>Specimen Description</u>	<u>Temp.</u>	<u>8000-Hr. Exposure</u>		<u>16,000-Hr. Exposure</u>	
		<u>Cond. of Test</u>	<u>Hrs. to Date</u>	<u>Cond. of Test</u>	<u>Hrs. to Date</u>
<u>Hastelloy "B"</u>					
As-Rec'd.	800 °C	Steady State	5960	Steady State & 10 Scram Cycles	5960
E-B-Welded	800 °C	Steady State	5631	Steady State & 10 Scram Cycles	5631
T.I.G.-Welded	800 °C	Steady State	5575	Steady State & 10 Scram Cycles	5575
<u>Hastelloy "X"</u>					
As-Rec'd.	800 °C	Steady State	5631	Steady State & 10 Scram Cycles	5631
E-B-Welded	800 °C	Steady State	5575	Steady State & 10 Scram Cycles	5575
T.I.G.-Welded	800 °C	Steady State	5960	Steady State & 10 Scram Cycles	5960
<u>Braze Evaluation</u>					
<u>Hast. "B" to Ni</u>					
Ni-Au Eutectoid	800 °C	Steady State	6007	Steady State & 10 Scram Cycles	6007
Ni Plate & Ti Shim	800 °C	Steady State	5185	Steady State & 10 Scram Cycles	5185
Ni With Si & B Alloy	800 °C	Steady State	5758	Steady State & 10 Scram Cycles	5342
<u>Hastelloy "X" to Ni</u>					
Ni-Au Eutectoid	800 °C	Steady State	5758	Steady State & 10 Scram Cycles	5342
Ni Plate & Ti Shim	800 °C	Steady State	5254	Steady State & 10 Scram Cycles	5254
Ni With Si & B Alloy	800 °C	Steady State	5220	Steady State & 10 Scram Cycles	5220

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TABLE VIII

HEAT-EXCHANGER PLATE DEVELOPMENT THERMAL STORAGE
TESTING IN VACUUM-ROOM TEMPERATURE TENSILE DATA

Specimen Description	Aging Temp.	Aging Time	Room Temperature Tensile Data After Aging												
			Strength Mpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi		
Hastelloy "B"															
As-Rec'd.	800°C	0	133.3	520 ²	117.7	^x 1064	126.5	1999	135.0	^x 4013	138.0	8000		^x 16000	
E.B.-Welded	800°C	0	120.2	520	145.7	^x 1000	136.3	2007	154.0	^x 4052	146.0				
T.I.G.-Welded	800°C	0	101.5	509	134.3	^x 1035	139.0	2014	138.3	^x 4068	122.0				
Hastelloy "X"															
As-Rec'd.	800°C	0	110.5	520 ²	115.0	^x 1000	130.5	2007	124.0	^x 4052	126.0	8000		^x 16000	
E.B.-Welded	800°C	0	107.3	509	121.0	^x 1035	120.0	2014	122.0	^x 4068	127.0 ³				
T.I.G.-Welded	800°C	0	107.5	520 ²	124.0	^x 1064	119.2	1999	120.2	^x 4013	121.0 ³				
316L Stainless Steel															
As-Rec'd.	340°C	0	83.5	309	82.3	^x 433 ²	92.0	1009	83.0	^x 1917	87.2	4005	86.6		
E.B.-Welded	340°C	0	77.9	309	78.1	^x 433 ²	85.0	1009	76.2	^x 1917	83.3	4005	64.3		
T.I.G.-Welded	340°C	0	75.4	309	76.9	^x 433 ²	78.3	1009	72.4	^x 1917	81.4	4005	61.2		
200 Nickel															
As-Rec'd.	340°C	0	66.7	309	65.0	^x 433 ²	69.9	1009	57.0	^x 1917	62.5	4005	61.1		
E.B.-Welded	340°C	0	63.1	309	59.6	^x 433 ²	63.7	1009	59.3	^x 1917	62.5	4005	46.6		
T.I.G.-Welded	340°C	0	64.1	309	63.4	^x 433 ²	69.0	1009	65.2	^x 1917	66.6	4005	51.6		

^xSamples Received 10 Scram Cycles

- NOTES: 1. Minimum sample size: 3 pieces each condition.
 2. Due to improper functioning of the Instron Test Equipment, data reported for 433, 509, and 520 hours is not reliable.
 3. Break outside weld area.

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The room temperature tensile data, after aging, for the various brazed assemblies are listed in Table IX. No significant change is noted even after the amount of aging indicated. It is apparent that braze uniformity may not be expected in these pieces because the time-temperature relationship was not optimized for this particular geometry. Moreover, certain specimens were subjected to test until failure occurred whereas others were limited to a maximum of 40,000 psi. These specific tests demonstrate the superiority of the NiSiB alloy braze in conjunction with Hastelloy "B". After 4003 hours of steady-state vacuum exposure at 800°C, this braze joint measured an ultimate strength of 6513 Mpsi, 316L stainless steel bonded to OFHC copper with the Ni-Au eutectoid alloy braze and aged in vacuum for 4011 hours at 340°C had an ultimate strength of 37.8 Kpsi.

To date, results of the evaluation of materials and bonds continue to support the selection of Hastelloy "B" and the 316L stainless steel for the hot and cold heat-exchanger plates, respectively.

