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TOPICAL REPORT

for

**PRELIMINARY INVESTIGATION OF III-V
COMPOUND THERMOELECTRIC MATERIALS
DIRECTED TOWARD SEGMENTED
SILICON-GERMANIUM III-V COMPOUND
ADVANCED NUCLEAR POWER SYSTEMS (u)**

CONTRACT (30-1)-3500

FOR THE PERIOD
SEPTEMBER 1, 1965 TO FEBRUARY 28, 1966

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Prepared for:
New York Operations Office
U.S. Atomic Energy Commission

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I. INTRODUCTION

A. Importance of Segmenting Silicon-Germanium Couples

The practical application of silicon-germanium couples in nuclear thermoelectric devices has been demonstrated in such systems as SNAP-10A. A major objective in the advancement of the State-of-the-Art of nuclear thermoelectric power systems with silicon-germanium couples is the development of couples with higher efficiency. This is best accomplished by the development of segmented couples designed around silicon-germanium. The thermoelectric materials that offer the most promise are III-V compound alloys in segmented structures.

B. Status of Segmented Silicon-Germanium, III-V Compound Alloy Couples Prior to the Present Program

The status of the development of a segmented silicon-germanium, III-V compound alloy couple at the conclusion of the previous program, AT(30-1)-3500, (Task I) was as follows:

- 1) A practical demonstration couple with a segmented n-leg was developed using n-type InAs-GaAs segmented to n-SiGe. Based on the material properties of its components, this couple performed as predicted. The losses noted were due to mismatch of thermoelectric material properties.
- 2) A SiGe-InAs-GaAs segment joint was developed which had minimum electrical and thermal losses.
- 3) An InAs-GaAs to Mo shoe joint was developed which was electrically stable after 2,000 hours of isothermal life test at 550°C in a sealed tube.
- 4) The upper temperature limit of InAs-GaAs, based on weight loss in sealed tubes and joint stability tests, was defined as 500 to 550°C.
- 5) Preliminary data indicated that the resistivity of InAs-GaAs was stable with life.
- 6) Work was initiated on a back-up segmenting joint using graphite.
- 7) A technique was developed that gives a direct comparison between a segmented couple and an all-SiGe couple under the same experimental conditions.

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- 8) Tensile tests on bonded InAs-GaAs elements indicated that the shoe bond was stronger than the bulk material.
- 9) A materials study produced a better theoretical understanding of III-V compound alloys as thermoelectric materials and to the methods of fabrication of p-type GaSb alloys.

C. Program Objectives

The primary objective of Task II of the program is the optimization of the present n-segmented couple and a preliminary determination of its performance and reliability. A parallel program on advanced technology is included. The principle objectives are:

- 1) The development of a segmented couple with high reliability and with improved performance due to the higher figure-of-merit of n-type InAs-GaAs and material property match.
- 2) Experimental demonstration of the potential performance gains due to the higher stability of n-type InAs-GaAs.
- 3) Analytical and experimental studies to determine growth potential.
- 4) Recommendations for future application.

D. Major Tasks

The following six major tasks are to be performed to accomplish the objectives of this program:

Task 1.0 ADMINISTRATIVE

This task covers the administrative effort required for the total project. It covers the expenditures in effort for items not specifically assignable to other tasks, such as; preparation of reports, manpower and materials budgeting, trips, visits, and meetings of general project nature.

Task 2.0 OPTIMIZATION OF COUPLE DESIGN

The objective of this task is the optimization of a SiGe couple with a segmented n-leg. Theoretical calculations have shown that the development of a SiGe couple with a segmented

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n-leg can lead to a potential improvement in couple efficiency of 15 to 20 percent over an all-SiGe couple. This improvement can be attributed to two factors: the higher Z and the unusually stable life characteristics of InAs-GaAs. The higher Z of the n-InAs-GaAs leads to a 7 to 12 percent improvement while the more stable material properties can give approximately 5 percent improvement.

Task 3.0 COUPLE TESTING AND RELIABILITY

The objective of this task is to evaluate the performance and reliability of the optimized couple. This includes mechanical, electrical, and thermal stability, both initially and after life test. It also includes an evaluation of the life stability of the material properties at various temperatures.

Task 4.0 OPTIMIZATION OF MATERIAL FABRICATION

An evaluation of material fabrication techniques is required to: 1) select a process fabrication technique with maximum reproducibility, 2) improve material properties, and 3) investigate methods of cost reduction.

An analysis of the optimized couple design will summarize the results of Tasks 2.0, 3.0, and 4.0, and provide recommendations for couple design from reliability and ease of fabrication viewpoints.

Task 5.0 ADVANCED TECHNOLOGY

The objective of this task is to develop approaches for growth potential of SiGe segmented couples. The improvement of the performance of the present segmented couple can be accomplished by: 1) the development of a p-III-V compound alloy of better performance than p-type SiGe, 2) the development of an improved n-III-V compound alloy, 3) consideration of other thermoelectric materials for segmenting, such as tellurides, and 4) the development of couple designs to increase segment interface temperature.

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Task 6.0 ANALYSIS AND RECOMMENDATIONS

Recommendations for the future application will be made based on the analysis of Tasks 2.0 through 5.0. The important factors that will be considered are: performance, reliability, ease of fabrication, and growth potential. Also, the effect on system design will be considered with particular emphasis on the interchangeability with an all-SiGe couple.

E. Status of the Present Program

Phase I of this program will be completed on February 28, 1966. The following pages of this report present a review of the progress made during this first six month phase. A summary and conclusions are presented in Section VI.

