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NYOO-3584-3

C-92a, 92b, 93b

M-3679 (44th Ed.)

MASTER

AEC Research And
Development Report

Phase II-A
**COMPACT
THERMOELECTRIC
CONVERTER (U)**
Quarterly Progress Report
(April 1 - June 30, 1966)

UNCLASSIFIED

July 14, 1966

Westinghouse Astronuclear Laboratory

P.O. Box 10864 Pittsburgh, Pa. 15236

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AEC Contract AT(30-1)-3584

Phase II-A

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THERMOELECTRIC
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Prepared For

U. S. Atomic Energy Commission

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PREFACE

This Quarterly Report is submitted in response to the terms of Atomic Energy Commission Contract AT (30-1)-3584, Phase II-A, Compact Thermoelectric Converter Program. The Phase II-A Program, which spans from January 1 through December 31, 1966, is being conducted by the Astronuclear Laboratory of the Westinghouse Electric Corporation under the direction of the Atomic Energy Commission.

This second Quarterly Report for Phase II-A presents the significant progress on the Compact Thermoelectric Converter Program for the period from April 1 through June 30, 1966. Also included are the pertinent results of certain other programs being performed in parallel with this contract. Such items are noted as they are reported.

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INTRODUCTION AND SUMMARY

Program Objectives

The ultimate objectives of the Compact Thermoelectric Converter Program are to develop and qualify for space applications a reliable, high performance thermoelectric module and, if required, other converter hardware for use in 1 to 20 kilowatt (electric) compact thermoelectric converters. These are to be used with radioisotope and SNAP reactor (uranium-zirconium hydride core) heat sources. Also, multimodule converter performance and reliability are to be demonstrated. To the extent possible, the converter will operate with hot (heat source) and cold (radiator) loops, and be compatible with loop components currently available or under development. The module will provide for maximum flexibility in system and mission integration. In addition, it should provide improved performance and reliability over the current state-of-the-art.

The complete program has been divided into four phases. Phase I, Engineering Study and Critical Component Evaluation, covered the period from June 28, 1965 through December 31, 1965. During Phase I, analyses and evaluations were performed to prepare a reference design which would meet the technical specifications. In addition, an aggressive experimental program was conducted to evaluate critical components. Three series of thermoelectric modules were fabricated and testing was initiated. Unique fabrication and processing problems were identified. In addition, Phase I provided data to be used in evaluating converter performance in potential space power systems. The results of the Phase I program were reported in AEC R&D Report NYOO-3584-1.

The objectives for Phase II, Module Development, are to perform detailed module design and analyses; to develop and evaluate module components, and fabrication and assembly processes; and to conduct development testing on module components and complete modules in order to evaluate performance, environment capability, failure modes, quality control and long-term behavior.

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The Phase II portion of the program is planned as three separate successive parts: Phase II-A, II-B and II-C. The objectives for each phase are as follows.

Phase II-A: Solution of Tubular Module Design Problem Areas. The analytical and experimental solution of the tubular module design problems identified in Phase I will be demonstrated. Module fabrication, component and process development, and testing will be performed, as required, with the specific purpose of achieving the following technical objectives.

- (1) Demonstrate significant progress toward the solution of the performance degradation problem.
- (2) Solve and demonstrate the solution to the BN cracking problem.
- (3) Demonstrate that modules can meet acceptance level shock and vibration specification.
- (4) Establish sufficient time versus temperature material behavior to support design work.
- (5) Show initial correlation between static electric heater tests and liquid metal loop testing.

Phase II-B: Tubular Module Development and Process Evaluation. Tubular module development will be completed during this phase of the program. In addition, the effects of process and design variables will be evaluated and components will be satisfactorily demonstrated.

Phase II-C: Satisfactory Demonstration of Prototype Tubular Modules. Module design, process and quality assurance procedures will be optimized and prototype modules will be fabricated and satisfactorily demonstrated.

Phase III will be concerned with the qualification of manufacturing, processes and the tubular module, while Phase IV will be directed toward prototype compact converter fabrication, development testing and preliminary qualification.

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Progress for Second Quarter

The following tabulation presents the status of tubular thermoelectric modules (TEMs) involved in development activities during this second quarter of Phase II-A.

<u>Series</u>	<u>Serial Nos.</u>	<u>Status as of 6-30-66</u>
TEM-1	1, 2 ✓✓	Performance tests continuing (approximately 6400 hours on each unit).
	4 ✓	Performance test terminated at 5700 hours; destructive examination started
TEM-3*	1 ✓	On loan to JPL; performance tests continuing
TEM-4A	1 ✓	Performance test continuing (approximately 1600 hours)
	2 ✓	Destructive examination completed
TEM-5	1 ✓	Destructive examination completed
TEM-6	1 ✓	Destructive examination completed
	2 ✓	Performance test (38 hours) and destructive examination completed
TEM-7	1 ✓	Performance test continuing (approximately 1300 hours)
	2 ✓	Performance test (382 hours) and destructive examination completed
TEM-7A	1 ✓	Being held for performance test when test stand is available
TEM-8*	1, 2 ✓✓	Performance test started in June
TEM-8A*	1, 2 ✓✓	Performance test continuing (approximately 900 and 1100 hours, respectively)
TEM-8B*	1 ✓	Non-destructive examination in process

* From other programs

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<u>Series</u>	<u>Serial Nos.</u>	<u>Status as of 6-30-66</u>
TEM-9	1, 2	Non-destructive examination in process
	3	Destructive examination in process
TEM-9A	1	Being assembled
TEM-9B	1	Being assembled

Based on a review of program developments through May, the number of TEM-9-type modules on order was increased from 4 to 15. This series of modules will be used for the systematic investigation of materials and processing variables in the Phase II-A program for solving the boron nitride cracking and electrical shorting problems.

The procurement of special test equipment for the new thermoelectric module test data acquisition system is proceeding. It is expected that all parts will be received by late July. Installation work has started on the new, individual, static test stands which complement the new data acquisition system.

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I. PROGRAM MANAGEMENT

To date, the major portion of the Phase II-A effort, including the thermoelectric module tests, has been concentrated on achieving an understanding of the boron nitride cracking and electrical shorting problems. While these problems have not been fully resolved, significant mechanical design improvements have been demonstrated and incorporated into the TEM-9 series module. On this basis, a decision was made to defer any further mechanical redesigns and to use the TEM-9 design for a materials and processing variables investigation. Accordingly, the quantity of TEM-9 modules on order was increased from 4 to 15. The additional modules would be used to systematically explore processing variables which might cause boron nitride cracking and electrical shorting. The general development plan was defined at the close of May, discussed with representatives of the Atomic Energy Commission and implemented immediately thereafter.

The new Westinghouse-funded test stands, designed to supplement existing capabilities and to complement the new data acquisition system funded by the AEC, should be checked out in July. The equipment for the new data acquisition system is being expedited, and delivery is expected to be completed by the end of July.

AEC Schedule 139a's for the planned Compact Thermoelectric Converter Program were prepared and submitted (WANL-PP(CC)-004, dated April 29, 1966). These embodied estimates for fiscal year 1968, and a review of fiscal year 1967 requirements and fiscal year 1966 experience.

Two technical papers were presented at the joint AIAA/AIEE meeting in Washington, D. C. on May 17, 18 and 19, 1966. These papers are documented in WANL-TME-1429, The Development Status of Tubular Thermoelectric Modules.

The First Quarterly Progress Report for the Phase II-A Compact Thermoelectric Converter

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Program was issued as AEC R&D report NYOO-3584-2, and distributed per C-92a, C-92b, and C-93b of M-3679.

The monthly progress letters for April and May were issued on schedule as WANL-PR(CC)-010 and -011, respectively.

WANL-TME-1443, "Failure Cause Analysis of a Tubular Thermoelectric Module," was issued at the end of June.

WANL-TME-1446, "The Investigation of the Penetration of 3N PbTe in 'Grade A' BN", was completed but final publication has been deferred until July.

The scope of work for Subtask 3.1.1-Stress Analysis of the Project Plan was increased to include the further development of the tubular thermoelectric module stress code.

As a result of the program changes noted above, the logic of the PERT Summary Network was revised at the end of May. The updated PERT Summary Network for the program is presented in Appendix A. A summary of PERT network events scheduled for completion in April, May, and June is given in Tables A-1, -2 and -3, respectively. The status of major program milestone events is presented in Table A-4.

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II. RELIABILITY AND QUALITY ASSURANCE

A. RELIABILITY

Work on the reliability optimization of the compact thermoelectric converter system was continued during the second quarter. Simplified computer techniques were developed to carry out the calculations for a reliability comparison of various converter arrays. To date, the following alternatives have been explored:

1. Circuits: four 7-volt thermoelectric modules in series; four 28-volt thermoelectric modules in parallel; and a series/parallel array of two pairs of 14-volt thermoelectric modules.
2. Diodes: reliability levels of 0.990 and 0.999; and the case of no diode required.
3. Converter power ratings: 1, 5, 10 and 20 kilowatts.
4. Converter reliability: 0.990 and 0.999.
5. Spare power installed: 0 to 5 kilowatts per converter, depending on rating and reliability.

Thermoelectric module reliability is treated as a dependent variable, with values from 0.990 to 0.999 being of special interest. For purposes of this phase of the parametric study, each converter module was assumed to be rated at a 0.5-kilowatt output.

The more recent computer studies considered higher powered, more complex arrays. For example, it is now possible to compare the reliability data for a 5-kilowatt converter, presented in the first quarterly report, with the corresponding data for a 20-kilowatt converter derived during this period. Table 2-1 presents the results of evaluations on the 5-kilowatt converter array, and Table 2-2 shows the results for the 20-kilowatt converter array. Both arrays have the same overall reliability goal of 0.990 and, nominally, two spare converter modules.

In comparing the data, it is evident that having a large number of branches by using 28-volt modules is advantageous for both converters, especially if diode reliability is relatively low

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TABLE 2-1

Required Thermoelectric Module Reliability for a 5-Kilowatt
Compact Converter with 0.999 Reliability

<u>Case</u>	<u>No. of Converter Modules</u>	<u>Configuration</u>		<u>Required Module Reliability</u>		
		<u>Other</u>	<u>7 Volts</u>	<u>14 Volts</u>	<u>28 Volts</u>	
A	10	Series array; no diodes	0.99998	0.99998	0.99998	
B	12	Redundant array with diodes having 0.990 reliability	0.99820	0.98807	0.95542	
C	12	Redundant array with diodes having 0.999 reliability	0.99594	0.98361	0.94682	
D	12	Redundant array with redundant short protection in module; no diodes	0.99569	0.98312	0.94587	

TABLE 2-2

Required Thermoelectric Module Reliability for a 20-Kilowatt
Compact Converter with 0.999 Reliability

<u>Case</u>	<u>No. of Converter Modules</u>	<u>Configuration</u>		<u>Required Module Reliability</u>		
		<u>Other</u>	<u>7 Volts</u>	<u>14 Volts</u>	<u>28 Volts</u>	
A	40	Minimal array; no diodes	0.99999	0.99999	0.99999	
B	42	Redundant array with diodes	*	*	0.99505	
B'	43	having 0.990 reliability	0.99997	0.99595	0.98226	
C	42	Redundant array with diodes having 0.999 reliability	0.99907	0.99609	0.98609	
D	42	Redundant array with redundant short protection in module; no diodes	0.99882	0.99559	0.98510	

* Impossible condition - required module reliability greater than 1.0

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(0.990). In the case of the 20-kilowatt converter, it is impossible to attain the specified reliability of 0.999 using arrays of 7- or 14-volt thermoelectric modules, since the required module reliability would have to exceed 100%. However, if another spare converter module (Case B') were added, any array could be used. There is no doubt that converter reliability levels can be raised - or module reliability requirements can be relaxed - by judicious trade-offs among converter module configuration, installed converter redundancy, and reliability of available diodes or equivalent disjunctive devices. It is essential, however, to weigh converter reliability gains against undesirable increases in overall system requirements such as mass, volume and complexity of heat source, heat sink, mechanical components, and piping. This work is being documented and a formal report on the reliability apportionment and optimization work will be submitted.

The results of the failure cause study performed during the first quarter were discussed with members of various technical groups, and the failure cause diagrams were updated. These diagrams depict all currently conceivable failure modes of a thermoelectric module, together with the individual chains of causative defects and conditions responsible for the failures. Parts of the study were modified to account for recent design changes, to improve the terminology, to clarify graphic presentation, and to delete several failure modes now considered incredible. The revised report, currently being issued as WANL-TME-1443, will serve as a guide for future development work. An important value of the failure cause diagram is that it draws attention to all conceivable failure modes and their causes, rather than focusing only on problems of current concern.

Toward the end of the reporting period, work was initiated on a program review of designs, materials, tests and inspections of all thermoelectric modules produced to date. The primary purpose of this effort is to interpret, summarize and arrange existing data in a format that will make it more readily accessible for evaluation.

Work on the thermoelectric module qualification test plan, which was discussed in the first quarterly report, has been deferred to permit the work described in the foregoing paragraph. It is expected that the qualification test plan will be further developed during the next quarter.

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B. QUALITY ASSURANCE

The major effort of Quality Engineering has been directed toward the expansion and completion of the unfinished objectives of the detailed Nondestructive Testing (NDT) Program Plan developed in the first quarter of Phase II-A, and shown in Figure 2-1. In addition, a survey was initiated in preparation for the accumulation of all data relative to thermoelectric module material and source lot identification, assembly and processing procedures, and testing results.

Nondestructive Testing

Ultrasonic methods

Ultrasonic methods, using shear-wave techniques for detecting discontinuities in the inner electrical insulating sleeves, were demonstrated on module assemblies TEM-3 S/N-5 and S/N-6, and TEM-7A S/N-1. Signal amplitudes may be reduced to remove extraneous electrical noise and give a detectable response for major cracks or discontinuities in the boron nitride sleeve next to the inner clad. The reduction in the signal amplitude, to monitor the major imperfections only, eliminates the opportunity to gain additional information. Full amplitude permits the measurement of spacing of interfaces or fit-up of component parts adjacent to the inner clad, the relative soundness of the Alsimag, electron beam welds, Inconel X-750 structure, and areas of poor acoustical transmission (low contact pressures). However, at full amplitude, the signals from tight cracks in the inner boron nitride insulating sleeve are quite weak.

Additional experience using the shear-wave technique has not been accumulated during this reporting period because design effort has been essentially devoted to the fabrication and testing of a series of modules which do not lend themselves to application of this technique.

The through transmission technique using dual sending and receiving transducers, described in the first quarterly report, was further refined during the second quarter. Evaluation of this technique indicates that it yields less information than that provided by the single-crystal shear-wave method.

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Figure 2-1. Non-Destructive Testing Program Plan

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Microwave testing

Boron nitride with inclusions and tight circumferential cracks was tested using microwave techniques. The inclusions appear to be minute graphite flakes, transparent to x-ray detection. Thin wall tubing with circumferential and axial cracks, visible to the eye, were also used to determine the capability of microwave noncontacting inspection. The microwave techniques would not resolve either of the above anomalies.

Radiation

Low voltage x-ray was used to determine the nature of small black particles noted in Grade A boron nitride, supplied by the Carborundum Company. These particles were quite different from those inclusions found in Carborundum's Grade H.P. material. The density of the inclusions in the Grade A material apparently match that of the boron nitride matrix and are not detectable by x-ray. The inclusions in the Grade H.P. material have a higher density than the boron nitride matrix and are easily detected by x-ray. Grade A has been used exclusively in the Compact Converter Program.

Electrical noise measurement

A Quan-Tech Model 327 noise analyzer, developed specifically to measure the electrical noise generated within semi-conductor diodes, was made available for experiments on assembled modules. Thermoelectric circuitry electrical characteristics, as well as circuit-to-clad shorting, should develop discrete noise signals as various test voltages and current are supplied to the module circuit. These noise signals may be indicative of operational performance of the thermoelectric module. The noise range that may be measured with Model 327 is 0.03 to 1 millivolt RMS full scale (for $\Delta f = 1$ cycle). The noise voltage is monitored by front panel meters at 10 cycles and 1 kilocycle simultaneously, as well as a broadband noise voltage jack for oscilloscope presentation. This instrument will automatically sweep the applied voltage and/or current over a wide range of direct-current operating conditions. Test voltage is supplied over a range of 0 to 500 vdc. Test current may be metered in over a range from 0 to 100 milliamperes. Impedance of the module circuit may be measured in a range from 0 to 300 ohms.

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No test data have been made available using Model 327 for evaluating thermoelectric modules, because the instrument was undergoing repairs during this reporting period.

