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AEC Research And
Development Report

AEC Contract AT(30-1)-3584

Phase II-A

COMPACT THERMOELECTRIC CONVERTER (U)

Quarterly Progress Report

(January 1 - March 31, 1966)

Prepared For
The New York Operations Office
U. S. Atomic Energy Commission

April 14, 1966

Westinghouse Astronuclear Laboratory
P.O. Box 10864 Pittsburgh, Pa. 15236

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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 New York Operations Office of the Atomic Energy Commission.

This first Quarterly Report for Phase II-A presents the significant progress on the Compact Thermoelectric Converter Program for the period from January 1 through March 31, 1966.

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INTRODUCTION AND SUMMARY

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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The pace of thermoelectric module design, fabrication, assembly, destructive examination and testing through the first quarter was accelerated slightly relative to the schedule given in the Project Plan. The following tabulation gives the status of modules involved in the activities of this first quarter of Phase II-A.

<u>Series</u>	<u>Serial Nos.</u>	<u>Status</u>
TEM-1	1, 2, 4	Tests are Continuing
TEM-3	5, 6, 7	Destructive Examination Completed
TEM-4A	1, 2	Assembly in Process
TEM-5	1	Destructive Examination in Process
TEM-6	1, 2	Assembly in Process
TEM-7	1, 2, 3	Fabrication in Process
TEM-8	1, 2, 3	Design Completed

Static testing has been continued on three modules placed on test during Phase I. These modules (TEM-1 S/N-1, -2 and -4) have accumulated approximately 4000 hours each; performance curves are shown on Figures 6-1, 6-2 and 6-3.

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I. PROGRAM MANAGEMENT

A review of the Phase I Compact Converter Program was presented at a briefing attended by agency and industry representatives at the AEC Headquarters on February 11, 1966.

The Phase II-A program is generally proceeding in accordance with Phase II-A Project Plan, WANL-PP(CC)-003. Midway through the first quarter, a realignment of the TEM design, fabrication and test schedule was accomplished. This was done primarily to accelerate the examination and evaluation of the several design and processing moves which were intended as methods of solution of known problems. These program changes are discussed in detail in Section III-A of this report.

The following reports were published and issued during the first quarter of Phase II-A:

NYOO-3584-1*	Phase I Final Progress Report
WANL-PP(CC)-003	Phase II-A Project Plan, dated 1-27-66
WANL-PP(CC)-007	Phase II-A January Progress Report
WANL-PP(CC)-008	Phase II-A February Progress Report
WANL-TME-1342	Investigation of the Short Circuit Problem in TEM Series Tubular Thermoelectric Modules - Phase I Status

The completely updated PERT network is presented in Appendix A, and it fully reflects the program changes. A summary of the completed PERT Network events is given in Table A-1. Table A-2 presents the status of major program milestone events.

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II. RELIABILITY AND QUALITY CONTROL

A. RELIABILITY

On February 2, 1966, representatives of the Reliability Department of the Westinghouse Astronuclear Laboratory (WANL) and the NYOO reviewed the Phase II-A reliability program at the Astronuclear Site. At that meeting, progress in the areas of failure mode analysis and reliability apportionment was emphasized. A similar review was held on March 15 by WANL representatives and additional NYOO personnel.

The reliability system optimization effort has been emphasized during this reporting period. A computer model was used for evaluating various converter arrays and for apportioning reliability goals for modules within these arrays. Arrays which have been considered to date include converters using 7-, 14- and 28-volt modules for total power requirements of 1, 5, 10 and 20 kilowatts. The potential reliability advantage in using redundant converter modules comprised of 7-, 14- or 28-volt tubular module arrays is illustrated in Table 2-1. Four cases are considered for each array. In each case the converter module redundancy and the diode device reliability are the principal variables. Their effect on the TEM reliability goals is indicated for an assumed Compact Converter reliability of 0.999.