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II. COUPLE DESIGN

A. Introduction

The development of a SiGe couple with a segmented n-leg can lead to potential improvement in couple efficiency of 15 to 20 percent over an all-SiGe couple in nuclear thermoelectric power systems. This improvement is due to two factors: the higher Z of the InAs-GaAs and the unusually stable life characteristics of the n-type InAs-GaAs. The higher Z of the n-type InAs-GaAs leads to a 7 to 12 percent improvement, while the more stable material properties can give approximately 5 percent improvement.

The development of a joint with negligible losses, the ductile metal shoe, has indicated that the effect of the higher Z can be realized in a practical couple design. The effort expended during Phase I has been directed toward optimization of the segmented n-leg couple design with a ductile metal segment, with respect to efficiency and reliability. The major tasks associated with optimizing efficiency are: 1) a reduction of the thermoelectric material property mismatch, and 2) developing ductile metal segment and joint fabrication techniques to obtain maximum reliability and process control.

An evaluation is also being conducted on alternate segment joint designs, in particular the graphite joint. This joint can lead to greater reliability due to the reduction in the number of interfaces to be fabricated, and will permit a wider range of SiGe compositions.

B. Optimum Material Match

Theoretical computations have shown that material properties, and in particular, material resistivities, have a significant effect on the efficiency of a segmented couple.

The theoretical efficiency computations for a segmented couple developed during the previous program have been used to generate efficiency curves for optimum couple design with the n-InAs-GaAs and p-SiGe resistivity as parameters. These curves are shown in Figure 1 for the temperature range $T_h = 700^\circ\text{C}$, $T_s = 550^\circ\text{C}$, and $T_c = 250^\circ\text{C}$. The curves compare the efficiency of an all-SiGe couple after 1,500 hours of life with that of a segmented n-leg couple. As shown, an efficiency gain of 14.5 percent can be realized by using n-type InAs-GaAs and p-type SiGe having optimum resistivities.

1. "Preliminary Investigation of III-V Compound Thermoelectric Materials Directed Toward Segmented Silicon-Germanium III-V Compound Advanced Nuclear Power Systems (u)", TH355-8-642, NYO 3500-8, Sept. 15, 1965.

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COMPARING A SEGMENTED n-leg InAs-GaAs-SiGe COUPLE WITH AN ALL SiGe COUPLE AFTER 1500 HOURS OF LIFE, AS A FUNCTION OF p-SiGe AND n-InAs-GaAs RESISTIVITY.

OPERATING CONDITIONS: $T_h = 700^\circ \text{C}$, $T_s = 550^\circ \text{C}$, $T_c = 250^\circ \text{C}$

NOTE: ALL RESISTIVITY VALUES ARE AT OPERATING TEMPERATURES

FIGURE 1 - THEORETICAL EFFICIENCY GAIN

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Based on the results of the theoretical curves shown in Figure 1, both n-type InAs-GaAs and p-type SiGe materials have been fabricated having the optimum material properties. Elements having the correct length to area ratios have been designed and fabricated for optimized test couples. Elements for an optimized all-SiGe couple have also been designed and fabricated to provide comparison test couples.

C. Shoe and Joint Optimization for Reliability

Preliminary life tests of segmented legs at 550°C have shown that certain materials, such as nickel, were not compatible with the InAs-GaAs material. Therefore, the initial effort was directed towards the elimination of these undesirable materials from the segment joint. Nickel has been used as a plating on the n-type SiGe tungsten shoe, and therefore, an investigation was made of various braze alloys which would wet bare tungsten. A copper-titanium reactive metal braze produced the desired result. The elimination of all traces of germanium which had deposited on the segment during the various high temperature processing steps was also desirable. This was accomplished by sandblasting the segment after processing. Life test results show the effectiveness of these measures.

On the previous program, a bond of InAs-GaAs to a rhodium plated molybdenum shoe was developed using a copper-silver-indium brazing alloy. Initially, this bond is stronger than the InAs-GaAs material. However, there is evidence that the bond strength deteriorates with life. Therefore, other brazing alloys were investigated. Life tests and tensile tests are now being performed on InAs-GaAs elements bonded with copper-silver-tin and copper-silver-phosphorus brazing alloys.

D. Couple Design and Fabrication

Basically, the overall couple design will be similar to the comparison couple developed on the previous program; however, the graphite shoe has been modified to accommodate an enclosed heater. Since the emissivity of graphite is known, hot shoe radiation losses can be computed. Measurements of heater power input can then be utilized to permit a reasonably accurate determination of power input to each couple. Power output can then be measured and efficiency calculated.

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The cold junction has also been modified to incorporate a copper mounting stud with a bolted-on aluminum radiator. This modification has been made to: 1) eliminate the radiator brazing operation, and 2) to permit interchangeable radiators for various cold junction temperatures. A detailed drawing of the modified test couple is shown in Figure 2. Figures 3 and 4 are photographs of a completed couple ready for test.

To insure accurate, reliable bonding of test couples, a brazing fixture having two pressure loaded rods has been designed and constructed. A photograph of this fixture is shown in Figure 5.

E. Alternate Joint Design

Preliminary fabrication studies performed on the previous program demonstrated the feasibility of using graphite as a segment with a direct bond of SiGe to graphite to InAs-GaAs. Graphite, due to its low Young's Modulus, appears to have sufficient ductility to prevent stress concentrations due to the thermal mismatch between SiGe and InAs-GaAs. Unfortunately, the introduction of the graphite segment introduces small, but not negligible, electrical and thermal losses which do not occur in the ductile metal segment. However, since graphite is chemically inert to both SiGe and InAs-GaAs at operating temperatures, this segment may provide more reliable long life operation. Although the major effort on this approach is scheduled for Phase II of this program, some preliminary theoretical calculations have been made to determine the electrical and thermal losses anticipated in the use of a graphite segment. These data are shown in Figure 6.