Infrared radiometry

Preliminary infrared scans of modules, using a Barnes Radiometer and Camera, gave single line scans and thermograms with less than acceptable definition of the location of internal electrical shorts. When the infrared equipment becomes available, experiments are planned to determine the effects of cooling of the inner clad during infrared scanning to partially prevent the dissipation of the heat source. Another experiment will involve a method of supplying sufficient current through the thermoelectric circuit or across the electrical shorting path to provide localized instant heat that may be detectable by infrared. To differentiate between radiometer system noise and true infrared hot spots where the ΔT is 1°C , a thermoelectric module without internal electrical shorts will be used. This will then be compared with a module having definite shorting problems.

Materials, Processing, and Testing Review

Quality Engineering is participating in a program review of Phases I and II-A documentation to develop an orderly summary of the data and to achieve a full understanding of the significant results obtained in the program to date. This documentation includes design drawings, assembly and processing procedures, nondestructive and module operation test results, destructive examination photographs, and materials and source lot records. To assist in this program, Quality Engineering has prepared flow charts for all modules, showing the chronological and sequential events in assembly, processing and testing. A similar chart was prepared giving the lot number and source of all materials used in the module assemblies.

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- OUTER CLADDING
- INNER CLADDING
- THERMOELECTRIC MATERIAL
- ELECTRICAL INSULATION
- ELECTRICAL CONDUCTOR

DESIGN

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III. DESIGN

A. MECHANICAL

During the first five months of the Phase II-A Compact Thermoelectric Converter Program, a number of modules were built, tested, and destructively examined to gain an understanding of the effect of design and process variations upon the performance and structural integrity of the tubular module assembly. In the experiments performed on these modules, the major effort was concentrated on resolving the problems of boron nitride cracking and module shorting by modifying the design and the processing steps. These changes or iterations in design and processing were the result of analysis and destructive examination of preceding modules. While the problems have not been fully resolved, significant mechanical design improvements have been demonstrated and incorporated in the TEM-9 series of modules. These improvements, and their basis in analysis and experiment, are described below.

Because it was felt that the mechanical design was becoming reasonably well understood, it was decided to hold the TEM-9 design constant over a number of units while processing, and constituent materials were separately varied to develop a further understanding of electrical shorting. The purpose of TEM-9, therefore, is to serve as a mechanical vehicle upon which the effects of process and material variations on module life and performance can be investigated. In the following paragraphs, several of the recent designs leading up to the TEM-9 configuration will be reviewed, and the TEM-9 design will be described in detail.

TEM-7

The TEM-7 module experiment was intended primarily to investigate improved performance and reduced degradation rates beyond the TEM-5 and -6 devices through increased contact pressure at internal thermal interfaces. In addition, the design includes a radially flexible end similar to that designed for TEM-5, and a metal membrane for electrical isolation of the thermoelectric circuit similar to that tested in TEM-6.

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To compensate for the increased internal pressure, the thicknesses of both the inner and outer clads were increased. The ends of the TEM-7 module (as shown in Figure 3-1) were redesigned to reduce thermal losses below those expected for TEM-5. A new conductor sleeve, insulated thermally from the inner clad, was provided. This sleeve is held in a positive axial location by the axial restraint members welded into the outer clad. As in TEM-5, the metal ends are designed to be sealed against boron nitride sleeves by the external pressure imposed during cold and hot autoclaving. Thus the inner and outer clads of TEM-5 and -7 (unlike those for TEM-1, -2, -3, -4, -6 and -8) are electrically isolated from one another.

One TEM-7A module experiment was also designed and built. It is identical to the TEM-7 units, except that the TEM-7A module has two concentric boron nitride sleeves (11 and 14 mils thick) as electrical insulation between the inner clad and the inner conductor rings in place of the boron nitride/metal sleeve arrangement in TEM-7.

TEM-8 and -8A *

The TEM-8 and -8A modules are similar to Phase I modules in the design of the end regions beyond the active thermoelectric stack. The TEM-8 module is similar to TEM-7 in its use of a boron nitride/416 stainless steel/boron nitride sandwich insulator in the region between the inner clad and the inner iron conductor rings. TEM-8A, on the other hand, has two concentric boron nitride sleeves in this same region, as was used in TEM-7A. In both designs, the heavy wall inner and outer clads associated with the TEM-7 series were used.

The TEM-8 and -8A modules were processed through a standard gas-purging cycle at 400°C. After processing, they were cold autoclaved at 50,000 psi and 45,000 psi, respectively, then hot autoclaved at 650°C and 15,000 psi. A uniform radial reduction of 0.020 inch was experienced over the thermoelectric couple area with a characteristic lesser reduction in diameter at the ends over the retaining rings.

* From another program.

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① ACTIVE THERMOELECTRIC MATERIAL	⑤ BORON NITRIDE ELECTRICAL INSULATION
② INACTIVE THERMOELECTRIC MATERIAL	⑥ IRON CONDUCTOR SLEEVE
③ ALSIMAG FILLERS	⑦ ALSIMAG THERMAL INSULATION
④ AXIAL RESTRAINT MEMBER	

Figure 3-1. Design Features of TEM-5 and TEM-7 Modules

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The 650°C temperature was selected because capsule and component testing showed that, at this temperature, the lead-rich material exhibited little penetration into the boron nitride. The 15,000-psi pressure was selected to fully densify the lead telluride. This pressure imposes the same compressive stress on the outside diameter of the lead telluride during sintering as had been imposed on lead telluride in thin-walled capsule tests at 10,000 psi and 650°C, during which full densification of the lead telluride was obtained.

After processing, room temperature testing of the TEM-8 units, coupled with initial hot test results from the TEM-8A devices, lead to the suspicion that incomplete densification had occurred during the processing. The reasons for this are still being investigated. A decision was reached to pressure-sinter the TEM-8 modules again at 750°C and 10,000 psi. After the second sintering operation, the module outside diameter had been reduced an additional 2 mils, verifying that initial densification was incomplete. The TEM-8 modules have been placed on hot-test, with results as reported in Section VI of this report.

TEM-9

The TEM-9 series of modules are of a 38-thermoelectric couple design with a pin arrangement for bringing the electrical circuit out from each end (Figure 3-2). The limit of 38 couples was based on use of 8-inch long cylinders of boron nitride. While some of the module experiments included boron nitride sleeves machined from longer cylinders, the variability in properties of that material is not as well understood as that of the standard 8-inch cylinders.

To create more room in the radial direction for increased thickness of boron nitride insulating sleeves, the outside diameter of the inner clad was specified as 0.650 inch, and the inside diameter of the outer clad was increased to 1.407 inches from 1.377 inches. This implied a sacrifice of contact pressure and resistance to relaxation; however, it was deemed

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TEM-8

TEM-9

- 1 THERMOELECTRIC MATERIAL
- 2 ALSIMAG FILLERS
- 3 RETAINING RING
- 4 BORON NITRIDE ELECTRICAL INSULATION
- 5 IRON CONDUCTOR
- 6 STAINLESS STEEL BARRIER

Figure 3-2. Design Features of TEM-8 and TEM-9 Modules

3-5

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worthwhile on this series of modules to improve the integrity of the electrical insulation without significant tooling changes.

The thermoelectric couples are formed from standardized pairs of n- and p-type washers, mica insulating rings, inner and outer iron conductor rings, and a boron nitride ring. Since TEM-1, a gradual design evolution has taken place in the thermoelectric couple components. These changes will be discussed in the following paragraphs.

The post-mortem analysis of Phase I modules indicated that the mica insulating rings were being driven into the inner boron nitride insulating sleeve during swaging, and possibly contributing to cracking of the sleeve. To alleviate this problem, the dimensions of the mica insulating rings were changed by an amount calculated to completely eliminate the interference with the boron nitride, at some sacrifice in insulating characteristics. However, sectioning of modules with this change incorporated showed that the electrical insulation was even less effective than expected; p- and n- materials were in contact over perhaps 20% of their width. In addition, because the mica insulating rings were no longer accurately positioned in the radial direction, sidewise slippage of the insulating rings occasionally occurred during assembly. As a result, a few iron conductor rings were not properly separated, although the mica still contacted the boron nitride at localized points. Accordingly, in the TEM-8B module experiment and, subsequently, in the TEM-9 design, the mica insulating ring dimensions were changed such that the large and the small insulating rings were radially positioned by the inner and outer iron conductor rings, respectively. The diameters, which could possibly contact the inner or outer insulating sleeve, were redimensioned on the basis of information gained on the non-linear radial deformation behavior of the mica insulating rings from earlier module experiments. These latest design modifications are expected to solve the electrical separation problem and still eliminate one potential contributor to cracking of insulating sleeves.

The outer iron conductor rings have been unchanged since TEM-4. The post-mortem examination of TEM-1, -2 and -3 modules indicated axial gaps between the conductor rings and

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the mica insulating rings. As a result, the length of the conductor rings was increased 0.001 inch, and the rings were selectively assembled to assure proper stack-up of couple components to minimize access of lead or lead telluride to the boron nitride insulation. In a similar manner, the length of the inner iron conductor rings was increased. In addition, the radial thickness of the inner iron rings has been reduced. The earlier modules had a 0.050-inch thick wall inner iron conductor ring. Destructive examination of these modules indicated that there was a high probability of crack occurrence in the inner boron nitride insulating sleeve adjacent to the joint between neighboring iron rings. This same effect was not noticed in the outer boron nitride insulating sleeve where the iron conductor rings were much more flexible. Therefore, the inner conductor rings, starting with the TEM-6 module experiment, were decreased in wall thickness. A corresponding decrease was noted in the frequency of cracking of the inner insulating sleeve. The TEM-8B and -9 designs incorporate an additional design refinement in the form of a boron nitride ring (concentric with the inner iron conductor ring) to act as a cushion for the inner insulating sleeve.

In addition to isolating the iron conductor ring from the boron nitride insulating sleeve, these boron nitride rings form chambers into which the mica insulating rings extend radially, providing complete axial separation of adjacent iron rings. This feature was selected because of observations which showed that intercouple shorting could be due to lead-rich material dispersing axially around the edges of the mica insulating washers and interconnecting adjacent iron rings.

The outer insulating sleeve in the TEM-1 design consisted of a free cylinder which was slid over the thermoelectric couples during the assembly of the module. To prevent fracture of this loose sleeve during initial handling, purging, and cold compaction of a module, the sleeve was redesigned to be shrink-fitted into the outer cladding. This design modification produced two desirable results: (1) the insulating sleeve was placed in compression immediately with a very small probability of cracking due to tensile stresses and (2) it removed one assembly gap, thereby lessening the radial reduction required during module processing. To prevent cracks that have been occasionally observed in the outer boron nitride sleeve, and

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to provide improved resistance to shorting because of penetration of lead-type material into the boron nitride, the TEM-9 outer insulating sleeve was made 0.037-inch thick, an increase of 0.015 inch over previous designs.

The inner insulating sleeve has been the focal point for most of the design modifications during Phase II-A. In the early designs, a single cylinder of boron nitride was utilized, and the cracking which occurred evidently had several causes. Shorting occurred to the inner clad due to a metallic conducting path extending from the couple area down through cracks or porosity to the outside diameter of the inner clad. Design modifications were coupled with processing changes to find a solution to the cracking and shorting problems. In general, the solutions can be grouped into the following categories:

1. Seal the lead telluride in place so that it cannot contact the insulating sleeves.
2. Eliminate the cracking of the boron nitride sleeves.
3. Provide additional layers of insulation between the thermoelectric material and the inner clad.
4. Vary the processing cycle or the method of application of pressure during the process cycle.

The TEM-5 design incorporated a number of sealing schemes which included a delta seal, a ring seal, and multiple insulation layers. This module and TEM-6 were described in detail in the first Quarterly Progress Report, WANL-PR(CC)-009. TEM-6 incorporated a brickwork inner insulating layer, combined with a sandwich-type inner insulating sleeve consisting of a boron nitride inner cylinder, a thin Armco iron middle cylinder, and a boron nitride outer cylinder. The iron sleeve was a positive barrier incorporated to prevent lead telluride in the inner sleeve region from reaching the inner clad. However, the oxidation sensitive iron proved difficult to work with and introduced potential contamination problems, and it was decided to use a Type 416 stainless steel sleeve in any similar succeeding designs.

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The TEM-7 and -8 modules used the sandwich concept, but with no brickwork seals. The TEM-7A and -8A series of modules were designed with a double-wall boron nitride inner insulating sleeve. This design was based on post-mortem examinations of prior modules which indicated that cracks did not propagate through a boron nitride/boron nitride interface. The stainless steel sleeve was not incorporated into the TEM-7A and -8A designs because of the additional thermal impedance it introduced into the module.

The design of TEM-8B was quite similar to TEM-9, except that the wall thicknesses of the boron nitride sleeves were thinner. The TEM-9 inner insulating sleeve is of a single, heavy-walled boron nitride cylinder 0.040-inch thick. The inner boron nitride rings rest on this cylinder and provide a cushion for any radial forces generated during processing or operation of the module.

An inspection of TEM-7 S/N-2 and TEM-9 S/N-3, the two most recent modules to be destructively examined, revealed no cracking of the innermost boron nitride sleeve on either unit. Both units obviously suffered from shorts between couples, as well as from circuit to clad in the case of TEM-9. However, these shorts were due to permeation of the boron nitride with lead-like material, and not to mechanical failure. The fact that the TEM-9 boron nitride sleeve is relatively thick made it possible to discern slip- or shear-lines in the material, along which plastic deformation had occurred; however, there were no cracks of the type seen in earlier modules. This offers confidence that present module processing, in which iso-static compaction either precedes or replaces swaging, can be further developed to result in consistent production of modules with crack-free boron nitride.

The TEM-7 and -8 series of modules used a thicker outer clad to investigate the influence of increased interface pressures on module performance. Because the inside diameter of the TEM-9 outer clad was increased to allow for a heavier outer insulating sleeve, this series of modules has a thinner outer clad than that specified for the TEM-7 and -8 series of modules.

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The outside diameter of the outer cladding could not be increased to account for the inside diameter change because the first three modules in the TEM-9 series are to be processed in the same manner as TEM-4, which includes a swaging operation after room temperature compaction.

The TEM-9 inner clad is an Inconel X-750 cylinder with a 0.500-inch inside diameter and a 0.650-inch outside diameter. The ends have an extension of Type 316 stainless steel, which permits a stainless steel to stainless steel type of weld. Except for a 0.700-inch outside diameter specified for TEM-7 and -8 series of modules, the inner clad has remained relatively static in its concept and configuration from TEM-1 through TEM-9.

Effects of Processing on Design

Because variations in processing can effect module performance and longevity as profoundly as the mechanical modifications discussed above, considerable effort during the second quarter has gone into understanding and summarizing the processing history of the TEM-1 through TEM-8 modules.

The results of processing TEM-1 through TEM-8 modules are summarized in the following paragraphs, and the conclusions which have been drawn regarding TEM-9 processing are presented. In addition, the results of processing the first group of TEM-9 units are discussed.

The purging procedure has remained essentially unchanged from that specified for TEM-1. However, analysis of the relative thermal growth of module components indicated the likelihood of cracking the inner boron nitride sleeve by the inner clad. This was verified by examination of TEM-3 S/N-7 after purging. Hence, the clearance between these components was increased starting with TEM-4A and continuing with the TEM-7, -8 and -9 series.

The TEM-1, -2 and -3 modules were swaged from a 1.624-inch initial diameter to about a 1.597-inch diameter. It was observed that the inner clad was distorted on these modules

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because the retainers at either end were being displaced radially inward. Severe cracking of the boron nitride was noted in the region of inner clad distortion. Furthermore, the jarring of the module during swaging was felt to be a likely cause for general cracking and breakage of internal parts since the clearances needed for purging and assembly were still open. In addition, in spite of the inclusion of a tension-bar preloading the outer clad in compression, the module characteristically elongated about 0.060 inch during swaging. This distortion of the thermoelectric array, though undesirable, was unavoidable using the swaging process to remove void volume from the module.

A partial resolution of these problems was investigated during the processing of TEM-4 S/N-1, which was isostatically compressed in an autoclave at room temperature and a pressure of 40,000 psi to remove the assembly voids. The module was then swaged to reduce the stiffer ends to the same diameter as the middle region.

As a result of the TEM-4 experience, no swaging was used during processing of the TEM-4A, -5, -6, -7 and -8 modules. These modules were only isostatically compacted at 45,000 to 50,000 psi to remove voids before sintering. Comparison of TEM-7 and -8 units with earlier ones (Figure 3-3) shows the progress made in achieving compaction with minimal axial distortion of the devices. However, destructive examination of TEM-4A, -5 and -6 units still showed some breakage of boron nitride and other internal components, whereas TEM-9 S/N-3, which was swaged, was free of mechanical cracks.