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TABLE IX

HEAT-EXCHANGER PLATE DEVELOPMENT THERMAL STORAGE TESTING IN VACUUM

Specimen Description	Aging Temp. °C	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Room Temperature Tensile Data After Aging									
						Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi	Aging Time	Strength Kpsi
<u>Hastelloy "B" to Nickel</u>															
Ni-Au Eutectoid Alloy	800	0	>40.0	499	>40.0	1000 ²	35.5	2002	35.0	4000 ²	35.3	8000	16000 ²		
Ni-Plate & Ti Shim	800	0	22.3	500	>40.0	1000 ²	37.9	2000	40.2	4000 ²	44.1				
Ni with Si & B Alloy	800	0	37.8	543	>40.0	999 ²	70.8	2000	>40.0	4003	65.3				
<u>Hastelloy "X" to Nickel</u>															
Ni-Au Eutectoid Alloy	800	0	>40.0	543	>40.0	999 ²	54.7	2000	38.3	4016	40.8				
Ni-Plate & Ti Shim	800	0	23.9	500	>40.0	1002 ²	59.8	2000	21.0						
Ni with Si & B Alloy	800	0	>40.0	496	>40.0	998 ²	56.7	2014	57.2	4000	63.1				
<u>316L S.S. to OFHC Copper</u>															
Ni-Au Eutectoid Alloy	340	0	>40.0	259	>40.0	518	39.2	1039	49.3	2016	>68.5 ⁴	4011	37.8		
Ge-Cu Alloy	340	0	17.9	281	22.9	496	18.9	1001	24.8	2016	16.9	4011	26.0		
<u>200 Nickel to OFHC Copper</u>															
Ni-Au Eutectoid Alloy	340	0	34.6	-	-	430 ²	35.5	1028	34.8	2032	32.8	4011	37.8		
Ge-Cu Alloy	340	0	13.6	-	-	430 ²	12.2	1028	16.1	2032	12.5	4011	15.2		

- NOTES: 1. Minimum sample size: 3 pieces each condition.
 2. Pieces representing this condition received 10 scram cycles in addition to aging.
 3. Strengths marked greater than (>) the indicated value did not break under this stress.
 4. These pieces were scram-cycled at 775°C in error.

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VIII. BeO CERAMICS

The flexural strengths of BeO ceramics produced commercially in 1967 by Vendors A, B, and C were found to be 11,000 psi, 23,000 psi, and 34,000 psi, respectively. Continuing with the upgrading of the testing program for evaluation of physical properties of BeO ceramics, a series of tests were run to determine if thinner gold shims could be used successfully in diffusion-bonded BeO test structures. Results of these tests indicate that such a substitution can be made. However, before the change is effected, rectangular gold shims, which are more in keeping with current module construction, will be evaluated.

A commercial quantity of metallized BeO ceramics received from Vendor C was evaluated. Standard metallizing adherence tests (peel tests of brazed assemblies) indicated marginal adherence, whereas diffusion-bonded tensile tests gave excellent results. Because that vendor had lowered the metallizing temperature for this run, both he and RCA remetallized samples from this lot at a higher temperature. Both of these runs were unsuccessful. The vendor then metallized a small experimental lot of ceramics at the initial temperature; the results of his peel-test, using a different braze material, were quite satisfactory. Discussion with the vendor indicated that in addition to the brazing alloy difference, his test differed from the RCA test in that his metallized test pieces were nickel-coated and not nickel-plated. To resolve the discrepancies, a program is underway at RCA and at the vendor's plant:

RCA will perform standard metallizing adherence tests on:

1. Vendor-C ceramics nickel-plated by RCA
2. Vendor-C ceramics nickel-coated by Vendor C.

Vendor C will perform standard brazed metallizing tests on:

1. Vendor-C ceramics nickel-plated by Vendor C
2. Vendor-C ceramics nickel-coated by Vendor C.

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IX. 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 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 is provided in the Phase IIA Final Report.

Table X is a current summary of metallographic, tensile, and constant deflection data accumulated on samples having the indicated life and environmental testing histories. No significant change has been observed in the physical characteristics and metallurgical properties of the flexible connector samples. Tensile strengths and the 25-mil constant deflection forces after 40 cycles continue to remain well within the acceptable limits described in the above-mentioned report. All bonds remain metallurgically sound. The small voids observed in as-bonded samples in the Cu-to-Cu braze have shown no indication of linking-up or increasing in size or quantity as noted in samples which have been subjected to 600 to 1000 hours of accelerated storage life at 340°C. This period is calculated to be equivalent to 6000- to 10,000-hour life at the design temperature of 270°C.

CONFIDENTIAL

CONFIDENTIAL

42

TABLE X

FLEXIBLE RELIEF CONNECTOR TEST & EVALUATION DATA

Group No.	TESTING				POST TEST EVALUATION													
	Hours Storage Life @ 340°C	Environmental Tests		Hours Storage Life @ 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) @			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	20,000	At Random in Web	20,500 & 21,200	At Random in Web	8.0	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 The % of Fractured Webs for a given side ranged from 0 - 50% of total	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	-	-	-		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	20	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	

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X. SUMMARY

In the second quarter of Phase IIB, the major effort has been directed at the assembly of test structures to demonstrate the capability of components and to evaluate possible problems. Six such structures were fabricated during this quarter. Fixtures and tooling variations required for these test structures and their components were also produced.

Work effort on the thermoelement assembly also continued with major emphasis on the evaluation of materials, processes, and methods for preventing braze penetration through the graphite shoe of the element assembly. Alternate brazes for the graphite-to-tungsten bond in the assembly were also investigated.

The development of the hot-stack assembly procedures continued with both diffusion-bonded and brazed systems. With either of these methods, metallurgical diffusion barriers are required to prevent interdiffusion of the hot-stack materials, and several barrier systems using refractories, inter-metallics, and ceramics were investigated.

No major problems remain with either the hot and cold heat-exchanger plates or with the flexible connector assembly. Life tests are continuing and are being evaluated on these components primarily for confirmation purposes.

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