These losses have been computed for two distinctly different types of graphite, both of which have been found suitable for bonds to n-SiGe. Type 886S is a premium grade extruded graphite of relatively low cost. Type JT-0981 is a composite material containing graphite, zirconium, and silicon; it is relatively expensive. Table I summarizes the room temperature physical properties of these two materials. Molded or extruded graphite normally has directional properties, as shown in Table I. When graphite is used for segmenting, it is positioned such that the with-grain direction is on axis. Therefore, the with-grain properties of electrical resistivity, thermal conductivity, and tensile strength, and the cross-grain thermal expansion are the important considerations.

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DESIGN SHOWN WITH A SEGMENTED n-leg, BOLT ON RADIATORS, AND A COMBINATION HOT STRAP-HEATER BLOCK WITH AN ENCLOSED COATED TUNGSTEN HEATER. THE ALL SiGe COMPARISON COUPLE UTILIZES IDENTICAL COMPONENTS.

FIGURE 2 - OPTIMIZED TEST COUPLE DESIGN

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FIGURE 3 N-LEG SEGMENTED COUPLE AFTER COMPLETION
OF ALL BONDING OPERATIONS

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FIGURE 4 N-LEG SEGMENTED COUPLE WITH ALUMINUM RADIATORS

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FIGURE 5 COUPLE BONDING FIXTURE

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CONSIDERING THE USE OF A SEGMENT 1 SQ. CM. IN AREA, AND 0.25 CM HIGH (0.1")
SEGMENT TEMPERATURE = 550° C

TYPE JT-0981 IS A COMPOSITE GRAPHITE MANUFACTURED BY UNION CARBIDE CORP.
TYPE 886S IS A PREMIUM GRADE GRAPHITE MANUFACTURED BY SPEER CARBON CO.

FIGURE 6 - ELECTRICAL AND THERMAL LOSS - GRAPHITE SEGMENT

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TABLE I

ROOM TEMPERATURE PROPERTIES OF GRAPHITES FOR USE AS SEGMENTS

<u>Characteristics</u>	<u>JT-0981</u>	<u>886S</u>
Resistivity ($m\Omega$ -cm)		
with-grain	0.2	0.68
across-grain	0.4	-
Tensile Strength (psi)		
with-grain	9000	2000
across-grain	4000	-
Thermal Expansion ($^{\circ}C^{-1}$)		
with-grain	5.0×10^{-6}	2.5×10^{-6}
across-grain	6.5×10^{-6}	4.2×10^{-6}
Thermal Conductivity (watt/cm- $^{\circ}C$)		
with-grain	0.6	1.6
across-grain	0.4	-
Density (gm/cm^3)	3.0	1.68

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While either of the two materials may prove suitable for segmenting, it should be noted that the Type JT-0981 is a much closer thermal expansion match to InAs-GaAs, and for all other characteristics with the exception of thermal conductivity, is to be preferred over Type 886S. It may be that the higher thermal loss associated with this material is more than canceled out by the lower electrical loss.

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III. COUPLE TESTING AND RELIABILITY

A. Introduction

A comprehensive evaluation program is being carried out to determine the reliability of the optimized couple design. This effort includes the evaluation of the basic InAs-GaAs material. Work on Phase I has primarily been directed towards the testing and evaluation of InAs-GaAs material per se, bonded InAs-GaAs elements, and InAs-GaAs - n-type SiGe segmented legs. These tests include mechanical strength and thermoelectric property measurements before and after isothermal life.

The tests conducted during Phase I will be continued during Phase II; however, the major effort of this Phase will be directed towards the test and evaluation of optimized couples.

B. Mechanical

Tensile tests have been performed on InAs-GaAs material per se, and on bonded InAs-GaAs elements. A limited number of tests have been performed after isothermal life tests at 550°C. The results of these tests are presented in Table II.

Table II shows that all failure points were located in the InAs-GaAs material on tests of initial and short term life elements. Nearly all of these failures were near the midpoint of the elements, which is an indication that the bond area is not a weak point initially. However, two tests on elements after 321 and 415 hours of life at 550°C did show a partial failure in the bond area, and lower tensile strength. Additional tests are in process on elements utilizing brazing alloys of various composition. Tests will also be performed on elements bonded to graphite.

Attempts are being made to improve the strength of the tellurium doped material, as evidenced by the tensile tests on elements fabricated from an ingot having a slower zone leveling rate. This work is discussed further under Section IV.

Shock and vibration tests, and thermal shock and thermal cycling tests on segmented couples are scheduled for the second phase of this program.

C. Isothermal Life

Two types of isothermal life tests on bonded elements are in process. The original tests, initiated on the previous program, were conducted under conditions in which the test specimens were

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TABLE II

TENSILE TEST RESULTS ON InAs-GaAs

<u>Specimen Description</u>	<u>Quantity</u>	<u>Life Hours</u>	<u>Average Tensile Strength psi</u>	<u>Fracture Location</u>
InAs-GaAs Material	2	0	800	Material
Bonded Elements:				
Se doped InAs-GaAs	9	0	1255	Material
Se doped InAs-GaAs	2	104	1171	Material
Se doped InAs-GaAs	2	160	1008	Material
Se doped InAs-GaAs	1	321	738	Material and Bond
Se doped InAs-GaAs	1	415	973	Material and Bond
Te doped InAs-GaAs	8	0	700	Material
Te doped InAs-GaAs*	3	0	1058	Material

* This particular ingot, IGAN-15, was processed at a slower zone leveling rate, processing data are shown in Table V.