TEM-1, -2 and -3 units were pressure-sintered at nominal autoclave conditions of 750°C and 10,000 psi. Short circuiting to clad through cracks in the boron nitride filled with lead-like material led to an attempt to densify the lead-telluride in TEM-4 at the lower temperature of 650°C to minimize the mobility of the lead-like impurity within the module. However, the unit did not fully densify, as can be seen from Figure 3-3. Return to the 750°C - 10,000-psi cycle for TEM-4A, -5, -6 and -7 continued to result in the migration of lead to the boron nitride sleeves. Thus, another attempt was made (on the TEM-8A modules) to sinter at 650°C but with a 15,000-psi pressure on the outer clad. Though better than the TEM-4 module,

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these devices apparently did not quite fully densify. The TEM-8 and -8B units, which were processed in the same manner, were later subjected to an additional 750°C - 10,000-psi sintering cycle to assure full density.

Late in the second quarter, it was discovered that the actual sintering temperatures of TEM-1 through TEM-8 modules were probably slightly lower than specified, because of a temperature difference between the modules and the points in the furnace at which temperature was measured. The first TEM-9 sintering cycle was controlled by module-mounted thermocouples, but the difference in temperature of the module from that of previous runs was not discovered until after the processing of TEM-9 S/N-1, -2 and -3. Thus, these three units were probably sintered at a somewhat higher temperature than the actual baseline temperature established earlier in the program.

In summary, it can be concluded that the purging process appears to be well in control. Cold compacted units can apparently be made crack-free, if isostatic compaction precedes mechanical swaging. Comparison of thermal impedances of modules on test also leads to the suspicion that swaging may give rise to higher final contact pressures between the inner clad and the boron nitride than isostatic compaction, because swaging forces a fixed displacement rather than a fixed load at the outer clad, and can result in a higher load on inner components under some conditions. In any event, swaging under carefully controlled conditions appears to merit further investigation. Another interesting concept is that of "warm compaction". If deformation of boron nitride can be done at elevated temperatures at which the material is reasonably ductile, there is less likelihood of developing cracks. Further, if the low expansivity boron nitride does not contact metallic parts, such as the iron conductors and the clads, until it is already near the peak pressure-sintering temperature, it will sustain little tensile strain in going to the top of the temperature cycle, and will be less likely to fracture. The temperature at which compaction may be done is limited by the onset of appreciable sublimation of constituents of the lead telluride mixture at some temperature above the 400°C purging temperature. Capsule experiments are being built to establish the true upper limit of this temperature. These experiments are described in the following paragraphs.

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Component Test Capsules

A capsule was designed, built and processed with the TEM-8 modules. The purpose of this capsule test was to evaluate a modified end retaining ring to improve the radial reduction occurring during processing. The flange of the end retainer ring was modified by milling out slots in the area between the collector pins, as shown in Figure 3-4. The results of this capsule test were compared against the radial reduction occurring on a typical module and also against the TEM-8B module which had special end retaining rings which were modified by drilling holes through the flange, which were then sealed with boron nitride plugs inserted into the holes.

An average 0.013-inch radial reduction in the flange area was obtained with the capsule. This is about a 50% increase in radial reduction when compared with a typical module, but still falls short of the goal, which is the average 0.021-mil radial reduction observed over the mid-region of a typical module. The TEM-8B module experienced about half the increase in radial reduction as that of the capsule.

The capsule was sectioned through the retaining ring area with no anomalies detected. The end cap and the Alsimag spacer were removed from the collector pins in an operation identical to that of opening a typical module. A helium leak test was performed on the boron nitride sleeves which are installed around the collector pins. The leak rate was low and comparable to that experienced in a typical module. The face of the sectioning cut-through end retaining ring was polished and examined. The component parts were examined with no unusual occurrences noted. The outside diameters of the boron nitride sleeves around the collector pins were found to be slightly oval, as was the case on typical modules.

Though progress has been made, it can be concluded that further efforts are required to establish an end retaining ring design which will reduce radially the same amount as experienced over the module active length, without detracting from its axial restraining function.

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Figure 3-4. Modified TEM-8 Flange and End Retainer Ring

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Thermal and Electrical Component Tests

Detailed planning for a program to obtain data on interface thermal impedance and electrical resistivity has been completed. These data are required for module analysis and design. The test program includes data on all material interfaces, and will concentrate initially on the boron nitride/iron and iron/lead telluride interfaces. The electrical resistivity measurements will be obtained from both test specimens and actual modules. The thermal conductivity of the various interfaces will be initially obtained from out-of-module samples.

Sublimation/Deposition Tests

These tests are being run to determine the highest temperature to which a module can be heated before compaction, without electrical difficulties caused by sublimation of lead telluride. The test module will consist of five couples with Alsimag washers at each end and uses standard spare couple parts. The test modules will be heated to various temperatures in excess of 400°C, and destructively disassembled and examined for evidence of sublimation.

All parts for five test capsules are available. Assembly, testing and post-mortem examination will follow.

Data Summary Efforts

The data collected on the Phase I and Phase II-A thermoelectric modules are being organized into a usable reference file. With the data organized in this manner, various parameters can be easily correlated for planning of future module design and processing, and to better understand the results of testing and examination of current units.

Assembly clearance studies for each module are complete. Curves which show dimensional changes resulting from various processing steps are in work. Information on the materials used in each module is being collected. A summary of destructive examination observations is also in work, as are summaries of room temperature and elevated temperature testing on each unit.

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IV. ANALYSIS

A. THERMAL

In the analyses of the tubular module design, an empirical value for the thermal conductances of the single layer boron nitride electrical insulation sleeves was used. This value lumps both interface and volume effects. However, the more complex configurations now being studied require modification to the established analytical techniques because empirical values for the thermal conductances of the multilayer insulation sleeves are not available. An approximate technique has been used to obtain an "effective" thermal conductance for the multilayer geometries. The CPBTE2 code has been modified to permit the input of separate values of effective thermal conductance for the inner and outer insulation layers.

B. THERMOELECTRIC

Calculational Technique

As part of the continuing effort to improve the accuracy of the theoretical thermoelectric performance calculations, the following changes have been incorporated into the CPBTE2 computer code.

1. A more exact method, suggested by Heikes and Ure*, is now being used to calculate the average values of electrical resistivity for the various lead telluride washers.
2. An appropriate calculation for Thompson heat has been included in the hot junction energy balance calculations.
3. A correction has been made in the Joule heat calculation.

* Heikes, R. R., and Ure, R. W., Jr., Thermoelectricity, Science and Engineering, Interscience Publishers, Inc., pp 500 and 501, 1961.

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Diagnostic Code

A code is being prepared to analyze load curve test data and to diagnose the causes of rapid degradation. The equations have been derived and partially programmed. The code is being debugged and the programming effort is continuing.

Analysis of TEM-8 and TEM-8A Designs*

Thermoelectric analysis, using the revised CPBTE2 code, has been performed on the TEM-8 and TEM-8A designs to predict their operation. These modules include multiple layers of material between the inner clad and inner conductor ring. The results of the analysis are shown in Table 4-1 for hot clad temperatures of 1000° and 1100°F, at a constant cold clad temperature of 400°F.

TABLE 4-1
Analysis of TEM-8 and TEM-8A

	TEM-8		TEM-8A	
Cold Clad Temperature (°F)	400	400	400	400
Hot Clad Temperature (°F)	1000	1100	1000	1100
Open Circuit Voltage (volts)	5.44	6.50	5.55	6.62
Load Current (amp)	32.1	34.8	32.4	35.1
Power Output (watts)	87.3	113.0	90.0	116.1
Power Input (watts)	1693	1943	1720	1972
Efficiency (%)	5.16	5.82	5.23	5.89

Data Analysis and Handling (DAHA)

TEM-3 S/N-1 data have been punched into IBM cards for input to the data handling code, as have the life test data for TEM-1 S/N-1, -2 and -4. This will provide a final check on the various modes of operation for this code as well as a mathematical check on most of the hand calculations performed to date on the test data. Data on future tests will be recorded directly on programming sheets so that DAHA can be used to keep a continuous record of the testing.

* From another program.

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In conjunction with DAHA, a plotting program is being written to permit any combination and permutation of test data (and information calculated directly from test data) to be plotted and listed whenever such information is desired.

Initial Warm-Up of Modules

Four modules (TEM-7 S/N-1 and -2, and TEM-8A S/N-1 and -2) were placed on test during the second quarter. Figures 4-1 and 4-2 are plots of the internal resistances and thermal impedances of the modules as functions of thermal power input during their initial warm-ups from room temperature to operating conditions of 1000°F on the inner clad and 400°F on the outer clad (average temperatures).

All of the modules show high room temperature thermal impedances (Figure 4-2) which decrease through minimums and then rise again to final high values at operating conditions. The thermal impedance is normally expected to increase by about a factor of three from the room temperature value to the value at the operating conditions mentioned in the previous paragraph.

Thus, the performance of the four modules is at variance with the expected performance. Such a variance can be accounted for by the existence of poor interfaces or contacts which slowly improve (due to differential expansion) during the early portion of the warm-up, until reasonably uniformly good contact is made. At this point the increase in thermal impedance follows qualitatively the expected form. The fact that the thermal impedance of TEM-8A S/N-1 is significantly higher than the other modules at operating conditions is not yet fully understood.

The internal resistances of the modules, as shown in Figure 4-1, display similar form between modules of the same type, but drastically different forms for the two unlike modules. The two TEM-8A modules start at an extremely high value, and then decrease rapidly as the temperature increases. On the other hand, the two TEM-7 modules start at a low value and increase

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Figure 4-1. Internal Resistance as a Function of Thermal Power Input During Initial Warm-Up

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Figure 4-2. Thermal Impedance as a Function of Thermal Power Input During Initial Warm-Up

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continuously. At the 20 watts/couple input level, the values of all four modules are within about 15% of each other and, thereafter, they increase together in a similar manner. The expected form of such curves would be an exponential increase with temperature from an initial value of about 0.3 to 0.6 milliohm/couple. The TEM-7 modules appear qualitatively to follow such a curve. This, coupled with the thermal impedance plots discussed above, indicates that the poor contacts which exist in the TEM-7 modules are probably external to the electric circuit of the module.

In TEM-8A S/N-2, the correspondence between the fall-off in internal resistance and thermal impedance indicates the likelihood that poor contacts exist within the electric circuit. Such a conclusion is suggested also for TEM-8A S/N-1, but, since the thermal impedance did not improve nearly as much as did the internal resistance, the interpretation must be that poor interface contacts exist both external and internal to the electric circuit.

As can be seen from the plots, more thermal power input was required (about 10%) to bring the TEM-7 modules to operating conditions than was required to bring the TEM-8A modules to the same conditions. This suggests the possibility that multiple intercouple shorts exist within the TEM-7 modules. Such shorts would cause the transport of additional heat across the module due to the Peltier effect. When TEM-7 S/N-2 was removed from test and destructively examined, multiple shorts between the couples and the iron sleeve were found over a large portion of the module.

TEM-8A Thermal Cycling*

Figures 4-3 and 4-4 show the effects of thermal cycling on the open-circuit voltage of the two TEM-8A modules. As can be seen, no detectable change occurred in this parameter as a result of a thermal cycle to room temperature and back to life test conditions. The small variations between the cool-down and heat-up curves of the cycle are to be expected as a result of variations in differential thermal expansions during the two processes. For comparison, the open-circuit voltages of the initial warm-ups are also plotted on these curves.

* From another program.

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Figure 4-3. TEM-8A S/N-1 Initial Heat-Up and Thermal Cycle

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Figure 4-4. TEM-8A S/N-2 Initial Heat-Up and Thermal Cycle

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Figures 4-1 and 4-2 have been presented as smooth curves for the purpose of qualitative comparison of the warm-up parameters for the various modules. However, point-wise plots of the actual data show incidences of severe deviation from such smooth curves. In some cases, the discontinuities can be directly related to unplanned perturbations in the operating parameters. Other discontinuities appear to indicate the making and breaking of contacts (both thermal and electrical) within the modules. More detailed examination will be necessary before assignment of conditions affecting each of the discontinuities can be made.

C. STRESS

The first quarterly progress report ⁽¹⁾ analyzed boron nitride sleeve cracking, radial stiffness of claddings, clad strength related to contact pressure, and interface pressures in the TEM-1 during operation. The work on interface pressure relations during operation has been continued, and in this report a comparison is made between the detailed layer pressures in TEM-1 and those in SNAP-10A. In addition, the internal pressure history of a typical module during a processing and operation cycle is discussed.

Work during the second quarter was concentrated in the following areas:

1. Interface pressures between SNAP-10A and TEM-1.
2. Summary in graphical form of the stress and contact pressure history of the thermoelectric module during processing and operation.
3. Volumetric analysis of the TEM-7 and -8 series of modules.
4. Analysis of the compaction of lead telluride during processing.
5. Calculated gap changes from the assembly condition to the final gas-pressure-sintering condition for TEM-8B.
6. Review of the assumptions and formulation used in the stress analysis code in an effort to improve its utility.

(1) WANL-PR(CC)-009, April 14, 1966

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Comparison of TEM-1 and SNAP-10A Interface Pressures

A stress analysis has been completed showing the contact pressures of the SNAP-10A module for comparison with the TEM-type thermoelectric modules. This analysis was performed to explain some of the differences in initial performance characteristics of the two types of modules. Figures 4-5 and 4-6 show that in the SNAP-10A module the interface pressures were highest at the lead telluride and outer iron sleeve interface (cold side), while in the TEM-1 module, stresses were highest at the inner interface between the lead telluride and iron sleeve. The SNAP-10A interface pressures were also significantly lower than those of TEM-1. The interface pressure in SNAP-10A was so low on the hot side that there would be no assurance of good contact. This may have accounted for the poorer performance of these modules.

Module Stress History

The previously calculated module contact pressures and displacements for TEM-1 are presented in graphical form in Figures 4-7 through 4-9. These curves help visualize the path and the dimensional changes in the module.

Figure 4-7 presents a plot of the contact pressure versus autoclave temperature during sintering and cooling. The autoclave pressure cycle also is shown for reference purposes. The initial pressurization of 7500 psi in the autoclave at room temperature results in a contact pressure of approximately 6400 psi. As the autoclave temperature increases from room temperature to 600°F, the contact pressure decreases because the free volumetric thermal expansion of the container formed by the clads is greater than the contained material during heating. This differential thermal expansion reduces the volumetric interference between the clads and the materials; therefore, the contact pressure is maintained at a lower value by the external autoclave pressure. At 600°F, the contact pressure begins to increase due to outer clad yielding.

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Figure 4-5. Radial Interface Pressure of SNAP-10A Generator at Operating Temperature

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Figure 4-6. Radial Interface Pressure of TEM-1 at Operating Temperature

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Figure 4-7. Module Average Contact Pressure History for Sintering and Operation (TEM-1)

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Figure 4-8. Module Clad Radial Displacement History for Sintering and Operation (TEM-1)

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Figure 4-9. Module Clad Axial Displacement History for Sintering and Operation (TEM-1)

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Further increases in temperature and autoclave pressure result in increased contact pressure. The densification of the lead telluride is assumed to begin at 900°F, and is simulated in the calculation by a void space corresponding to a 5% densification. Since the density change with temperature and pressure is not accurately known, it was simulated by four steps between 900° and 1382°F. The sharp drop in contact pressure at 1200°F reflects the effect of this simulation. The creep effect of the outer clad results in an increase in contact pressure. This was also simulated by several steps between 1200° and 1382°F. During cooling, the contact pressure continues to increase because the interference increases as the container volume contracts faster than the composite material. When the autoclave pressure is completely removed at room temperature, a residual contact pressure of about 6300 psi remains.

The radial displacement history for sintering and operation is summarized in Figure 4-8. These dimensional changes include the effect of both stress and free thermal expansion. The outer clad dimensional changes are shown by U_{b_3} . The effect of simulation of the densification of the lead telluride again results in the step change as shown at 1200° and 1382°F. During cooling, the radius of the module decreases to a net contraction at room temperature of approximately 8 mils. Springback upon removal of the autoclave pressure results in a change of only several tenths of a mil. Upon reaching the operating temperature of 1025°F, the radius of the module is about 3 mils larger than after the sintering process. The inner clad radial change is shown by the curve labeled U_{a_1} . Since plastic deformation does not occur in the inner clad, the curve follows approximately the same path during heating and cooling. The net radial change in the inner clad is very small after sintering. In coming to the operating temperature, the inner clad expands approximately 2 mils radially.

The axial displacement history of the module is presented in Figure 4-9. This curve shows a small effect of clad yield, amounting to about 1.2 mil per inch of module length. During operation, the module expands approximately 4 mils per inch of length.

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The radial and axial displacements of the module have been compared with measurements obtained at room temperature before and after sintering. The calculated results compare very well with these measurements. For example, the outer clad was found to contract an average of 8 mils radially when measurements were made after completion of the sintering process.