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sealed in an evacuated tube. Four of these test specimens are still on life: one (1), at 500°C, has accumulated 9,370 hours at temperature; while the other three (3), at 550°C, have passed the 5,000 hour point. No appreciable degradation in total resistance, or bond resistance has been observed.

Isothermal life testing which was initiated on the present program utilizes a continuously pumped vacuum system to house the test specimens. Three types of specimens are being tested, i.e. 1) bonded InAs-GaAs elements, 2) bonded InAs-GaAs with ductile metal segments, and 3) InAs-GaAs - SiGe segmented legs. These three groups are in isolated tubes to avoid possibilities of cross-contamination. Typical life test performance curves for a segmented leg are plotted in Figures 7 and 8. Individual life test data are tabulated for all current isothermal tests in the Appendix.

The InAs-GaAs - SiGe segmented legs utilizing the copper-titanium reactive metal braze appear to be satisfactory. It should be noted that on prior tests employing copper-silver bonds to nickel plated shoes, visual deterioration was evident after short term life. This has not been observed on these new tests. Optimized segmented couples and all SiGe comparison couples have been fabricated and are being prepared for test and evaluation.

D. Material Properties Evaluation

1) Thermoelectric Properties

All InAs-GaAs material was selenium doped on the previous program. Recently, a number of ingots have been fabricated with tellurium doping. The thermal diffusivity technique has been utilized to measure the thermoelectric properties of this material. These data indicate that the tellurium doped material is equivalent to selenium doped material. The results of these measurements and the prior measurements on selenium doped InAs-GaAs have been summarized to generate the figure-of-merit curves shown in Figure 9. These curves show that for the operating range of $T_s = 550^\circ\text{C}$ to $T_c = 250^\circ\text{C}$ (average temperature = 673°K), discussed under Section II, a Z of $0.875 \times 10^{-3} \text{ }^\circ\text{K}^{-1}$ is obtained for the optimum InAs-GaAs resistivity of $1.5 \text{ m}\Omega\text{-cm}$.

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FIGURE 7 SEGMENTED LEG ELEMENT RESISTANCE AS A FUNCTION OF LIFE AT 550°C.

FIGURE 8 SEGMENTED LEG ELEMENT SEEBECK AS A FUNCTION OF LIFE AT 550°C.

FIGURE 9 - InAs-GaAs Z VS ρ

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2) Life Stability

Room temperature resistivity and Seebeck as well as weight loss have been monitored on all of the life tests now being conducted at 550°C in a pumped vacuum system. A number of tests have completed 1000 hours with no appreciable change in resistivity or Seebeck, and with negligible weight loss.

The stability of the InAs-GaAs has also been noted in tests of non-optimized segmented couples. This type of couple has been tested on a cycled basis for 150 hours; data obtained on the InAs-GaAs element resistance and operational Seebeck is shown in Table III.

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TABLE III

InAs-GaAs OPERATIONAL STABILITY

<u>Time</u> <u>(hours)</u>	<u>T_{seg.}</u> <u>(°C)</u>	<u>T_{cold}</u> <u>(°C)</u>	<u>R_{tot.}</u> <u>(mΩ)</u>	<u>Q</u> <u>(μV/°K)</u>
0	513	321	1.43	204
6	501	316	1.43	205
26	491	313	1.43	206
100	491	315	1.43	206
150	490	314	1.43	205

NOTE: These data are obtained from measurements on the InAs-GaAs element in a segment leg of a non-optimized comparison couple fabricated during Task I of this program. The couple was thermal cycled to room temperature three times during the 150 hour time period.

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IV. OPTIMIZATION OF MATERIAL FABRICATION

A. Introduction

The evaluation of various material fabrication techniques is desirable for the following reasons:

- 1) Improvement of material properties.
- 2) Selection of process fabrication techniques with maximum reproducibility.
- 3) Cost reduction.

Phase I effort has been directed primarily towards the optimization of material properties for strength and thermoelectric characteristics.

B. Optimized Thermoelectric Characteristics

Theoretical computations have indicated that material of optimum ρ is desirable for minimum mismatch - maximum efficiency, in a segmented couple. Experimental tests have indicated that, by varying the doping level, and the type of dopant, material of various resistivity could be made. Room temperature resistivity and surface Seebeck data indicated that tellurium doped material appears to have an improved Q^2/ρ in comparison to selenium doped InAs-GaAs. Therefore, tellurium doped ingots having the optimized resistivity, as computed theoretically, were fabricated to provide elements for optimized test couples.

The theoretical computations also indicated the desirability of using a p-type SiGe material of slightly higher resistivity than normally used in an all-SiGe couple. Therefore, p-type SiGe ingots of the desired resistivity were also fabricated to provide elements for optimized test couples.

During the first phase of this program, a total of nine InAs-GaAs ingots were produced, three with selenium doping, and six containing tellurium. Table IV presents the room temperature thermoelectric data obtained on these ingots. All ingots have a basic composition of 70 at. percent and 30 at. percent GaAs.

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TABLE IV

CHARACTERIZATION OF ZONE LEVELED InAs-GaAs INGOTS

<u>Ingot</u>	<u>Growth Rate (inch/hour)</u>	<u>Dopant</u>	<u>Concentration (wt.% of total)</u>	<u>Resistivity (mΩ-cm)</u>	<u>Seebeck (μv/$^{\circ}$C)</u>
IGAN-9	0.025	Se	0.0013	1.2	140
IGAN-10	0.025	Te	0.0065	0.9	150
IGAN-11	0.025	Se	0.0014	1.7	180
IGAN-12	0.025	Te	0.0039	1.3	190
IGAN-13	0.025	Te	0.0038	1.3	180
IGAN-14	0.025	Te	0.0055	0.85	140
IGAN-15	0.015	Te	0.0055	1.5	180
IGAN-16	0.025	Se	0.0024	1.0	125
IGAN-17	0.025	Te	0.0045	1.2	185

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C. Optimum Material Strength

The data tabulated in Table II, Section III, indicated that initially and on short term life, the strength of the InAs-GaAs is the limiting factor in bonded element strength. These data also indicated that using standard growth rates, the tellurium doped material was weaker than selenium doped material. However, a reduction in the growth rate, i.e. IGAN-15, did produce stronger material. Modifications in zone leveling techniques to improve strength will continue during Phase II. However, it is believed that vacuum hot-pressing provides the most promise for a major increase in material strength. Experimentation with this technique has recently been started. Several samples have been made with densities as high as 99 percent. These pellets are now being prepared for bonding and subsequent tensile tests.

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V. ADVANCE TECHNOLOGY

A. Introduction

The objective of this work is to develop approaches for the growth potential of SiGe segmented couples. This can be accomplished by:

- 1) Development of a p-type III-V compound alloy of better performance than p-type SiGe.
- 2) Development of an improved n-type III-V compound alloy.
- 3) Consideration of other thermoelectric materials for segmenting, such as tellurides.
- 4) Development of couple designs to increase segment interface temperature.

The technique of segmented thermocouple construction employing III-V compound alloys in the thermoelement portion operating below 600°C, and SiGe alloys in the portion operating at higher temperatures holds promise of improved performance over SiGe alloy thermocouples alone. This was shown first in the case of n-type InAs-GaAs alloys which have a higher figure-of-merit than n-type SiGe alloy. In the previous contract period, p-type alloys in the systems GaSb-InSb, GaSb-GaAs, and GaSb-GaP were investigated for material superior to p-type SiGe alloy.

The evaluation of the P-GaSb-GaP alloy was not completed in the previous contract period and was continued to determine the feasibility of preparing p-type GaSb-GaP alloys. These studies showed that the solid solubility of GaP in GaSb was too low to provide good thermoelectric properties. The evaluation of p-type materials was discontinued early in the present phase. While the best material, GaSb-InSb alloy, was only approximately as good as p-type SiGe, the resulting data on the thermoelectric properties indicated that as n-type material these systems might be not only better than n-type SiGe alloy, but also better than n-type InAs-GaAs. Accordingly, the work in the past six months consisted of measuring the thermoelectric properties of n-type GaSb-InSb and GaSb-GaAs alloys.

The zone-leveling technique used to prepare the n-type samples was similar to that used in preparing the p-type samples with Te substituted for Zn as the dopant. Therefore, this method of preparation will not be described in detail in this report. However, it was found necessary to introduce a vacuum casting step into the preparation of the charge material for the zone-leveling operation to reduce cracking in the final ingot.

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B. Measurement Techniques

Room-temperature measurements of the Hall coefficient R_H and Seebeck coefficient Q were made to characterize the materials prior to measurements in the high-temperature apparatus. High-temperature measurements of the thermoelectric parameters were made using a thermal diffusivity apparatus.¹ The thermal diffusivity k/c_p was converted into thermal conductivity k using the computed specific heat c_p per unit volume at constant pressure for each composition. It has been shown previously^{2,3} that it is possible to fit all available experimental specific heat data on the III-V compounds to one curve by plotting c_p/c_{dp} against T/θ , where c_{dp} is the Dulong-Petit specific heat and θ is the Debye temperature. For $T/\theta \geq 1.2$, the c_p/c_{dp} values used for the GaSb alloys can be expressed by the relationship, $c_p/c_{dp} = 0.88 + 0.068 T/\theta$ ($J\ cm^{-3}\ deg^{-1}$). The Dulong-Petit specific heats per unit volume, $c_{dp} = 3.312 \times 10^{-24}/a^3$ ($J\ cm^{-3}\ deg^{-1}$), were computed from the lattice parameter a . The Debye temperatures of the alloys were derived from the expression,⁴ $\theta = 4.19 \times 10^8 a^{-(3/2)M-(1/2)G}$, where M is the mean "gram atomic weight" of the alloy and G is a function of the elastic constants. The value of G was obtained from a linear interpolation between the values⁴ for GaSb ($G = 0.931$), InSb ($G = 0.867$), and GaAs ($G = 0.938$).

C. Thermoelectric Properties of n-Type GaSb Alloys

Listed in Table V are the compositions and Hall data on n-type GaSb-InSb alloys measured in this investigation. The temperature dependences of the Seebeck coefficient Q , electrical resistivity ρ , and thermal resistivity $1/k$ for GaSb-InSb alloys are shown in Figures 10, 11, and 12, respectively.

The figures-of-merit, z , for the n-type GaSb-InSb alloys calculated from the data in Figures 10 through 12 are shown in Figure 13. For comparison, the figures-of-merit for the best n-type SiGe and n-type InAs-GaAs alloys are also shown in Figure 13. The figure-of-merit values for the GaSb-GaAs alloys were not included in Figure 13, since they are significantly lower than those of the GaSb-InSb alloys.