The results summarized above, obtained on a three-layer model using a simplified computer code method, indicate that contact pressure is maintained throughout the complete cycle. However, to determine whether this is true at every internal interface requires completion of a more detailed stress analysis code.

Volumetric Analysis of TEM-7 and -8 Series of Modules

The results of the volumetric analyses performed on the TEM-7, -7A, -8 and -8A modules have been compared to the results of the previous TEM-1 analysis. The method used was revised and, therefore, the TEM-1 analysis was upgraded. The significant items determined by these analyses follow:

1. The nominal percentage void volume in the module at assembly. This was obtained by comparing the nominal volume of the materials contained within the clad to the volume between the inner and outer clad based on nominal drawing dimensions.
2. The percentage interference volume in the module at room temperature after the modules are processed. This was obtained by considering the compaction of the lead telluride and boron nitride during processing and temperature changes. It was assumed that the module contained no void volume at the top of the gas-pressure-sintering cycle, and that the materials compacted their maximum amounts. The compaction was assumed to remain constant during the cooldown to room temperature. The interference volume was caused by the volume change due to the thermal expansions of the materials enclosed within the clad and the volume

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between the cladding.

3. The percentage interference volume in the module at operating temperature. This was determined by adding the percentage interference volume caused by the differential thermal expansion in going from room temperature to operating temperature to the percentage interference volume at room temperature after processing.
4. The percentage volume of each material within the module was determined for TEM-8 and compared to TEM-1.

The results of the volumetric analyses are presented in Tables 4-2 and 4-3.

TABLE 4-2

Results of Volumetric Analyses on Various Modules

<u>Module</u>	<u>Nominal Void Volume at Assembly (%)</u>	<u>Interference Volume at Room Temperature After Processing (%)</u>	<u>Interference Volume at Operating Temperature (%)</u>
TEM-1	2.0659	0.7284	1.7227
TEM-7	3.0569	0.6509	Temperature profile not available
TEM-7A	2.5595	0.7062	Temperature profile not available
TEM-8	2.4521	0.5780	1.4405
TEM-8A	2.0122	0.6191	1.4506

Based on these analyses, the gross contact pressure between elements of the TEM-1 module should be larger than the TEM-7, and the TEM-7 larger than the TEM-8.

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TABLE 4-3
Material Breakdown of TEM-1 and TEM-8

<u>Material</u>	<u>Percent in TEM-1</u>	<u>Percent in TEM-8</u>
Boron Nitride	9.9678	12.0869
Alsimag	14.2034	14.4207
Iron	16.0985	9.8873
Mica	3.5166	3.3573
Lead Telluride	56.2135	58.9227
Steel	None	1.3248

Analysis of Compaction of Lead Telluride During Processing

An analysis was made to determine the compaction of the lead telluride during processing of the TEM-1, -7, -7A, -8, -8A and No. 8B modules. The analysis was based on a unit couple length considering the void volume in the module at assembly, the percentage by volume of lead telluride in the module, and the measured changes in outside diameter of the module during processing. Table 4-4 lists the percentage change in the lead telluride for specific modules.

Since these percentages are approximately 8%, this analysis indicates that the assumption of compaction of the lead telluride from 92% (initial density) to 100% after sintering is nominally correct. However, it shows a small apparent variation in compaction of the lead telluride for the various module designs.

Revisions of the Stress Analysis Code

A module stress analysis code has been partially developed on another contract (AT (30-1)-3365). Although this code has not yet been completed, it has been used for some of the calculations reported, primarily for the swaging process. In order to obtain stress results

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without further development of this code, other stress codes have been utilized. One of these was a simplified code without the logic which traces the stress-strain curve of the material through the cycle. This simplified code was used to obtain the results described earlier in this section under the heading Module Stress History.

Efforts during the second quarter have shown the necessity of more accurately predicting contact pressures between each individual layer, rather than on the gross interval pressure basis. As a result, work on the stress code has been reinitiated. The basic equations of the stress code have been reviewed with the goal of incorporating more exact relationships between stress and strain in the plastic range, with temperature variations included. Reprogramming, using the new relationships, has been started.

TABLE 4-4
Change in Lead Telluride Volume by Module

<u>Module</u>	<u>Change in PbTe Volume (%)</u>
TEM-1 S/N-1	8.38
S/N-2	8.40
S/N-4	8.67
TEM-7 S/N-1	8.03
TEM-7A S/N-1	7.85
TEM-8 S/N-1	7.85
S/N-2	7.86
TEM-8A S/N-1	7.74
S/N-2	7.93
TEM-8B S/N-1	7.39

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IRON

STAINLESS STEEL

INCONEL X-750

LEAD TELLURIDE

BORON NITRIDE

MICA: ELECTRICAL
INSULATION

ALSIMAG-222:
THERMAL INSULATION

FABRICATION

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V. FABRICATION

A. THERMOELECTRIC MODULE

From January through June 1966, 19 thermoelectric modules representing 10 separately defined designs were fabricated. Table 5-1 has been compiled to present a summary of the principal differences in fabrication parameters among these modules. These data should be considered in conjunction with the descriptions of differences in detailed design (Section III-A), destructive examination of modules (Section V-B), and non-destructive and performance testing (Section VI). Some of the more important aspects of the various fabrication processes are discussed in the following paragraphs.

Component Fabrication and Processing

The technology related to the fabrication and processing of individual module components has continued virtually unchanged from the end of the Phase I effort to the present. This work is described in detail in the Compact Thermoelectric Converter Phase I Final Report, WANL-PR (CC)-006. Extensive work has been performed in machining the final diameters of mica insulating washers in order to facilitate the implementation of the many design changes which have been made in these components. The reject rate for this approach has been extremely high, however, and it appears that punching directly to size with precision dies is the best method to use. Dies have been made for the TEM-9 series modules which will constitute a large percentage of the remaining modules in Phase II-A.

In the recent fabrication of TEGS-2P PbTe washers, many outer washers were found to be over-size after heat treating. This problem had not occurred previously, and investigations are underway to determine the cause and correct the problem. Candidate sources of trouble are too high a density in the as-pressed washers, an odd particle size distribution in the as-received powder, or some portion of the heat treating cycle not conforming to the established procedures.

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TABLE 5-1
Fabrication Data for Phase II-A Thermoelectric Modules

Module Design Series	Module Serial Number	Date Fabrication Completed	Assembly Atmosphere	Axial Compressive Load Prior to Welding (lb)	Hydrogen Purge - Vacuum Degas Cycle Parameters			Room Temperature Compaction															Gas Pressure Sintering									
								Cold Hydrostatic Compaction							Swaging						Final Geometry		Cycle Parameters						Final Geometry			
								1st Step Pressure (kpsi)	2nd Step Pressure (kpsi)	3rd Step Pressure (kpsi)	4th Step Pressure (kpsi)	Final Geometry			Average Center Diameter After Each Swaging Pass (in.)																	
					Reduction in Center Diameter (%)	Elongation (%)	Reduction in Total Volume (%)					Reduction in Center Diameter (%)	Elongation (%)	Reduction in Total Volume (%)							Initial Pressure at Room Temp. (psi)	Intermediate Hold Parameters	Sintering Temperature (°C)	Sintering Pressure (psi)	Sintering Time (Hr)	Final Pressure at Room Temperature (psi)	Reduction in Center Diameter (%)	Elongation (%)	Reduction in Total Volume (%)			
					Maximum Cycle Temperature (°C)	Time at Maximum Cycle Temperature (Hr)	Final Pressure Prior to Sealing Degassing Tubes (Torr)	Initial	1st Pass	2nd Pass	3rd Pass	4th Pass	5th Pass	Reduction in Center Diameter (%)	Elongation (%)	Reduction in Total Volume (%)	Initial Pressure at Room Temp. (psi)	Intermediate Hold Parameters	Sintering Temperature (°C)	Sintering Pressure (psi)	Sintering Time (Hr)	Final Pressure at Room Temperature (psi)	Reduction in Center Diameter (%)	Elongation (%)	Reduction in Total Volume (%)							
TEM-3	5	2-25-66	Argon	1100	403/408	4.8	2.0 × 10 ⁻⁶	--	--	--	--	--	--	--	1.6253	1.6186	1.6126	1.6043	1.5969	1.5944	1.92	+0.420	--	7500	--	750	10,000	2	**	**	**	**
	6	2-25-66	Argon	1100	393/408	3.8	6.8 × 10 ⁻⁶	--	--	--	--	--	--	--	1.6253	1.6179	1.6125	1.6052	1.5969	1.5947	1.92	+0.490	--	--	--	--	--	--	--	--	--	--
	7	2-23-66	Argon	1100	400/423	4.2	1.2 × 10 ⁻⁵	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
TEM-4A	1	4-13-66	Argon	1100	389/409	4.8	2.0 × 10 ⁻⁶	30	40	45	--	1.54	+0.024	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7300	2.46	+0.016	--
	2	4-13-66	Argon	1100	398/418	4.8	3.2 × 10 ⁻⁶	30	40	45	--	1.53	+0.080	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7300	2.46	-0.020	--
TEM-5	1	3-25-66	Air	1100	94/410	5.7	2.2 × 10 ⁻⁶	20	30	40	45	1.32	--	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	6800	2.36	+0.140	--
TEM-6	1	4-13-66	Argon	1100	314/512	1.0	3.0 × 10 ⁻⁶	30	40	45	--	1.34	+0.097	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7300	2.39	+0.030	--
	2	3-25-66	Argon	1100	304/410	2.0	2.0 × 10 ⁻⁶	30	40	45	--	1.57	+0.081	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	6800	2.46	+0.003	--
TEM-7	1	4-20-66	Argon	1100	397/408	2.1	2.0 × 10 ⁻⁶	45	50	--	--	1.77	-0.035	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7200	2.47	-0.140	--
	2	4-20-66	Argon	1100	396/410	2.2	3.6 × 10 ⁻⁶	45	50	--	--	1.77	-0.035	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7200	2.59	-0.070	--
TEM-7A	1	4-20-66	Argon	1100	393/402	3.3	6.1 × 10 ⁻⁶	45	--	--	--	1.42	-0.044	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7200	2.35	-0.104	--
TEM-8	1	6-2-66	Argon	1000	397/407	2.3	2.0 × 10 ⁻⁵	50	--	--	--	1.71	+0.083	--	--	--	--	--	--	--	--	--	125	30 min at 750°C & 15,000 psi	650	15,000	2	13,600	2.35	+0.100	--	
	2	6-2-66	Argon	1000	394/406	2.1	1.8 × 10 ⁻⁶	50	--	--	--	1.59	+0.075	--	--	--	--	--	--	--	--	--	125	30 min at 750°C & 15,000 psi	650	15,000	2	13,600	2.33	+0.075	--	
TEM-8A	1	5-2-66	Argon	1000	378/410	1.4	2.4 × 10 ⁻⁶	45	--	--	--	1.53	+0.033	--	--	--	--	--	--	--	--	--	--	10,000	--	650	15,000	2	12,600	2.35	+0.025	--
	2	5-2-66	Argon	950	396/410	2.1	2.4 × 10 ⁻⁶	45	--	--	--	1.53	+0.017	--	--	--	--	--	--	--	--	--	--	10,000	--	650	15,000	2	12,600	2.28	-0.017	--
TEM-8B	1	6-2-66	Argon	900	390/404	2.1	2.9 × 10 ⁻⁶	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	125	30 min at 750°C & 15,000 psi	650	15,000	2	13,600	2.27	-0.100	--
								7500	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	7500	--	750	10,000	2	7100	2.30	+0.017	--
TEM-9	1	6-15-66	Argon	1100	392/405	3.0	3.0 × 10 ⁻⁶	50	--	--	--	1.52	+0.051	3.4	1.6545	1.6305	1.6297	1.6302	1.6301	--	1.46	+0.137	3.4	7500	--	750	10,000	2	7000	2.46	+0.060	5.1
	2	6-15-66	Argon	1100	390/407	2.3	5.7 × 10 ⁻⁶	50	--	--	--	1.56	+0.034	3.1	1.6553	1.6299	1.6303	1.6303	--	--	1.52	+0.146	3.1	7500	--	750	10,000	2	7000	2.50	+0.060	4.7
	3	6-15-66	Argon	1100	375/407	3.4	6.9 × 10 ⁻⁶	50	--	--	--	1.74	+0.043	3.4	1.6546	1.6264	1.6268	1.6269	1.6270	--	1.66	+0.146	3.5	7500	--	750	10,000	2	7000	2.54	+0.060	5.1

* Prior to 2nd Gas Pressure Sintering Cycle

** Data not available

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Module Assembly

Module assembly techniques have remained essentially unchanged except for the jig and fixture variations necessitated by the geometric differences among the various TEM designs. All modules except TEM-5 S/N-1 were assembled in an argon-filled glove box. A much improved axial compression fixture, which essentially permits uniform axial loading of circuit components from both ends of a module prior to welding, was designed, fabricated and used on all modules from TEM-7 S/N-1. Design changes have eliminated the need for custom-machined insulating components in TEM-8 and TEM-9 series modules, so that the steps associated with making and fitting such components have been dropped from the fabrication sequence.

Hydrogen Purge-Vacuum Degassing

Certain variations in the parametric configuration of the hydrogen purge-vacuum degassing cycle have been tried on an experimental basis. On TEM-5 S/N-1 and TEM-6 S/N-1 and -2, attempts were made to cool the module bore with flowing air or water during the entire cycle. This cooling was intended to prevent the creation of a tensile hoop stress in the inner boron nitride insulating sleeve through interference between the sleeve and the radially expanding inner clad. It was unsuccessful, however, due to the problems encountered with inadequate temperature measurements, inability to maintain uniform axial temperature distributions, and equipment limitations. This approach was ultimately discarded in favor of providing a larger radial clearance between the two components.

A completely new purging-degassing system has been designed, built and is in the final check-out stage for use with future modules. This new system will provide the following advantages:

1. Greatly improved gas purification and flow monitoring apparatus.
2. Elimination of the need for using argon to initiate and terminate the various hydrogen purging steps.

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3. Ability to obtain pressure and high vacuum measurements very close to the module rather than near the vacuum pumps as was previously the case.
4. Simple error-free cycle operation through the use of solenoid-operated high vacuum valves and central control stations.

It is anticipated that this system will be placed in service during the proposed TEM-9 module fabrication program.

Room Temperature Module Compaction

Most of the work in this area has been performed using cold hydrostatic compaction in a liquid autoclave, with pressures in the range from 30,000 to 55,000 psi. A number of design approaches have been used to provide acceptable uniform deformation under the constant applied stress. In many cases, the results have been reasonably good, but it is believed that this process alone may not be adequate for producing all the desired compaction prior to gas-pressure-sintering. To evaluate this observation, the TEM-9 modules were processed with a combination of cold hydrostatic compaction, followed by a small amount of rotary swaging. The hydrostatic compaction produces most of the required deformation in the thermoelectric circuit region with little or no axial elongation and attendant axial shifting of circuit components. Swaging compacts the end assemblies, bringing the module to a uniform outside diameter, and provides a small amount of additional compaction in the thermoelectric circuit region. Although a certain amount of elongation occurs during the swaging, it is primarily in the structural shell of the module and not in the circuit.

Gas-Pressure-Sintering

Certain variations in the parametric configuration of the gas-pressure-sintering cycle have been explored. These variations have been directed principally toward achieving an adequate final compaction in the modules, while limiting the temperature and pressure conditions

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which are believed to essentially control the transport of lead telluride base materials out of the thermoelectric circuit envelope. The cycles which have been used to date are shown in Figures 5-1, 5-2 and 5-3.

The standard 750°C - 10,000 psi - 2 hour cycle shown in Figure 5-1 is believed to accomplish the desired final compaction when used in conjunction with a cold deformation process which provides adequate initial compaction. However, it cannot overcome a condition of inadequate cold compaction, nor can it smooth out the geometric irregularities in a module which has been cold processed by hydrostatic compaction alone. Unfortunately, this cycle is the one which has been used on modules which, to date, have exhibited the most severe lead telluride-base material transport problems.

The standard 650°C - 15,000 psi - 2 hour cycle shown in Figure 5-2 was intended to accomplish the same degree of compaction as the cycle shown in 5-1, while limiting the material transport by reducing the cycle temperature. Originally, this cycle was to use a higher pressure (20,000 to 25,000 psi), but no adequate autoclaves were available. The modules run in this cycle were cold deformed by hydrostatic compaction only, and the degree of compaction after gas-pressure-sintering was good but not optimum. Judging from the test data accumulated on these modules, the severity of the material transport problems has been reduced but not eliminated. Similar cycles (with higher pressures, if possible) are planned for TEM-9-type modules.

A somewhat modified version of the cycle presented in Figure 5-2 is shown in Figure 5-3. This cycle is intended to accomplish the objectives noted above and to achieve most of the compaction at a lower temperature by applying the full cycle pressure (15,000 psi) at a temperature of 400°C and holding at this point to permit the operation of time-dependent compaction mechanisms before proceeding to the ultimate sintering temperature of 650°C . Three modules were processed in this cycle: two were cold deformed by hydrostatic compaction only, and one had no cold deformation. After the cycle, the degree of compaction of the

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Figure 5-1. Standard 750°C - 10,000 psi - 2 Hour Gas-Pressure Sintering Cycle

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Figure 5-2. Standard 650°C - 15,000 psi - 2 Hour Gas-Pressure-Sintering Cycle

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611203-16B

Figure 5-3. Modified 650°C - 15,000 psi - 2 Hour Gas-Pressure-Sintering Cycle

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former was judged to be marginal and that of the latter to be inadequate. Therefore, these modules were all reprocessed in a standard 750°C - 10,000 psi - 2 hour cycle.

At present, it appears that the standard 650°C - 15,000 psi - 2 hour cycle is the best used thus far, but that a higher autoclave pressure, a more complete compaction at room temperature, and/or a larger fraction of the total compaction occurring well before the maximum autoclave temperature is reached, are the directions to follow to attain a more optimum cycle. It is presumed that, if sufficient compaction and attendant mechanical "sealing" of the lead telluride within the circuit envelope can be achieved at a low enough temperature, the material transport problems will be eliminated or at least reduced to an acceptable level.

B. DESTRUCTIVE EXAMINATION OF MODULES

The purpose of destructively examining thermoelectric modules is to reveal the relative movements of components that occurred during module fabrication and testing. The results, conclusions and evaluations of these examinations are later factored into various modules. Details of module design changes are given in Section III of this report.

The disassembly of modules was initially performed by peeling away or removing one layer at a time, beginning with the outer layer and then proceeding toward the inner clad. This method of module disassembly is slow and could not keep pace with the accelerated changes of module design, fabrication and assembly. The most recent method of destructive examination is to make transverse cuts, 90° to the module axis, resulting in cylindrical pieces. The transverse faces are rough and fine ground by using a series of successively finer abrasives. These transverse faces are examined for radial defects. To examine the module components longitudinally for circumferential and axial defects, the cylindrical sections are mounted in a resin by using a cold mounting technique. These mounted sections are cut axially and the cut faces are ground in the same manner as the transverse faces.

The thermoelectric modules which have been destructively examined during Phase II-A are presented in Table 5-2.

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TABLE 5-2
Destructively Examined Modules

<u>Series</u>	<u>Serial Numbers</u>	<u>Process</u>
TEM-3	7	Assembled and hydrogen purged (400°C)
TEM-3	6	Assembled, hydrogen purged (400°C) and swaged
TEM-3	5	Assembled, hydrogen purged (400°C), swaged and gas pressure sintered (750°C - 10,000 psi)
TEM-4	1	Assembled, hydrogen purged (400°C), cold isostatically compressed (40,000 psi) and gas-pressure-sintered (650°C - 7500 psi)
TEM-4A	2	Assembled, hydrogen purged (400°C), cold isostatically compressed (45,000 psi) and gas-pressure-sintered (750°C - 10,000 psi)
TEM-5	1	Assembled, hydrogen purged (325°C), cold isostatically compressed (45,000 psi) and gas-pressure-sintered (750°C - 10,000 psi)
TEM-6	1 and 2	Assembled, hydrogen purged (400°C), cold isostatically compressed (45,000 psi) and gas-pressure-sintered (750°C - 10,000 psi)
TEM-7	2	Assembled, hydrogen purged (400°C), cold isostatically compressed (50,000 psi) and gas-pressure-sintered (750°C - 10,000 psi)

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TEM-3 S/N-5, -6 and -7

These three modules were assembled and fabricated through various stages of the overall assembly sequence, as shown in Table 5-2, for the purpose of destructive examination to establish the effect of various processing changes upon the insulating sleeve integrity and component displacement. The principal steps and methods of destructive examination of these modules are illustrated in Figures 5-4 through 5-6. Examination of these modules revealed the following:

1. Axial cracks in the outer boron nitride insulating sleeves of S/N-6 and -7.
2. Slight penetration of lead telluride-type material into outer boron nitride insulating sleeve of S/N-5.
3. Radial cracks in the inner boron nitride insulating sleeves of S/N-5 and -6. (Figure 5-7 shows the cracks in S/N-6.)
4. Some of the radial cracks in the inner boron nitride insulating sleeve of S/N-5 appear to be filled with a lead telluride-type material.
5. Axial cracks in the inner boron nitride insulating sleeve of S/N-7.

TEM-4 S/N-1

This module was fabricated with several design and process changes made to eliminate the low resistance between the thermoelectric circuit and clad. Mica washers were redimensioned; couples were selectively assembled; gas-pressure-sintering temperature and pressure were limited to 650°C and 7500 psi; and the module was initially compressed by cold isostatic compaction. The following observations were made during the destructive examination of this module:

1. The inner boron nitride insulating sleeve contained one axial crack which was filled with a conducting material.
2. Several radial cracks in the inner boron nitride insulating sleeve were observed to be filled or partially filled with a conducting material.
3. The lead telluride washers did not appear to be fully densified, and the bonding

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Figure 5-4. TEM-3 S/N-5 Module

Figure 5-5. TEM-3 S/N-6 Module

Figure 5-6. TEM-3 S/N-7 Module

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between concentric washers was poor.

4. No penetration of the boron nitride by materials from the lead telluride was observed.

The pre-post operational testing of this module indicated that electrical shorting to the cladding developed just prior to removal from test at about 120 hours and was probably due to movement of conducting material into defects. The low temperature-low pressure autoclave cycle of this module experiment probably established a less than minimum required condition for compaction, as noted in the density of the lead telluride and lack of complete adherence of lead telluride interfaces.

TEM-4A S/N-2

In the TEM-4A module, the inner clad was thicker, additional clearance was provided between the boron nitride insulating sleeve and the inner clad, the inner clad was sealed with plugs for the room temperature hydrostatic pressing operation, the collector rings were made more flexible, and the dimensions of the mica washers were modified.

TEM-4A S/N-2 was sectioned and examined. Significant penetration of lead telluride-type material into the inner and outer boron nitride insulating sleeves was observed. Figure 5-8 shows this penetration to be near the mica washer joints, principally in the form of filling cracks in the sleeve. It also illustrates the typical difference in response of the p- and n-type materials to the dry grinding-polishing surface preparation because of the less ductile nature of the p-type material at room temperature. The p-type material has many surface fractures or discontinuities resulting from the surface preparation.

TEM-5 S/N-1

This module had five different "sealing" arrangements designed into it. These seals were employed to eliminate shorting by preventing the lead telluride-type material from flowing radially toward the inner clad. These seals were described in WANL-PR(CC)-009, Compact

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Figure 5-8. TEM-4 S/N-2 Module Showing
Penetration of PbTe-Type Material
Near Mica Joints

Figure 5-7. TEM-3 S/N-6 Module Showing
Radial Cracks

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Thermoelectric Converter Quarterly Progress Report, dated April 14, 1966. TEM-5 S/N-1 also included an experimental end assembly. This module was sectioned by making radial cuts normal to the module axis at the interface of each seal array. Subsequent axial cuts of these cylindrical pieces showed several radial cracks in both the outer and inner boron nitride insulating sleeves. Approximately 50% of the cracks were at or near mica washers. In several instances where double boron nitride sleeves were applied, the cracks did not appear to propagate across the BN/BN interface, which led to the conclusion that the interface might serve as a crack propagation barrier.

TEM-6

The design for TEM-6, described in detail in Section III of this report, used the seal configuration array "H" from the TEM-5 design over the entire active length of the module. In addition, these modules incorporated an electrically isolated thin tubular iron membrane sandwiched between two layers of boron nitride, extending the full length of the thermoelectric circuit and adjacent to the inner clad.

The TEM-6 S/N-2 module was instrumented and placed on test. After 38 hours at operating temperature, a low resistance reading was observed from the thermoelectric circuit to the clad. On removing the module from the test stand, the resistance from circuit to clad was found to be unstable, as previously noted before testing and shown in Figure 6-1, and seemed to be a function of temperature. When the module was cool, resistances of up to 100,000 ohms were measured.

Upon heating the negative end of the module with a torch, the low resistance returned. It was then decided to remove the negative retainer ring, the outer clad, and the insulation to expose the end of the iron membrane sandwich. The end of the iron member of the sandwich was bent, was very close to the inner clad, and could be shorted by light pressure. This defect was attributed to an assembly fixture deficiency. The module was then sectioned into four pieces by making three radial cuts at 2, 4 and 6 inches from the exposed negative end closure.

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These sections were cut axially, ground, and visually examined. The outer boron nitride insulating sleeves had indications of light radial cracks and some penetration of lead telluride-type material at the mica insulating rings. Because of the mica ring displacement, there was shorting of adjacent iron conductor rings. The boron nitride backup rings had penetration of lead telluride-type material at the mica joints, as shown in Figure 5-9. The individual boron nitride backup rings varied from pure white to gray in color, with the gray-colored rings displaying a different texture. The boron nitride sleeve next to the inner clad showed a definite discoloration. These variations are being considered in an evaluation of the quality and uniformity of the original boron nitride material, and reflect its susceptibility to penetration by the lead telluride-type material under process conditions which are being investigated experimentally.

TEM-6 S/N-1 was sectioned for visual examination by making radial cuts so that each end section contained the end closure configuration and five terminal thermoelectric couples. These sections were cut axially, ground flat, and visually examined, revealing defects similar to TEM-6 S/N-2, as shown in Figure 5-10. A considerably larger amount of lead telluride-type material penetrated the outer boron nitride insulating sleeve and backup rings than was observed in TEM-6 S/N-2. There was also an indication of lead telluride-type material forced axially along the boron nitride and iron conductor interfaces. Additional metallographic work is planned for a more detailed investigation of this module and confirmation of the observations, particularly with respect to the transport of lead telluride. Although these two modules were supposed to have the same autoclave process, they were done in separate cycles, and it is believed that local temperature variations could cause the observed difference. Special thermal profile mapping runs are going to be made to gain better control of cycle parameters.

TEM-7 S/N-2

This module was destructively examined after it was tested for nearly 400 hours. During the later part of this testing, a decrease in open-circuit voltage, power output, and internal resistance with no corresponding decrease in resistance of thermoelectric circuit to metal

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Figure 5-9. TEM-6 S/N-2 Module Showing Penetration of PbTe-Type Material Near Mica Joints

Figure 5-10. TEM-6 S/N-1 Module Showing Penetration of PbTe-Type Material Near Mica Joints

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clads was observed. Destructive examination of this module revealed heavy penetration of material into the 0.011-inch boron nitride sleeve (as shown in Figures 5-11 and 5-12), a transverse and longitudinal section at No. 16 thermoelectric couple and through No. 8 to No. 16 thermoelectric couple, respectively.

Figure 5-13, a longitudinal section of the first seven thermoelectric couples, shows the following:

1. A crack in the outer boron nitride sleeve indicated by the arrow.
2. Curvature of thermoelectric material from axial displacement during fabrication.
3. Offset of the outer and inner iron conductor rings toward the center thermoelectric couples.
4. Penetration of conductive material at the inner iron conductor and mica washer interfaces.

The destructive examination of the module, showing penetration of the inner boron nitride insulating sleeve by the lead telluride-type material, tends to confirm the observed electrical characteristics of the module just prior to termination of the performance test. These changing characteristics listed in Section VI indicate intercouple shorting. It is now believed that the autoclaving temperature of 750°C to which the TEM-7 modules were exposed is quite excessive and may be a major cause of the shorting observed.

Examination of the end collector and power lead through assemblies showed them to be good electrically but that further work would be needed for satisfactory mechanical arrangement to prevent excessive distortion during fabrication.

Figure 5-14, a longitudinal section from the neagative end of the module, shows similar defects as enumerated for Figure 5-13.

Figure 5-15 shows the flexible end design that was used in this module and the inward deformation of the end retaining assembly during fabrication.

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Figure 5-11. Transverse Section of TEM-7 S/N-2 Module Showing Heavy Penetration of Boron Nitride Sleeve

Figure 5-12. Longitudinal Section of TEM-7 S/N-2 Module Showing Heavy Penetration of Boron Nitride Sleeve

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Figure 5-13. Longitudinal Section of First Seven Thermoelectric Couples of TEM-7 S/N-2

Figure 5-14. Longitudinal Section of Negative End of TEM-7 S/N-2

Figure 5-15. Flexible End Design of TEM-7 S/N-2

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The principal conclusions drawn from the destructive examination of modules, to date, are that the higher temperature autoclave cycles cause a lead-rich material to move into the boron nitride, that there is some variability in response to penetration which is related to heterogeneity of the boron nitride, and that control by design and fabrication process changes can effect improvements by minimizing the tendency for boron nitride to crack.

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TESTING

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VI. TESTING

A. NON-DESTRUCTIVE TESTING

With few exceptions, all of the modules fabricated during the second quarter were subjected to the same initial examinations prior to static testing or destructive examination. Electrical resistances of the thermoelectric circuit and of leakage paths to clad were measured. The open-circuit voltage was also measured at low average temperature values as a function of $\bar{T}_H$, and as a function of axial position along the module.

Room Temperature Resistance Measurements

The circuit resistance of each module was measured immediately after fabrication. In all cases, the standard four-terminal method of resistance measurement was used. Of the 12 modules measured during this quarter, all but the two TEM-8A* modules had circuit resistance values between 10 and 20 milliohms. The calculated room temperature circuit resistance of a thermoelectric module is 15 milliohms.

<u>Module</u>	<u>Internal Resistance, R_g (ohms)</u>
TEM-8A S/N-1	0.112
TEM-8A S/N-2	0.107

The TEM-8A modules, having an internal resistance > 0.020 ohm, would be expected to have a number of poor electrical contacts; however, the circuit resistance decreased to normal values, as expected, due to differential expansion of module components during static testing. This effect is shown in Figure 4-1 of Section IV.

* From another program.

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The leakage resistance from the thermoelectric circuit to its cladding was also measured for each module. The method of measurement, depending on the magnitude of the resistance, was either a four-terminal method, an impedance bridge, or a megohm bridge-type insulation-resistance meter. The leakage resistances for the modules are shown in Figure 6-1.

On particular modules which had exhibited a very low leakage resistance from circuit to clad, a direct current was passed through the thermoelectric circuit and voltages were measured along the module cladding. Between points of high leakage paths, some of this current would flow through the cladding causing sharp changes in a voltage versus axial position plot. In this manner areas of specific interest were pinpointed for careful observation during destructive examination.

Hot-to-Cold Water Testing

To accomplish a cursory evaluation of each module's output and to provide some low-temperature performance data, the open-circuit voltage was measured while passing hot water through the inside diameter of each module and cooling its outer surface with cold water. The results of the hot/cold water tests are given in Figure 6-2. The output is given in millivolts per centigrade degree at an average module temperature of 50°C.

Heater Probe Testing

To examine module performance as a function of axial position, a heat pulse was supplied to the inside surface of each module by a short electric resistance heater along a small portion of the overall module active length. The outer clad of the module is cooled with a water jacket throughout the test. The procedure is as follows. The peak open-circuit voltage resulting from a 1500-joule pulse of energy into the heater is measured. After temperature equilibrium has returned, the process is repeated in the module section adjacent to that just tested and so on for the entire active length. The results are plotted

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Figure 6-1. Leakage Resistances for Various TEM Series Modules

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Figure 6-2. Phase II-A Hot/Cold Water Test Results

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as open circuit voltage versus axial position along the module. All of the plots have the same general shape; however, the voltage outputs vary and result in curves of different heights. The plots are enveloped by the curves in the order shown in Figure 6-3.

B. STATIC TESTING

Thirteen modules were tested during the second quarter under simulated design conditions. The 1/2-inch bores of the modules were fitted with 3/8-inch diameter cartridge-type resistance heaters. The annulus between the heaters and the inside diameter of each module was filled with a liquid metal to function as a heat transfer medium. The outside diameter of each module was fitted with cooling fins so that the outer clad could be maintained at a lower temperature relative to the inner clad. All the instrumentation necessary to obtain the desired data was placed on the module (i.e., thermocouples, voltage probes, etc.). The entire assembly is operated in a test stand which serves to control the various testing parameters to be observed. Data are gathered on strip chart recording instruments and a digital voltmeter system. To compensate for a difference in axial length between the heater and the thermoelectric circuit, correction factors were calculated for each module. The reported efficiencies are corrected efficiencies where applicable. In all cases, the variance between measured and reported efficiency is less than 6%. For the TEM-7 and -8 series of modules, no correction factor has been used.