-
1. B. Abeles, G. D. Cody, and D. S. Beers, J. Appl. Phys. 31, 1585(1960).
 2. E. F. Steigmeier and I. Kudman, Phys. Rev. 132, 508(1963).
 3. R. E. Miller, to be published.
 4. E. F. Steigmeier, Appl. Phys. Letters, 3, 6(1963).

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TABLE V

HALL MEASUREMENTS OF n-TYPE GaSb-InSb ALLOYS

<u>Sample</u>	<u>Composition (mole % GaSb)</u>	<u>Carrier Concentration n (cm⁻³)</u>	<u>Mobility μ (cm²/V-sec)</u>
19	83	3×10^{17}	10,400
20	84	1.1×10^{18}	4,700
22	55	8×10^{17}	12,000
23	35	2.1×10^{17}	19,800
26	80 (nom.)	4.0×10^{17}	10,200
27	70 (nom.)	1.55×10^{18}	8,600

FIGURE 10 THE TEMPERATURE DEPENDENCE OF THE ABSOLUTE SEEBECK COEFFICIENT Q FOR n-TYPE GaSb-InSb ALLOYS.

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FIGURE 11 THE TEMPERATURE DEPENDENCE OF THE ELECTRICAL RESISTIVITY ρ FOR n-TYPE GaSb-InSb ALLOYS.

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FIGURE 12 THE TEMPERATURE DEPENDENCE OF THE TOTAL THERMAL RESISTANCE $1/K$ FOR n-TYPE GaSb-InSb ALLOYS.

FIGURE 13 THE TEMPERATURE DEPENDENCE OF THE FIGURE OF MERIT
Z FOR n-TYPE GaSb-InSb ALLOYS.

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GaSb-InSb alloy samples with GaSb contents of 35 percent and 55 percent, and electron concentrations of $2 \times 10^{17} \text{ cm}^{-3}$ and 8×10^{17} , respectively, were found to have the highest figures-of-merit. In the temperature range, 300 to 600°K, the improvement in figure-of-merit over the best n-type InAs-GaAs is approximately 40 percent. Above 700°K, the figures-of-merit decrease and are smaller than those of the InAs-GaAs system. In the same temperature range, 300 to 600°K, the InSb-GaSb alloys represent an improvement of almost 100 percent over that of SiGe alloys. The figures-of-merit of these alloys are comparable to those of the SiGe alloys at 800°K.

These alloys, therefore, are promising n-type materials for thermoelectric power generation in the temperature range 300 to 800°K.

D. Increased Segment Temperature

Two approaches to increasing the InAs-GaAs segment interface temperature are under investigation. The first is a mechanical design approach which consists essentially of a mechanical structure that will limit the molecular flow of the volatile species. An initial design has been formulated and components are now being fabricated for initial test.

The second approach entails the use of a protective coating to limit the flow of the volatile species. Initial attempts to fire on a glass of matching thermal expansion have shown promise, however, glass adherence was marginal. Additional effort is required to establish the feasibility of this approach.

VI. SUMMARY AND CONCLUSIONS

A. Introduction

The objective of this section is to present a brief review of the work detailed in Sections II, III, and IV; to analyze the results with regard to the overall objectives of the Task II program, and to summarize the conclusions reached at this time.

B. Analysis

Theoretical computations have shown that efficiency gains of 14 percent or more can be obtained by using an InAs-GaAs segmented n-leg. However, in order to realize this gain in practice, the following conditions must be met:

- 1) InAs-GaAs having the high figure-of-merit used in the computations must be fabricated with a reproducible process.
- 2) This material must also be fabricated with the optimum resistivity for minimum mismatch loss.
- 3) P-type SiGe must be fabricated with the optimum resistivity for minimum mismatch loss.
- 4) The segment joint must have no appreciable electrical or thermal losses.
- 5) The InAs-GaAs to shoe contact resistance must be negligible.
- 6) Feasible couple fabrication processes must be established.
- 7) Accurate techniques for comparing the efficiency of a segmented couple with an all-SiGe couple must be developed.

If these conditions are fulfilled, a segmented couple could be made and, in initial operation, would show some, but not the maximum, efficiency gain over an all-SiGe couple. However, in order to realize the maximum gain with reliability, the segmented couple must take advantage of more stable material properties on life. This imposes additional conditions as follows:

- 8) The InAs-GaAs must exhibit stable material properties during operation at temperatures as high as 550°C.
- 9) The InAs-GaAs must maintain its material strength with life at operating temperatures.

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- 10) Bonds to InAs-GaAs must be stable electrically and maintain mechanical strength with life at operating temperatures.
- 11) SiGe, segment components, and bonding materials must be compatible with InAs-GaAs.
- 12) Thermal stress must be negligible in the InAs-GaAs.

At the conclusion of Phase I of the program, it is believed that conditions 1 through 8 can be satisfactorily met.

Segmented couples utilizing InAs-GaAs and p-type SiGe of optimum resistivity are now being prepared for test and evaluation. Thermal diffusivity measurements have confirmed the high figure-of-merit of the InAs-GaAs, (Conditions 1,2,&3).

During the previous program, the ductile metal segment was developed which had negligible electrical and thermal losses, (Condition 4).

The InAs-GaAs shoe bond contact resistance is extremely low; measurements made on bonded elements normally average 0.02 milliohm. This is approximately one-fourth that of the shoe contact resistance of n-type SiGe, (Condition 5).

Optimized segmented couples and all SiGe comparison couples have been fabricated and are now being prepared for initial testing. It is believed that the back-to-back comparison couple design, utilizing a common hot shoe with enclosed heater, will provide the basis for an accurate comparison of segmented versus all SiGe couple efficiency, (Conditions 6&7).