Performance tests (of all modules beginning with the TEM-7 series) were operated under open-circuit conditions to increase analytical capabilities. The ability to subsequently switch to load operation and to evaluate what the matched load conditions would be has been demonstrated experimentally on several occasions. The power inputs, temperatures, and open-circuit voltage are monitored as previously, but the circuit resistance is now being measured using a four-terminal alternating current method. The resistance from the thermoelectric circuit to the clad of the module is also being monitored. The power output and efficiency for open-circuit testing are calculated as follows:

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Figure 6-3. Heater Probe Test Results

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$$P_e = \frac{V_{oc}^2}{4R_g}$$

$$\eta = \frac{P_e}{P_i - \frac{P_e}{2} + 2 \frac{\bar{T}_H (^{\circ}K)}{\bar{T}_H - \bar{T}_C} P_e}$$

P_i = Input power to main heater.

TEM-1 S/N-1 and -2

These modules have each logged over 5500 hours of post-seasoning test time, and remain on test. Both are being run at a constant $\bar{T}_H$ of 538°C. The $\bar{T}_C$ of S/N-1 is being held constant at 200°C, and the $\bar{T}_C$ of S/N-2 is being held constant at 190°C. The efficiencies of each of these modules are very stable, as shown in Figures 6-4 and 6-5. Both modules are being tested under load.

At present, the electrical output of the modules for matched load conditions is as follows.

	V_{oc} (volts)	I_L (amp)	R_g (ohms)	P_e (watts)	Corrected Efficiency, η
TEM-1 S/N-1	5.84	20.12	0.146	58.5	4.01
TEM-1 S/N-2	5.94	20.44	0.146	60.5	4.23

It is significant that these modules are performing well even though they have leakage paths from circuit to cladding of about 1 ohm.

TEM-1 S/N-4

This module which had been operated for over 5000 post-seasoning test hours, suffered a sharp decrease in efficiency and an accelerated degradation due to an overtemperature caused by a voltage regulator failure (Figure 6-6). To provide testing space for a more recently fabricated module, it was removed from the test stand. Its electrical output characteristics at the time of termination of testing were as follows:

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Figure 6-4. Efficiency Versus Time: TEM-1 S/N-1

Figure 6-5. Efficiency Versus Time: TEM-1 S/N-2

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Figure 6-6. Efficiency Versus Time: TEM-1 S/N-4

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V_{oc}	=	6.24 volts	P_e	=	48.8 watts
I_L	=	15.60 amp	η	=	3.20% (corrected)
R_g	=	0.199 ohms			

TEM-4A S/N-1

Performance testing of this module was initiated during the second quarter (Figure 6-7). Its current electrical output is as follows:

V_{oc}	=	5.61 volts	P_e	=	74.1 watts
I_L	=	26.48 amp	η	=	4.60% (corrected)
R_g	=	0.106 ohms			

The leakage resistance from circuit to clad is approximately 1 ohm.

TEM-6 S/N-2

This module was instrumented and placed on test in April.

As testing progressed, the leakage resistance from the thermoelectric circuit to the cladding, which had been unstable initially, became measurable, and was seen to be decreasing with time. Since leakage resistance was the prime parameter to be investigated, the module was removed from test at that point, after a total testing time of 384 hours, for destructive examination.

TEM-7 S/N-1

This module was placed on static test in May and is continuing to operate at a constant power input of 1400 watts and a constant $\bar{T}_C$ of 200°C. The efficiency versus time plot is

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Figure 6-7. Efficiency Versus Time: TEM-4A S/N-1

Figure 6-8. Efficiency Versus Time: TEM-7 S/N-1

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shown in Figure 6-8. The decreasing efficiency is accompanied by an increase in R_g and V_{oc} . The resistances to both the inner and outer clad remain in the 70,000- to 80,000-ohm range.

TEM-7 S/N-2

The performance testing of TEM-7 S/N-2 was started and terminated during the second quarter. This module was tested under open-circuit conditions. The power inputs, temperatures, and open-circuit voltage were monitored as usual; however, the circuit resistance was measured using a four-terminal alternating current method. The resistances from the circuit to each of the clads were also monitored during the test.

The first 260 hours of testing were completed while holding the power input and average cold side temperature constant. During this period, a 20% degradation was accompanied by a decreasing average hot side temperature. At this point, the testing method was changed from a constant power input condition to one of constant average hot side temperature. During the next 120 hours of testing the following conditions were noted with a constant $\bar{T}_H$ and $\bar{T}_C$.

1. Dropping open-circuit voltage.
2. Dropping internal resistance with no drastic decrease in resistance to clads.
3. Dropping power output.

It was believed that serious shorting to the metal tube sandwiched in the inner insulating sleeve was occurring, and, therefore, the module was taken off test. Destructive examination of the module found this to be true. The TEM-7 series of modules were gas-pressure-sintered at 750°C, which is now thought to be excessive in terms of lead-type shorting of the insulating sleeves, based on recent experiments.

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TEM-8 S/N-1 and -2*

These modules were placed on test near the end of the second quarter. They will both be tested under open-circuit conditions, constant power input, and constant $\bar{T}_C$. The leakage resistances from circuit to clad of the S/N-1 and S/N-2 modules are 46 and 12 ohms, respectively. These readings were taken at static testing temperatures.

TEM-8A S/N-1*

This module was placed on test in May. Testing was begun with no load and with circuit resistance being measured by a four-terminal alternating current method. For the first 518 hours, the module was tested at a constant power input of 1280 watts and a constant $\bar{T}_C$ of 200°C. The module was then subjected to a planned thermal cycle to room temperature and back to its initial running conditions.

When the module power input and $\bar{T}_C$ were returned to the conditions existing before the thermal cycle, the module power output was unchanged.

Shortly after the thermal cycle, the $\bar{\Delta T}$ was increased while holding the $\bar{T}_H$ constant. This was done in increments while observing the resulting changes in device efficiency. The temperatures were held constant at a point where the device efficiency no longer increased with an increasing $\bar{\Delta T}$. This change resulted in an overall efficiency increase from 4.92 to 5.58%. The module temperatures and output data at this point were as follows:

$\bar{T}_H$	=	538°C	R_g	=	0.089 ohms
$\bar{T}_C$	=	140°C	P_e	=	111.7 watts
V_{oc}	=	6.32 volts	η	=	5.59%

Approximately 150 hours were logged under these conditions with no noticeable degradation.

*From another program.

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At 812 hours of running time, a load resistor was placed across the module terminals. A discrepancy of 0.4% was noted between open-circuit and matched-load efficiency calculations. The reason for the difference lies in the use of temperature readings in open-circuit voltage calculations which are not actually the $\bar{T}_H$ and $\bar{T}_C$ of the thermoelectric couple junctions, but of the claddings. The module input, output, and temperature data are as follows:

$\bar{T}_H$	=	540°C	R_g	=	0.093 ohms
$\bar{T}_C$	=	140°C	P_e	=	100.7 watts
V_{oc}	=	6.130 volts	η	=	5.22%
I_L	=	32.86 amp			

The efficiency versus operating time plot is given in Figure 6-9.

TEM-8A S/N-2*

Testing was conducted with no load, constant power input, and constant $\bar{T}_C$ for the first 180 hours of operation. After observing that the $\bar{T}_H$ was 11°C lower than at the initial point, the power input was increased until the $\bar{T}_H$ was again at its initial value (538°C). In order to observe the effect of increasing interface pressure, an additional increase in power input was made to bring the $\bar{T}_H$ to 565°C. This power input was then held constant and no significant decrease in $\bar{T}_H$ has been observed.

After 637 hours total operating time, a planned thermal cycle was completed from a $\bar{T}_H$ of 565°C to room temperature to a $\bar{T}_H$ of 565°C. The overall efficiency was identical to the efficiency before the thermal cycle. The module has been running since that time with no appreciable degradation. The efficiency plot is shown in Figure 6-10, and the temperatures, input and output data are as follows:

* From another program.

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$\bar{T}_H$	=	569°C	R_g	=	0.113 ohms
$\bar{T}_C$	=	202°C	P_e	=	79.7 watts
V_{oc}	=	6.000 volts	η	=	4.70%

The total kilowatt hours of energy produced by thermoelectric modules through Phase I and the first half of Phase II-A is as follows:

772 Kilowatt-hours at $\bar{T}_H = 538^\circ\text{C}$

299 Kilowatt-hours at $\bar{T}_H = 565^\circ\text{C}$

266 Kilowatt-hours at $\bar{T}_H = 590^\circ\text{C}$

C. HEATER TESTING

The two module heater tests were discontinued in May to provide additional module testing capability on the test stands. At the time of shutdown, these heaters had been tested for about 4300 hours each at a sheath temperature of 1350°F in NaK. This was a more severe thermal test than the 1000° to 1100°F NaK environment in the present module performance tests. Module heaters continue to operate satisfactorily with no failures.

D. DYNAMIC LOOP TESTING

The NaK liquid metal loop has operated for 871 hours at 1350°F with the module instrumentation test section ⁽¹⁾ in place. The loop was shut down in June to make final preparations and modifications for insertion of a module on test. The thermoelectric module hot side liquid metal inlet and discharge assemblies have been fabricated and tested, and are ready for welding to a module. The cold side air cooling manifold assembly has been assembled on the loop. The module instrumentation and testing equipment is on hand, and instrumentation assembly is in progress.

⁽¹⁾ Compact Thermoelectric Converter Progress Report, NYOO-3584-2, April 14, 1966, Figure 6-2.

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Figure 6-9. Efficiency Versus Time: TEM-8A S/N-1

Figure 6-10. Efficiency Versus Time: TEM-8A S/N-2

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VII. MATERIALS DEVELOPMENT

During the second quarter, the materials effort was largely devoted to characterizing module components and their complex interactions under the processing parameters of the thermoelectric module. Boron nitride, lead telluride and mica were investigated individually, and the boron nitride/lead telluride materials were investigated under conditions of intimate contact established at elevated temperature and interfacial pressure.

Characterization of Boron Nitride

During Phase I and thus far in Phase II-A, a major effort has been made to solve the problem of electrical shorting, which results in a degradation of module output. The problem has been one of constant study to determine how the component parts can best be manipulated to reduce or eliminate the shorting. One of the most probable causes of the shorts appears to be the result of the penetration of excess lead from the lead telluride into the boron nitride insulation. In order to establish a mechanism for this effect, it has become necessary to fully characterize the state of the boron nitride.

Numerous discussions were held with representatives of the two major suppliers of boron nitride (Carborundum Company and National Carbon Company) to determine what criteria could be used to predict optimum performance. While no firm inspection standards have evolved, various types of measurements have been investigated. Most of these investigations were designed to measure the factors which would control or affect the penetration of lead into the boron nitride. Throughout these investigations the general lack of fundamental physical and mechanical data resulting from processing variables has been most apparent. In addition, the boron nitride generally contains numerous very small black speck impurities. These impurities (believed to be carbon) present an undetermined effect on module performance.

Proceeding on the premise that a fully dense structure (2.25 gm/cc) would present the greatest barrier to vacancy or interconnected void type diffusion-penetration, numerous attempts were

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made to devise a density-measuring technique. The use of immersion measurements for irregularly shaped particles is limited by the susceptibility of the boron nitride to absorb the liquid and create erroneous data. Samples immersed in toluene and air dried showed a continual weight loss with little change in dimensions beyond the experimental scatter, as shown in Table 7-1. Toluene was selected because its density versus temperature relationship is well known, and also because toluene "wets" boron nitride very well.*

TABLE 7-1

Density of Boron Nitride After Immersion in Toluene

<u>Height (in.)</u>	<u>Diameter (in.)</u>	<u>Weight (gm)</u>	<u>Density (gm/cc)</u>	<u>Immersion Time (hr)</u>
0.9315	1.5206	58.3951	2.106	--
0.9319	1.5202	58.3930	2.107	17
0.9318	1.5205	58.3917	2.106	24
0.9311	1.5204	58.3844	2.108	48

Krylon is commonly used to coat porous refractory metal carbides for water immersion density measurements; therefore, this technique seemed feasible for boron nitride. However, attempts to coat the samples with krylon plastic showed erratic results when the numerous coating values were extrapolated to zero thickness. The results of these measurements indicate that, at the present time, there is no accurate immersion technique available.

Following the assumption that a fully dense structure would be the most impermeable to the atmosphere, thin discs of boron nitride were polished to a 1/8-inch thickness, and placed across the port of a Veeco leak detector system which was pumped down for one-half hour.

*Personal communication from J. Capriulo, Ratheon Company, Waltham, Massachusetts.

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The ultimate vacuum attained differed as a function of the type of boron nitride and the density, as shown in Table 7-2. Attempts to measure the leak rate by closing the valve and monitoring the rise in pressure failed because the initial rise was so great that the gages went off scale in a few seconds. Therefore, results of this screening test will be qualitative at best.

Measurements of the texture of the as-hot-pressed Carborundum Grade A boron nitride were attempted through the use of pole figures, but the degree of anisotropy was insufficient to produce a texture pattern.

While the theoretical density of boron nitride has been calculated as 2.25 gm/cc, the most dense material commercially available ranges from 2.10 to 2.15 gm/cc. Two attempts to densify the material by Dynapak extrusion through a 4:1 reduction at 1100°C actually produced a less dense material, with a decrease of 0.138 gm/cc.

Personnel of the Carborundum Company examined a rod of boron nitride for hardness and B_2O_3 content. Their analysis indicated a B_2O_3 content of 9 wt % and 10 wt % at opposite ends of the as-pressed slug. They also found the hardness to be lower for the 10 wt % B_2O_3 content, with Knoop values ranging from 200 to 600 for the B_2O_3 content of 10 to 9 %, respectively.

Based on this work and from discussions with Carborundum personnel, a special slug of uniform density and B_2O_3 content was ordered. In addition, this slug was supposed to be of very high purity and to contain a minimum of black specks believed to be carbon impurities. This last requirement was not met as a large number of impurity defects were found.

The as-hot-pressed slug (9 inches in diameter x 8 inches in length) was machined on the surface, and the average density was determined as 2.116 gm/cc. The B_2O_3 content analyzed from the circumferential area was 7.4 %. This slug was marked and two cylinders 1.5 inches in diameter were taken — one from the center of the slug and the other from the circumferential area. In addition, a rod 1/2 inch in diameter was taken from an area adjacent to that cylinder

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TABLE 7-2

Properties of Various Grades of Boron Nitride

Sample	Density (gm/cc)	Vacuum Attained In 1/2 Hour (mm Hg)	X-Ray Radiography
A-8-A ⁽¹⁾ A-8-B A-8-D A-8-F A-8-G	2.117 2.106 2.105	 2.0 x 10 ⁻⁴ 1.5 x 10 ⁻⁴	No Defects
A-12-A ⁽²⁾ A-12-B A-12-D A-12-F A-12-G	2.087 2.089 2.098	 5.2 x 10 ⁻⁴ 1.7 x 10 ⁻⁴	No Defects
HBR-12-A ⁽³⁾ HBR-12-B HBR-12-D HBR-12-F HBR-12-G	1.885 1.903 1.830	 (5) (5)	Many High-Density Impurities
Ca-12-A ⁽⁴⁾ Ca-12-B Ca-12-D Ca-12-F Ca-12-G	2.050 1.929 1.964	 1.2 x 10 ⁻³ 3.65 x 10 ⁻³	No Defects

(1) Carborundum Grade A, 8 Inches Long

(2) Carborundum Grade A, 12 Inches Long

(3) National Carbon Grade HBR

(4) Carborundum High-Purity Grade

(5) Material Too Porous to Attain Value on Meter

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which was taken from the circumferential area. This 1/2-inch diameter rod was sectioned into 9 pieces, and the bulk density was measured by size and weight along the rod. As shown in Table 7-3, the bottom of the slug had the lowest density, 2.09 gm/cc, a value which was uniform along the entire bottom half of the slug. The density increased toward the top of the slug, reaching a maximum value of 2.114 gm/cc. Since all of the measured densities are lower than the slug density, it appears that the portion toward the center of the slug must be more dense or that the size of the as-pressed slug is such as to preclude obtaining accurate measurements. Analysis of the ingot taken from the center of the as-hot-pressed slug should clarify the discrepancy. Sections from the top, center and bottom of the 1/2-inch diameter rod are currently being analyzed for the B_2O_3 content.

TABLE 7-3

Density of Boron Nitride Taken from Special Slug 6604-5A
Carborundum Grade A As-Pressed Material

	<u>Density (gm/cc)</u>
B-1 (Bottom)	2.090
B-2	2.090
B-3	2.090
B-4	2.090
B-5	2.105
B-6	2.108
B-7	2.108
B-8	2.114
B-9 (Top)	2.105

A slice was cut from the top of the cylinder which was taken from the circumferential area, and polished to a 1/8-inch thickness. This slice, containing numerous impurity defects, was tested to determine the vacuum to which the Veeco leak detector could be evacuated. After one-half hour, a vacuum of 6.4×10^{-4} mm Hg was reached. A Tesla coil spark will be impressed across the defect, and the slice again measured for permeability. The Tesla coil is currently being used in the quality control inspection of thin-walled boron nitride sleeves. There is some indication that the voltage dissipated at the coil tip across the thin-walled sleeve could be causing a breakdown in the dielectric properties. From this study, a relative indication of the permeability caused by the defects may be determined.

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Discussions with the suppliers of boron nitride have led to the belief that the B_2O_3 content and theoretical lattice parameter density may be related; this, in turn, should be related to the actual density and resistance to diffusion penetration. A rod of unknown orientation (5/8-inch diameter x 8-inch length) was sectioned to obtain samples from the top, center and bottom. After the density was measured, the samples were submitted for Debye-Scherrer X-ray powder pattern analysis of the lattice parameter. These studies are currently in progress. After the X-ray density has been measured, each piece will be analyzed for its B_2O_3 content and hardness.

After the analysis of the slice taken from the top of the circumferential cylinder has been completed, a thin-walled sleeve will be machined in such a manner as to contain a large number of defect impurity specks. The electrical resistance across the defects will be measured before and after the defects are tested on the Tesla coil.

As part of the program to develop a reliable incoming inspection, various grades of boron nitride were examined by X-ray radiography. In addition, the slugs were sectioned for density and permeability measurements. These data are presented in Table 7-2.

Thermal Expansion of Boron Nitride

The theoretical stress analysis design aspect of the program is still being hindered by the lack of complete thermal expansion data on boron nitride. As reported in the first quarterly progress report, samples were taken from the longitudinal and transverse directions of an as-pressed slug, canned in thin-walled AISI 316 stainless steel, heated to 350°C under vacuum, and gas-pressure-sintered (GPS) at 750°C and 10,000 psi. After GPS, the stainless steel can was removed and the boron nitride machined to 3/8-inch diameter x 1.750-inch long and 3/8 x 2.500 inch long samples for the longitudinal and transverse as-pressed directions, respectively. A comparison of the as-pressed dimensions with the dimensions after GPS showed that the boron nitride had been compressed and the density had increased approximately 2.5%. From this, it appears that boron nitride is susceptible to sintering/pressure reactions to increase the density. The thermal expansion of these samples is currently being determined.

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The percent of thermal expansion of boron nitride had been measured previously at Astro-nuclear Laboratory under Contract AT(30-1)-3365. Four samples were measured: two in a direction parallel and two perpendicular to the hot pressing direction. Each set of samples was further divided into an as-received sample and one that had been gas-pressure-sintered at 750°C and 10,000 psi. Figures 7-1 and 7-2 show the calculated mean coefficient of expansion for the parallel and perpendicular directions, respectively. The available Carborundum data for Grade A material is also shown on these curves.

The variation of the measured values from those quoted by Carborundum is quite significant. However, reliance on either set of data requires confirmation by utilizing the results of the additional experiments in progress.

Fracture of Boron Nitride

The fracture mode of boron nitride is critical in the design application of the module. To further define the mechanical stress, compression deformation, and shear mode, samples of Carborundum Grade A as-pressed boron nitride were machined to either 5/8- or 1-inch cubes. Strain gages were attached on the cube faces perpendicular to the x- and z-planes, as shown in Figure 7-3. The compressive stress, therefore, was applied either normal or parallel to the pressing direction.

Room temperature compressive tests were run to failure. The tests at 650°C were run to 3000 psi, the samples removed from the furnace, the strain gages removed, and the test resumed in a different furnace. These high-temperature tests were interrupted to ensure that the furnace assembly which had been instrumented for the strain gage readout would not be destroyed if the boron nitride fractured. The data presented in Table 7-4 show that the ultimate compressive stress is about two times the stress imposed on the sample when the gages were removed. It is believed that all of the significant data were obtained in the initial stress-strain loading portion of the curves. These data clearly reveal two important results and trends. The first is that Poisson's ratio for the samples tested with the compressive stress transverse to the pressing

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Figure 7-1. Calculated Mean Coefficient of Expansion: Boron Nitride Measured Parallel to Pressing Direction

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Figure 7-2. Calculated Mean Coefficient of Expansion: Boron Nitride Measured Perpendicular to Pressing Direction

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TABLE 7-4

Compression Data Obtained at Room Temperature and 650°C on Cubes
of As-Pressed Boron Nitride

Specimen	BN-1	BN-3	BN-4	BN-5
Temperature (°C)	25	25	650	650
Area (in. x in.)	5/8 x 5/8	1 x 1	1 x 1	1 x 1
Hot Pressing Direction of Ingot	Z	Y	Y	Z
Compression Test Direction	Y	Y	Y	Y
Ultimate Compressive Strength (psi)	38,300	39,000	5480	6630
<u>Tangent Young's Modulus (psi x 10⁶)</u>				
Strain Gage on X-Plane	5.8 to 3.9 ⁽¹⁾	4.03 to 1.86 ⁽¹⁾	0.67 to 0.96 ⁽²⁾	2.05 to 1.60 ⁽²⁾
Strain Gage on Z-Plane	7.7 to 3.3 ⁽¹⁾	3.92 to 1.50 ⁽¹⁾	0.78 to 1.24 ⁽²⁾	2.05 to 1.30 ⁽²⁾
<u>Poisson's Ratio</u>				
Strain Gage on X-Plane	0.16 ± 0.01 ⁽¹⁾	0.15 ± 0.02 ⁽¹⁾	0.15 ± 0.05 ⁽²⁾	0.71 ± 0.10 ⁽²⁾
Strain Gage on Z-Plane	0.06 ± 0.02 ⁽¹⁾	0.17 ± 0.04 ⁽¹⁾	0.11 ± 0.05 ⁽²⁾	0.40 ± 0.07 ⁽²⁾
Total Lateral Strain	$\epsilon_{zx} = 2.4 \text{ mils/in.}$ At 38,300 psi	$\epsilon_{xz} = 2.90 \text{ mils/in.}$ At 35,000 psi	(3)	(3)

(1) Measured from 5000 to 20,000 psi stress.

(2) Measured from 900 to 2700 psi stress.

(3) Not measured — strain gages removed at 3000 psi.

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direction (BN-1 and BN-5) had significantly greater ratios for the x:z planes than did the samples orientated with the compressive direction and the pressing direction parallel to each other (BN-3 and BN-4). This condition was true for both the room temperature and the elevated temperature tests. The second result is that in both cases the tangent Young's modulus was higher for the samples compressed normal to the pressing direction. These results show the anisotropic properties inherent in hot pressed material. Also, the degree of anisotropy decreases as the test temperature is increased.

Mica

The absence of accurate thermal conductivity data for the phlogopite mica being used in the module has presented difficulties in the theoretical analysis of the thermal performance of the module. As stated in the first quarterly progress report,⁽¹⁾ three different approaches are being taken to obtain these data. The first two will utilize mica strips and washers with the conductivity being measured axially and radially, respectively. The mica has been cut or pressed, and heat treated. Fixtures for making the measurements have been designed and are currently being machined. The third approach is somewhat different in that the actual laminar orientation of the mica in a tubular module application will be simulated. This module has been fabricated as shown in Figure 7-4. Unfortunately, during fabrication some of the thermocouples were damaged. As a result, the capsule has been redesigned and is being prepared for assembly.

Boron Nitride-Lead Telluride

The problem of electrical shorting between the thermoelectric circuit and the cladding of the tubular modules has been under investigation for the past eight months. During this time, evidence has been accumulated to allow a reasonable explanation for the cause of electrical shorting.

(1) WANL-PR(CC)-009, April 14, 1966.

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Figure 7-3. Strain Gage Orientation on Boron Nitride

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Figure 7-4. Schematic of Mica Capsule Used to Measure the Thermal Conductivity

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The shorting problem appears to be caused by a lead-rich material that fills cracks in the boron nitride and/or penetrates directly through it. As a result, a shorting bridge is created between the iron conductor rings and the clad, or directly between the rings. Complementary capsule tests show that this lead rich component is exuded from the n-type material under the pressures and temperatures (750°C and 10,000 psi) encountered in the GPS cycle.

Two experimental methods are being used to explore the effects of time, pressure and temperature on the penetration of the lead-rich component into the boron nitride. The first method uses direct uniaxial compression to obtain pressure at a PbTe/BN interface. The results of the experiments on 3N PbTe and Grade A boron nitride are essentially complete and will be documented in WANL-TME-1446.

Figure 7-5 shows 3N PbTe that had been cold pressed (approximately 97% dense) into a 0.500 inch diameter x 0.500 inch long cylinder, hydrogen treated for two hours at 575°C, and fitted into mating boron nitride and graphite parts. After the composite (BN/PbTe/graphite) was placed in a vacuum chamber, evacuated to 5×10^{-5} torr, and back-filled with helium, it was loaded to 0.1% of the final stress. The furnace was brought to temperature with a helium flow maintained during the test. At temperature, the load was increased to the stress levels shown in Table 7-5, held for 30 minutes and radiation cooled. The load was removed when the composite was below 160°C. Each sample was cold mounted, sectioned longitudinally, polished and etched to show the lead telluride and boron nitride.

The as-polished lead telluride samples were tested for hardness using Vickers and Tukon machines to determine the bulk hardness as well as any hardness profile variations. A final measurement of the lead telluride cross-sectional length was taken to determine the percent reduction which had occurred during the test. The results are shown in Figures 7-6 and 7-7 for the 750°C and 650°C tests, respectively. These data indicate that a maximum density can be obtained at 650°C with a drastic reduction in the contamination at stresses of 10,000 psi or greater. An explanation for the dip in the 650°C - 7500 psi points in the hardness and density curves is not available at the present time.

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Figure 7-5. Schematic of PbTe/BN Temperature Pressure Test Set Up

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Figure 7-6. Hardness, Percent Theoretical Density, and Percent Reduction in Length Versus Stress for 3N PbTe/BN Composite - Compressed 30 Minutes at 750°C.

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Figure 7-7. Hardness, Percent Theoretical Density, and Percent Reduction in Length Versus Stress for 3N PbTe/BN Composite - Compressed 30 Minutes at 650°C

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TABLE 7-5

3N PbTe/Grade A Boron Nitride Compatibility Study Testing Schedule

<u>Pellet No.</u>	<u>Temperature (°C)</u>	<u>Pressure (psi)</u>	<u>Time (min)</u>
137 P	750	4,000	30
138 P	750	6,500	30
141 P	750	10,000	30
136 P	650	6,500	30
139 P	650	7,500	30
140 P	650	10,000	30
142 P*	650	10,000	30
143 P	650	12,500	30
144 P	650	15,000	30

* Duplicate sample

Figure 7-8 shows the increase in contamination, measured as depth of penetration, for the 4000-, 6500- and 10,000-psi stress levels at 750°C. In each case, the apparent density and depth of the contaminated layer increased with the increasing load. Contrary to the results obtained on the 750°C test, the penetration observed on the samples tested at 650°C showed a very slight increase in depth as the pressure was increased. However, as shown in Figure 7-9, the penetration was progressively much more localized in each sample at stresses of 6500, 10,000 and 15,000 psi, respectively. In fact, the 15,000-psi stress is tending to produce a zone of contaminated material.

The second method utilizes the evacuated test capsules of concentric boron nitride and stainless steel cylinders which surround the lead telluride pellets. These test capsules are gas-

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(a) Pellet 137P
50X
4000 psi

(b) Pellet 138P
50X
6500 psi

(c) Pellet 141P
50X
10,000 psi

Figure 7-8. 3N PbTe/Grade A Boron Nitride Penetration After 30 Minutes at 750°C
Under Load

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(a) Pellet 136P
50X
6500 psi

(b) Pellet 140P
50X
1000 psi

(c) Pellet 144P
50X
15,000 psi

Figure 7-9. 3N PbTe/Grade A Boron Nitride Penetration After 30 Minutes at 650°C Under Load

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pressure-sintered at the same time as the thermoelectric modules. After opening the capsules, samples of the boron nitride are metallographically examined to determine the extent of radial penetration.

Figure 7-10 shows the boron nitride from test capsules containing 3N PbTe. The capsules were gas-pressure-sintered at 750°C and 10,000 psi for two hours and treated at $610 \pm 10^{\circ}\text{C}$ for different periods of time (0, 500 and 1000 hours). The photomicrographs are not typical of the entire length of the boron nitride, but represent the area where penetration was most severe. It is quite apparent that additional time at the 610°C temperature resulted in a considerable increase in both the depth of penetration and amount of material in the penetrated zone.

It would be logical to question whether the boron nitride shown in photos (b) and (c) of Figure 7-10 was not completely penetrated before the 610°C "soak". Another series of capsules containing 2N PbTe was subjected to the same sequence of events, using another autoclave run as the primary step. The results parallel those obtained using the 3N material; minimal penetration is observed directly after gas-pressure-sintering, and extensive penetration following the 500- and 1000-hour treatment at 610°C .

If, as suspected, the n-type lead telluride is the primary cause for shorting, three solutions to the problem are offered: (1) alter the n-type PbTe, (2) change the processing variables and (3) substitute or modify the boron nitride to obtain a material more resistant to penetration.

Both the 3N and 2N PbTe are made to a nominal composition of 62.15 wt% lead*. The electrical properties are modified by altering the dopant carrier concentration. Stoichiometric lead telluride contains 61.89 wt% lead, which means the 3N and 2N materials contain 0.26 wt% or 0.4 mol% excess lead. Analysis of the 3N and 2N materials yields a lead content of 62.12 wt %. However, analysis of one sample of 3N and one of 2N material after gas-pressure-sintering at 750°C and 10,000 psi for two hours disclosed a lead content of 62.01

* 3M information

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(a) Gas-Pressure-Sintered at 750°C and
10,000 psi (50x Polarized Light)

(b) Gas-Pressure-Sintered at 750°C and
10,000 psi Plus 500 Hr at 610°C
(50x Polarized Light)

(c) Gas-Pressure-Sintered at 750°C and
10,000 psi Plus 1000 Hr at 610°C
(50x Polarized Light)

Figure 7-10. Boron Nitride Gas-Pressure-Sintered in Contact with 3N PbTe

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and 61.99 wt %, respectively. Additional work is being performed, but it is possible that the 0.1 wt % lead loss from the bulk material could be the cause of the shorting problem.

A modified 3N material containing a nominal 62.02 wt % lead* has been obtained from the 3M Company. This material is being used for both uniaxial compression and isostatic tests, and subsequent comparison with standard material. In addition, it is planned to use some of the material to fabricate a TEM-9-type module to examine its behavior therein.

It has been pointed out that, in the control of penetration, temperature is a more critical parameter than pressure. Figure 7-11 shows boron nitride from capsule tests that were autoclaved at 650°C and 15,000 psi. Regular 3N and reduced lead 3N pellets were enclosed by the boron nitride shown in Figure 7-11, photos (a) and (b), respectively. Although little comparison between the two 3N types of lead telluride is possible, the reduction in penetration by standard 3N PbTe at the lower temperature (650° versus 750°C) is quite apparent. A series of capsule and compressive tests is planned in order to obtain more details on the temperature-pressure relationship as a function of time.

The following types of boron nitride have been obtained and will be evaluated for resistance to penetration: (1) high purity boron nitride containing 3% calcium and less than 0.2% B_2O_3 ; (2) a 50-50 mixture of boron nitride and SiO_2 ; (3) a rod of boron nitride containing a uniform reduced B_2O_3 concentration of 7%; (4) standard boron nitride that has been processed to provide an SiO_2 impregnated structure; and (5) HD 0090 experimental grade from National Carbon.

Oxygen Contamination

The probability of oxygen degrading the electrical properties of lead telluride modules resulted in an experimental program to measure the oxygen concentration before and after processing of 3M material. The method for quantitative measurement of the oxygen level, as well as the process necessary to reduce the oxygen concentration, has been accomplished for

* The standard 3N prior to 12-63 contained 62.05 wt % lead.

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(a) Boron Nitride in Contact with 3N PbTe
at 650°C and 15,000 psi
(50x Polarized Light)

(b) Boron Nitride in Contact with Reduced
Lead Content 3N at 650°C and 15,000
psi (50x Polarized Light)

Figure 7-11. Boron Nitride in Contact with Lead Telluride

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2N, 3N and 2P materials. In addition, measurements of the Seebeck voltage versus temperature for selected 2N and 3N material indicates that the final oxygen content in these materials is not deleterious to long time-temperature stability.

Measurement of the oxygen concentration is performed by neutron activation analysis. The results obtained on various lots of material are shown in Table 7-6.