Data obtained on weight loss, resistivity, and room temperature Seebeck indicates that the InAs-GaAs material stability is satisfactory for operation at temperatures up to 550°C. The data reported in Table III provides a preliminary confirmation of operational stability on a short term basis, (Condition 8).

Preliminary tensile tests indicate that there is some deterioration in mechanical strength with life. This deterioration is concentrated in the InAs-GaAs shoe bond, and is believed to be due to the continued reaction of copper and/or silver with InAs-GaAs during life. Bonds utilizing alloys other than copper-silver are now being investigated. The bond contact resistance does appear to be stable with life, (Conditions 9, 10 & 11).

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Microsections of segmented legs after bonding indicate that both the InAs-GaAs and SiGe are free of cracks. The ductile metal segment has sufficient compliance to reduce thermal expansion mismatch stresses generated in the cool-down from bonding temperature. The bond cooling, and cooling from isothermal tests at 550°C, (two hours), were the only specific thermal tests performed during Phase I. Therefore, insufficient information is available to judge the ability of the segmented couple to withstand thermal shock or thermal cycling, (Condition 12).

The major milestones, to be reached in the near future will be:

- 1) The initial test and evaluation of the optimized couple. This couple, and its counterpart, the all SiGe comparison couple, should be in operation by March 1, 1966.
- 2) A stable joint with respect to mechanical properties.

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APPENDIX

This appendix provides the actual life test data obtained on isothermal tests currently in process. The data presented indicates the status as of March 1, 1966.

INDEX TO TESTS

<u>Test No.</u>	<u>Type of Specimen</u>	<u>Page No.</u>
	Data Code	
21	IGA Bonded Element - Sealed Tube	40
22	IGA Bonded Element - Sealed Tube	41
24527-66	IGA Bonded Element - Sealed Tube	42
24527-61	IGA Bonded Element - Sealed Tube	43
54	IGA - SiGe Segmented Leg	44
62	IGA - SiGe Segmented Leg	45
64	IGA - SiGe Segmented Leg	46
65	IGA - SiGe Segmented Leg	47
78	IGA Bonded Element plus Segment	48
58	IGA Bonded Element plus Segment	49
55	IGA Bonded Element	50
57	IGA Bonded Element	51
79	IGA Bonded Element	52
56	IGA Bonded Element	53
73	IGA Bonded Element	54
74	IGA Bonded Element	55
75	IGA Bonded Element	56
80	IGA Material Only	57

Tests are arraigned in order of decreasing life hours for each type of specimen. Only those tests exceeding 500 hours have been included.

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THERMOELECTRIC DATA CODE

LIFE TEST SPECIMEN DATA POINTS

The following illustration indicates the location of the various resistance data points tabulated in the raw life test data. Seebeck surface probes were made at the mid-point on the elements

- NOTES:
1. All resistance data obtained with Keithley 503 Milliohmmeter. All values reported in milliohms, at room temperature.
 2. Surface Seebeck data obtained with Cambridge Systems Model #112B Thermoelectric Power Probe. All values reported in $\mu\text{V}/^\circ\text{C}$, at room temperature plus approximately 20°C .
 3. Weight data obtained with Fisher Scientific Company Gram-atic Balance, all data reported in grams.

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SPECIMEN TYPE IGA Element Only - Sealed Tube Test

TEST TEMPERATURE 550°C IGA VOLUME - cc IGA DOPING Se

[illegible]

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SPECIMEN TYPE IGA Element Only - Sealed Tube Test

TEST TEMPERATURE 550°C IGA VOLUME - cc IGA DOPING Se

[illegible]

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LIFE TEST NO. 24527-66SPECIMEN TYPE IGA Element Only - Sealed Tube TestTEST TEMPERATURE 550°C IGA VOLUME - cc IGA DOPING Se

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0					.11	.01	.01			-	2.5037
764					.17	.02	.04			-	-
* 1364					.175	.03	.02			-	-
1394					.17	.03	.02			-	2.5034
1601					.16	.03	.03			-	2.5024
1864					.16	.03	.01			-	2.5002
2364					.18	.03	.04			-	2.4993
3364					.18	.03	.02			-	2.4988
4372					.18	.05	.03			-	2.4985
5380					.18	.06	.03			-	2.4985
6380					.17	.03	.03			76.5	2.4975
7380					.24	.05	.05			74.3	2.4965

*NOTES: Life Tested at 500°C for First 1364 Hours

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LIFE TEST NO. 24527-61SPECIMEN TYPE IGA Element Only - Sealed Tube TestTEST TEMPERATURE 500°CIGA VOLUME - ccIGA DOPING Se

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0					.28	.03	.05			-	-
760					.30	.06	.05			-	-
1524					.27	.05	.05			-	-
2124					.29	.05	.04			-	-
2868					.29	.04	.04			-	-
3370					.29	.03	.03			-	-
4370					.29	.02	.05			-	2.4372
5370					.28	.02	.03			-	2.4365
6370					.29	.06	.07			-	2.4365
7370					.24	.05	.05			-	2.4355
8370					.26	.03	.03			66.3	2.4360
9370					.25	.02	.02			69.7	2.4342

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LIFE TEST NO. - 54 -

SPECIMEN TYPE Bonded IGA Element, Segment and SiGe *TEST TEMPERATURE 550°C IGA VOLUME .354 cc IGA DOPING Se