Group 1 samples are test specimens which were pressed for material experiments. Group 2 specimens are test pellets which had been pressed and heat treated with lead telluride washers used in the fabrication of the TEM series modules. The Group 2 samples that duplicate lots in Group 1 (starred results) were pressed about four weeks later in time. The comparable oxygen content of these samples shows that the storage techniques employed for the lead telluride are effective in preventing oxygen pickup. In addition, the hydrogen heat treatment on the duplicate Group 2 samples produces material with a reduced oxygen content extremely close to that of the pellets pressed earlier in time.

Heat treating the n-type powder prior to pressing reduced the oxygen content, but did not produce a lower oxygen content material than the pellets which were heated to 575°C without prior heat treatment of the powder.

Heat treatment of p-type lead telluride (2P) powder at 350°C did not reduce the oxygen content and, in fact, material treated in this manner showed a small but definite increase in oxygen level. Regardless of whether the powder was treated at 350°C, pellets which were subjected to the 575°C temperature exhibited a marked decrease in oxygen content compared to as-received material.

As a result of the above measurements, the powder is not heat treated prior to pressing. The procedure followed at the present time is for test pellets to be pressed at the same time as the thermoelectric washers. Some of these pellets are hydrogen treated at 575°C with the washers, and then all test pellets are sent for oxygen analysis. Thus, the oxygen content

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TABLE 7-6

Oxygen Analysis of PbTe (Neutron Activation), ppm Oxygen by Weight

Group	Type - Lot Treatment	2N			3N			2P				3P
		1050	1065	1078	1051	1066	1079	1052	1080	1067	1084	1053
1	Pressed As-Received Powder	66**			88**			112**				343
	575°C H ₂ Treatment	30**			19**			27**				
	450°C H ₂ Treatment											264
	500°C H ₂ Treatment											245
	550°C H ₂ Treatment											217
	600°C H ₂ Treatment											187
2	Pressed As-Received Powder	59*	79	75	74*	86	72	130*	119	152	117	
	575°C H ₂ Treatment	27*	31	14	26*	24	18	30*	18	50	42	
	Pellets Pressed from Powder Heat Treated at 350°C	39	34	33	38	25	36	149	143	178		
	Powder Heat Treated at 350°C and Pressed Pellet H ₂ Treated at 575°C	29	<30	10	<31	18	16	48	48	43		

* Condition A represents samples pressed from original powder batch.

** Condition B represents samples pressed from powder batch after storage for one month.

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of all lead telluride lots is checked in the as-received and final-processed conditions.

The 3P Pb-Sn-Te alloy contains an extremely high oxygen content when compared to lead telluride. In addition, while treating the 3P Pb-Sn-Te alloy in hydrogen at increasingly higher temperatures produces an orderly decrease in the oxygen content, the highest temperature treatment (600°C) has not reduced the level below that of the incoming lead telluride. Higher temperatures are being tried to determine a lower limit for the oxygen content.

Electrical Measurements

This portion of the materials program is designed to measure the electrical properties of lead telluride after processing and after long-time exposure to elevated temperatures. Each material is being examined separately to determine if degradation in the thermoelectric module can be attributed to changes in the electrical properties of the thermoelectric materials.

Test pellets of 3M PbTe (2N Lot 1050 and 3N Lot 1051) were pressed and then hydrogen treated at 575°C for two hours. The oxygen content of representative samples treated with each batch was 30 and 19 ppm, respectively, for the 2N and 3N material. Seven evacuated test capsules of five pellets each were prepared and then gas-pressure-sintered at 750°C and 10,000 psi for two hours. After this treatment, six of the capsules were put in a "soaking" furnace maintained at $610 \pm 10^{\circ}\text{C}$. The remaining capsule was opened and the samples removed for measurement.

Measurements of the Seebeck voltage (relative to chromel and a cold junction of 22°C) were made as a function of temperature for all samples described above plus those that had accumulated 500 and 1000 hours at the elevated temperature. Figures 7-12 and 7-13 show the Seebeck voltage as a function of temperature for the 2N and 3N material, respectively. Each point represents the arithmetic mean of five individual samples. There is a decrease in voltage after gas-pressure-sintering, but succeeding curves are almost superimposed.

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Figure 7-12. Seebeck Voltage as a Function of Temperature for 2N Material

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Figure 7-13. Seebeck Voltage as a Function of Temperature for 3N Material

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Table 7-7 shows the difference in Seebeck voltage (mean of five samples) between the temperatures of 555° and 197°C. There is a slight decrease of 1 to 3% in the voltage of the hydrogen treated 2N and 3N PbTe after gas-pressure-sintering. However, the change in voltage after 1000 hours at the elevated temperature differs from the gas-pressure-sintered values by only -0.25% and + 1% for the 3N and 2N materials, respectively, which is within the mean deviation of the samples checked. The data indicate that n-type lead telluride, treated in this manner, should not contribute to any significant changes in the output voltage of a fabricated module. Electrical resistivity measurements of these samples are in progress.

P-type samples have not been obtained after gas-pressure-sintering because the lead telluride bonded to the boron nitride, resulting in fragmented pieces rather than intact pellets. Design ideas are being considered for ways to measure the p-type material "in-situ" using modified test capsules. SiO₂ impregnated boron nitride, as well as tubular mica insulators, will also be tried as enclosures in an attempt to obtain intact pellets.

TABLE 7-7
Seebeck Voltage of Lead Telluride Relative to Chromel

Treatment	Difference in Seebeck Voltage Between 555° and 197°C, Cold Junction at 22°C - Millivolts Relative to Chromel	
	Lot 1051 3N	Lot 1050 2N
A. H ₂ treated at 575°C for two hours	82.4 ± 0.6	100.9 ± 0.3
B. A plus gas-pressure- sintered at 750°C - 10,000 psi for 2 hours	81.1 ± 0.7	97.5 ± 1.0
C. A plus B plus 500 hours at 620°C	80.6 ± 0.4	98.8 ± 1.3
D. A plus B plus 1000 hours at 620°C	80.9 ± 1.0	98.5 ± 0.9

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VIII. FACILITIES AND EQUIPMENT

A. PLANNING

Facilities and equipment for Phase II-A are reviewed on a continuing basis. Detail tooling has been designed and procured, as required by module configuration and construction. Where possible, existing tooling has been modified to meet the requirements of development hardware without destroying its usefulness on existing designs.

The WANL Safety Committee has approved the Waltz Mill site for installation of a gas-pressure-sintering facility. As a result of recent processing experience in autoclave cycles for thermoelectric modules, a decision was made to change the autoclave from a 15,000 psi - 1000°C capability to 30,000 psi - 1000°C capability. New quotations are being obtained and the site facility requirements are being revised. The modifications to the Waltz Mill facilities for the larger autoclave have been determined to be considerably more extensive than those originally approved. The revised equipment is physically larger with more component hardware, such as: two gas compressors (versus one in the original scheme), doubled bottle gas capacity, and greater power and water requirements. Detail installation requirements and costs are in the process of being established.

B. EQUIPMENT

The following capital equipment being furnished by Westinghouse will be used in direct support of the Compact Converter Program:

Delivery of the 60,000-psi room temperature isostatic compacting system, with a chamber 4 inches in diameter and 20 inches long, was originally scheduled during this quarter; however, it has been delayed by the Industrial Board of the Pennsylvania Department of Labor and Industry. It is expected that the equipment will be code-cleared during the July meeting of the Industrial Board, at which time it will be shipped to WANL.

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Rearrangement of equipment was continued during the second quarter. The installation of a clean room for machining of component hardware was completed, and equipment was in operation by mid-June. The installation consisted of two Hardinge bench lathes; one 10-foot LeBlond lathe; one turret lathe; and one Universal tree milling machine.

An additional hydrogen purge and vacuum system was constructed for the multiple processing of modules.

The small press used for compacting thermoelectric washers has been completely rebuilt and is in operation.

Orders were placed for equipment to be utilized in a new series of individual test stands which will be used with the new AEC-provided data acquisition system. Construction of the first of three prototype test stands was started in mid-June. The new stands will provide module isolation, safety features, power input regulation, and control to a degree which is not possible in the facilities currently being used.

A rearrangement was made which divides the existing thermoelectric storeroom into the following control areas: (1) raw material stores, (2) termination stores, (3) specification part stores, (4) incoming receiving stores, and (5) quality control inspection. This rearrangement provides for barrier partitions and control access doors to each area. The necessary hardware has been delivered and soon will be installed.

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APPENDIX A

PERT DATA

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Figure A-1. PERT Network
A-2

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TABLE A-1
Summary of PERT Network Events Scheduled for Completion
During April

Event No.	Event	Last Scheduled Date	Actual Date
234 0106	Complete Special Test Equipment Order	4- 7	4-29
232 0036	Complete Manufacturing, TEM-4A	4- 8	4- 4
233 2110	Complete Assembly, TEM-6	4- 8	4-11
233 3000	Complete Manufacturing, TEM-7	4- 8	4-15
233 1126	Complete Preliminary Destructive Examination, TEM-5	4-11	4-11
233 4000	Complete Manufacturing, TEM-8	4-11	4-22
233 2125	Complete Processing, TEM-6	4-12	4-13
233 2126	Complete Preliminary Destructive Examination, TEM-6	4-15	4-20
234 0098*	Complete New Test Stand Order	4-15	5-11**
232 0038	Complete Assembly, TEM-4A	4-15	4-11
213 0004	Submit First Quarterly Progress Report	4-15	4-14
233 3110	Complete Assembly, TEM-7	4-18	4-20
234 2128	Start Performance Test, TEM-6	4-19	4-19
233 4110	Complete Assembly, TEM-8	4-20	4-29
234 0107*	AEC P.O. Approval	4-21	5-13**
232 0042	Complete Processing, TEM-4A	4-22	4-14
233 3125	Complete Processing, TEM-7	4-22	4-22
231 0072*	Complete Design, TEM-9	4-26	5- 6**
234 3128*	Start Performance Test, TEM-7	4-29	5- 4**
233 3126*	Complete Preliminary Destructive Examination, TEM-7	4-29	6- 1**
234 0128	Start Performance Test, TEM-4A	4-29	4-25
232 0126	Complete Preliminary Destructive Examination, TEM-4A	4-29	4-22

*Events not completed during April.

**Rescheduled completion date.

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TABLE A-2

Summary of Pert Network Events Scheduled for Completion During May

Event No.	Event	Last Scheduled Date	Actual Date
234 3128	Start Performance Test, TEM-7	5-4**	5-6
231 0072	Complete Design, TEM-9 Series	5-6	5-6
233 4125	Complete Processing, TEM-8A	5-6	5-18
234 4128	Start Performance Test, TEM-8A	5-11	5-13
233 5000	Complete Manufacturing, TEM-9	5-11	5-24
234 0098	Complete New Test Stand Order	5-11	5-27
234 0107	Complete AEC P.O. Approval (Data System)	5-13	5-19
232 0026*	Complete PbTe Penetration of BN Report	5-15	6-15**
231 0074*	Complete Design, TEM-10	5-17	Deferred***
233 5110*	Complete Assembly, TEM-9	5-17	6-14**
233 5125*	Complete Processing, TEM-9	5-24	6-22**
233 6000*	Complete Manufacturing, TEM-10	5-25	Deferred***
231 0075	Start Phase II-A Second-Half Development Plan	5-31	5-31
233 5001	Complete Manufacturing, TEM-9A	6-8**	
233 5002	Complete Manufacturing, TEM-9B	6-10**	
233 5126	Complete Processing, TEM-9A	6-24**	
233 5127	Complete Processing, TEM-9B	6-27**	

* Events not completed during May

** Rescheduled completion dates

*** Program change resulted in a series of TEM-9-type modules in place of the previously scheduled TEM-10 modules

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TABLE A-3
Summary of PERT Network Events
Scheduled for Completion During June

<u>Event No.</u>	<u>Event</u>	<u>Last Scheduled Date</u>	<u>Actual Date</u>
232 0028	Complete Evaluation of Cold Isostatic Pressing	6-1	6-30
233 4126	Complete Processing, TEM-8	6-7	6-12
232 0026*	Complete PbTe Penetration of BN Report	7-20**	
233 5110	Complete Assembly, TEM-9	6-14**	6-10
234 4129	Start Performance Test, TEM-8	6-13	6-20
233 5125	Complete Processing, TEM-9	6-22**	6-20
234 5128*	Start Performance Test, TEM-9	6-28**	Test Deleted
234 0100	Start New Static Test Stand Installation	6-23**	6-15
233 5001*	Complete Manufacturing, TEM-9A	7-15**	
233 5002*	Complete Manufacturing, TEM-9B	7-15**	
233 5111*	Complete Assembly, TEM-9A	7-22**	
233 5126*	Complete Processing, TEM-9A	8-5**	
233 5112*	Complete Assembly, TEM-9B	7-27**	
233 5127*	Complete Processing, TEM-9B	8-5**	
232 0024*	Complete Mica Thermal Conductivity Report	9-1**	
231 0076	Complete Phase II-A - Second Half Development Plan	6-15	6-30
232 0020	Complete Mid-term Material Fabrication and Processes Evaluation	6-30	6-30
233 3126	Complete Preliminary Destruct Examination, TEM-7	7-5	6-30
234 5136	Complete Preliminary Destruct Examination, TEM-9	6-30	6-30

* Events not completed in June

** Rescheduled completion dates

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TABLE A-4
Phase II-A Compact Thermoelectric Converter Program
List of Major Milestone Events

<u>Event No.</u>	<u>Event</u>	<u>Last Scheduled Date</u>	<u>Actual Date</u>
200 0000	Phase II-A Go-ahead	1-1-66	1-1-66
212 0002	Submit Phase II-A Project Plan to AEC	1-31-66	1-31-66
232 0088	Complete Destructive Examination, TEM-3	3-1-66	3-28-66
234 3128	Start Performance Tests, TEM-6	4-19-66	4-19-66
213 0004	Submit First Quarterly Progress Report	4-15-66	4-14-66
234 3128	Start Performance Tests, TEM-7	5-4-66*	5-6-66
232 0028	Complete Evaluation of Cold Isostatic Pressing	6-1-66	6-1-66
232 0020	Complete Mid-Term Material, Fabrication and Process Evaluation	6-30-66	6-30-66
234 0110	Complete Checkout of Special Test Equipment	8-5-66*	
211 0006	Submit Second Quarterly Progress Report	7-15-66	
232 0016	Complete PbTe Densification Report	8-15-66	
234 0094	Complete Initial Performance Test Evaluation	9-15-66	
213 0010	Submit Third Quarterly Progress Report	10-15-66	
212 0012	Issue Phase II-B Project Plan (Draft)	12-1-66	
234 0096	Complete Phase II-A Performance Tests and Evaluations	12-15-66	
213 0000	Submit Phase II-A Final Report (Draft)	12-31-66	

* Rescheduled completion date

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APPENDIX B
NOMENCLATURE

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NOMENCLATURE

α	Seebeck Coefficient
$^{\circ}\text{C}$	Degrees Centigrade
η	Overall Efficiency
η_c	Carnot Efficiency
$^{\circ}\text{F}$	Degrees Fahrenheit
HEP	High Efficiency Prototype: a code selected by WANL to identify one of the thermoelectric module designs which was designed and tested by WANL prior to beginning the Compact Thermoelectric Converter Program.
ID	Inner Diameter
$^{\circ}\text{K}$	Degrees Kelvin
k	Thermal Conductivity, watts/cm - $^{\circ}\text{K}$
k_n	Thermal Conductivity of n-type Thermoelectric Material, watts/cm - $^{\circ}\text{K}$
k_p	Thermal Conductivity of p-type Thermoelectric Material, watts/cm - $^{\circ}\text{K}$
P_e	Electrical Power Output, watts
Q_t	Total Thermal Power Output, watts
ρ	Electrical Resistivity, ohms - cm
ρ_n	Electrical Resistivity of the n-type Thermoelectric Material, ohms - cm
ρ_p	Electrical Resistivity of the p-type Thermoelectric Material, ohms - cm
ρ'	Electrical Contact Resistivity between the thermoelectric material and the electrical conductor, ohms - cm^2
R	Radius
R_i	Inside Radius
R_o	Outside Radius
$^{\circ}\text{R}$	Degrees Rankine
R_g	Internal Resistance of a module, ohms

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NOMENCLATURE (Continued)

T	Temperature
$\bar{T}$	Average Temperature
T_C	Mean Cold Side Temperature
T_H	Mean Hot Side Temperature
ΔT_H	Axial Temperature Drop on the Hot Side of the Module
ΔT_C	Axial Temperature Drop on the Cold Side of the Module
T/E	Thermoelectric
TEM	Thermoelectric Module: a code selected by WANL to identify all tubular modules to be fabricated under the Compact Converter Program.
V_{oc}	Open Circuit Voltage
V_L	Load Circuit Voltage, volts
I_L	Load Current

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APPENDIX C

DRAWINGS

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