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0	.95	.11	.07	0	1.6	.15	.05	2.5	145	57.0	12.370
118	1.35	.15	.17	0	1.6	.23	.11	3.0	171	74.7	12.370
277	1.50	.20	.19	0	1.6	.11	.09	3.1	128	61.0	12.357
388	1.50	.11	.12	0	1.6	.10	.09	3.2	175	31.0	12.3120 **
547	1.70	.25	.25	0	1.6	.30	.07	3.35	185	51.0	12.3100
687	1.60	.20	.13	.06	1.7	.10	.15	3.35	184	46.0	12.3070
	AT THIS POINT THE SEGMENT TO BARE TUNGSTEN SHOE BOND FAILED										
837					1.78	.09	.15			26.2	5.6182
998					1.73	.09	.14			25.5	5.6173
1164					1.78	.02	.02			55.4	5.6165
1329					1.80	.02	.02			93.0	5.6163
1564					1.80	.10	.05			38.0	5.5983

*NOTES: This was an attempt to utilize a copper-silver braze to a bare tungsten shoe on the SiGe**NOTES: Sandblasted

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IGA Element, Segment and SiGe Element *

550°C

IGA VOLUME

- CC

IGA DOPING

Se

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Segment brazed to bare tungsten shoe with copper-titanium

SPECIMEN TYPE IGA Element, Segment and SiGe Element *

TEST TEMPERATURE 550°C IGA VOLUME .506 cc IGA DOPING Se

[illegible]

*NOTES: Segment brazed to bare tungsten shoe with copper-titanium

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IGA Element, Segment and SiGe Element *

550°C

IGA VOLUME

.394 cc

IGA DOPING

Se

17

LIFE TEST NO. 78

SPECIMEN TYPE IGA Bonded Element Plus Segment * _ _ _

TEST TEMPERATURE 550°C IGA VOLUME .514 cc IGA DCPI C Se _ _ _

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0					1.07	.03	.02			131.0	-
118					.92	.02	.015			125.0	-
277					1.05	.01	.01			92.0	-
388					1.05	.01	.01			83.0	-
547					1.05	.01	.01			128.0	-
687					1.00	.01	.01			117.0	10.6275
837					1.10	.01	.02			133.0	10.6223
998					1.08	.03	.03			124.0	10.6194
1164					1.10	.03	.03			133.0	10.6180
1329					1.15	.03	.03			130.0	10.6162
1564					1.15	.10	.10			110.5	10.6132

*NOTES: Copper-silver brazed segment to nickel plated tungsten

SPECIMEN TYPE IGA Element Plus Segment

TEST TEMPERATURE 550°C IGA VOLUME .507 cc IGA DOPING Se

[illegible]

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SPECIMEN TYPE IGA Element Only

TEST TEMPERATURE 550°C

IGA VOLUME .508 cc

IGA DOPING Se

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0					.80	.02	.05			82	-
118					.80	.04	.06			107	4.6480
277					.80	.01	.01			118	4.6470
388					.80	.02	.02			67	4.6470
547					.80	.02	.02			107	4.6460
687					.86	.07	.07			104	4.5805 *
837					.87	.08	.08			134	4.5802
998					.89	.08	.08			133	4.5800
1164					.91	.02	.05			89	4.5798
1329					.93	.03	.06			116	4.5802
1564					.92	.03	.05			116	4.5802

*NOTES: Sandblasted

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LIFE TEST NO. 57

SPECIMEN TYPE IGA Element Only

TEST TEMPERATURE 550°C IGA VOLUME .514 cc IGA DOPING Se

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0					1.10	.07	.02			58	5.1390
159					1.05	.01	.01			78	5.1390
271					1.05	.01	.01			95	5.1380
430					1.05	.01	.01			128	5.1350
571					1.05	.01	.01			84	5.0635 *
721					1.09	.01	.05			125	5.0632
882					1.07	.02	.05			143	5.0630
1048					1.08	.02	.05			108	5.0624
1213					1.00	.02	.05			109	5.0626
1448					1.10	.03	.03			114	5.0625

*NOTES: Sandblasted

SPECIMEN TYPE IGA Element OnlyTEST TEMPERATURE 550°CIGA VOLUME .410 ccIGA DOPING Se

LIFE HOURS	SiGe RESISTANCE			SEGMENT R ₂	IGA RESISTANCE			OVERALL R ₄	SEEBECK		OVERALL WEIGHT
	R ₁	R _{1a}	R _{1b}		R ₃	R _{3a}	R _{3b}		SiGe	IGA	
0					2.55	.07	.05			141.0	-
118					2.60	.20	.22			144.0	-
277					2.55	.05	.02			149.0	-
388					2.20	.10	.15			149.5	-
547					2.50	.10	.05			174.0	-
687					2.55	.07	.08			102.0	4.4465
837					2.55	.06	.06			156.0	4.4462
998					2.55	.06	.07			163.0	4.4460
1164					1.88	.05	.05			161.0	4.4458
1329					2.70	.05	.05			154.0	4.4461
1564					1.40	.01	.01			154.5	4.4458

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IGA Element Only *

550°C

WGA WCLIME

IGA DOF INC

Se

[illegible]

*NOTES: A bond test using a copper-silver-phosphorous braze alloy was evaluated, note high contact resistance data

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IGA Element Only

IGA VOLUME .487 cc IGA DOPING

Se

[illegible]

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SPECIMEN TYPE IGA Element Only

TEST TEMPERATURE 550°C IGA VOLUME .500 cc IGA DOPING Te

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SPECIMEN TYPE IGA Material Only

TEST TEMPERATURE 550°C IGA VOLUME .450 cc IGA DOPING Se

[illegible]