As noted in Table 2-1, Cases B, C and D indicate lower TEM reliability requirements than does Case A, thus reflecting the advantage of using converter module redundancy. It is further noted that for any particular case the array of 28-volt tubular modules has the lowest reliability requirement to achieve a given converter reliability. This reflects the fact that in a compact converter where converter modules are redundant, a 28-volt array will contain the most redundant branches and thus cause less degradation per failure.

Case D does not employ diodes, but assumes a redundant protection against shorts to the inner clad within the tubular module. In this case, no diodes are required since the low probability of having two or more shorts to inner clad places this type of failure in the non-critical

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TABLE 2-1

Required TEM Reliability for a 5 KWE Compact Converter with 0.999 Reliability

<u>Case</u>	<u>Configuration</u>		<u>Required TEM Reliability</u>		
	<u>No. of Converter Modules</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 hav- ing 0.990 reliabil- ity	0.99820	0.98807	0.95542
C	12	Redundant array with diodes hav- ing 0.999 reliabil- ity	0.99594	0.98361	0.94682
D	12	Redundant array with redundant short protection in TEM; no diodes	0.99569	0.98312	0.94587

category. Elimination of the diode device offers significant advantages in terms of reliability, compactness of design, and development costs. TEM-6 with the stainless steel sleeve sandwiched between two boron nitride sleeves provides a redundant internal protection against shorts to inner clad as in Case D.

The optimization studies will be continued to evaluate further the effect of the elimination of the diode device. Consideration of additional factors may result in an optimum array of parallel branches, each containing more than one tubular module in series; this represents a change from the present apparent optimum, i.e., using single 28-volt tubular modules in parallel.

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For study purposes, a series of Failure Cause Diagrams (FCD) has been constructed for a single thermoelectric module. The study considers four types of TEM breakdowns following satisfactory performance, then traces all conceivable chains of failures through a logic diagram to establish the condition or defect responsible for the breakdown. The FCD are being updated in accordance with comments from cognizant groups, the results of module evaluations, and anticipated design changes. A report reflecting the current FCD status will be issued during the next quarter.

Reliability personnel evaluated several modules which were opened after testing. This participation has proven useful in correlating the results of failure mode analyses, and further development of the failure cause diagrams.

A preliminary TEM qualification test plan was drafted and discussed with NYOO personnel. A revised version, incorporating results of the discussions and further study, will be completed in the next quarter.

B. QUALITY CONTROL

A detailed Non-Destructive Testing (NDT) Program Plan was developed during the first quarter of Phase II-A. The goal set forth within the scope of the NDT program was to identify and develop those non-destructive test methods and techniques which are applicable to the known and predicted thermoelectric module problems. The approach to attain this goal was to develop an experimental program plan of selected NDT methods, and to explore the state-of-the-art of these methods with techniques that would lead to a reliable evaluation of module materials and processes. The program plan, which requires that NDT responses are correlated with destructive evaluation of modules, is shown in Figure 2-1. The plan provides for the expansion of applicable NDT methods and techniques as Phase II-A progresses, as well as the deletion of those methods which are not applicable to the program.

Figure 2-1. Non-Destructive Testing Program Plan

A significant problem to be resolved during Phase II-A is the cracking found in the boron nitride insulation sleeves. To help in the identification of the processing operations responsible for initiating these cracks, Quality Control personnel developed an NDT method of detecting the discontinuities in the boron nitride sleeves in the assembled module. For this purpose, ultrasonic test methods were explored using shear wave techniques. In this procedure, the sound velocities of Inconel, boron nitride and iron were measured. As shown in Figure 2-2, calculations were made and plotted to give the optimum angles of acoustic incidence and refraction for shear wave propagation in these materials. The loss of acoustic energy at the interfaces through reflection is a function of the impedance of the various materials. The loss of energy in the acoustical path was calculated as follows:

$$\text{Impedance} = \text{Velocity (Shear)} \times \text{Density}$$

$$\text{Reflected Energy} = \left(\frac{\text{Impedance A} - \text{Impedance B}}{\text{Impedance A} + \text{Impedance B}} \right)^2$$

giving:

Acoustical impedance

$$3.02 \times 8.25 = 24.92 \quad (\text{Inconel})$$

$$1.95 \times 2.1 = 4.095 \quad (\text{Boron Nitride})$$

$$3.23 \times 7.8 = 25.29 \quad (\text{Iron})$$

$$1.49 \times 1.0 = 1.49 \quad (\text{Water})$$

and:

Reflected Energy Loss

$$\left(\frac{24.9 - 1.49}{24.9 + 1.49} \right)^2 = 78.8\% \text{ at } \text{H}_2\text{O}/\text{Inconel} \text{ Interface}$$

$$\left(\frac{24.9 - 4.09}{24.9 + 4.09} \right)^2 = 51.7\% \text{ at } \text{Inconel}/\text{BN} \text{ Interface}$$

$$\left(\frac{25.29 - 4.09}{24.9 + 4.09} \right)^2 = 52.1\% \text{ at } \text{BN}/\text{Iron} \text{ Interface}$$

$\frac{\sin \phi_1 \text{ (WATER)}}{\sin \phi_2 \text{ (INCONEL)}}$	$\frac{\text{VELOCITY (WATER)}}{\text{VELOCITY (INCONEL)}}$
---	---

$\frac{\sin \phi_1 \text{ (INCONEL)}}{\sin \phi_2 \text{ (BN)}}$	$\frac{V_1 \text{ (INCONEL)}}{V_2 \text{ (BN)}}$	$\frac{V_1}{V_{2S}} = \frac{3.02}{1.975} = 1.53$
		$\frac{V_1}{V_{2L}} = \frac{7.82}{3.95} = 1.98$

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Figure 2-2. Shear Versus Longitudinal Wave

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Special transducer probe designs, obtained through the Research Division of Automation Industries, were used in an attempt to resolve the circumferential cracks in the inner boron nitride tube. Such an arrangement is shown in Figure 2-3 in which a 5-mc crystal is mounted in a probe 65 degrees to the normal, and is inserted in the inside diameter of a module inner cladding filled with water. The probe was fixtured and driven by a micrometer screw and a variable speed motor as shown in Figure 2-4. The various angle beam probes used in these experiments are shown in Figure 2-5. Test equipment stability and a slow even drive arrangement for the probe were necessary to distinguish between the mica-iron interface and the boron nitride cracks. The iron conductor ring interfaces orient the exact location of cracks along the axial length.

The TEM-1 S/N-3 module suffered circumferential cracks in the inner boron nitride sleeve as disclosed by a longitudinal destructive cut which exposed the sleeve. This and other module sections were used as tentative standards since the cracks were visible and could be correlated with the ultrasonic test. These tentative calibration standards are shown in Figure 2-6.

The ultrasonic signals were recorded on a CEC recorder at slow speeds with the chart speed approximately six times that of the axial probe speed. Two full-length modules were ultrasonically tested with this arrangement before the end caps were removed. The recorder charts for TEM-3 S/N-5 and S/N-6 indicate that multiple cracks exist in the inner boron nitride sleeve of TEM-3 S/N-5, and possibly one crack in TEM-3 S/N-6.

On the TEM-3 S/N-5 ultrasonic recorder chart, predominant ultrasonic indications appear in the inner boron nitride sleeve in areas of the sleeve located within the length or between the following iron conductor rings starting at the positive end of the module: 3, 4, 6, 7, 12, 16, 18, 21, 23, 24, 25, 29-30, 32, 36, 36-37. These indications, which may be the result of circumferential cracks or concentrations of migrated lead, correlate very well with a potential plot made after destructive examination of a section of the module. A typical view of the ultrasonic test chart of TEM-3 S/N-5 is shown in Figure 2-7.

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Figure 2-3. Angles of Incidence and Refraction

Figure 2-4. Experimental Ultrasonic Test Stand

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Figure 2-5. Experimental UT Test Probes

Figure 2-6. Calibration Standards

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Figure 2-7. Ultrasonic Test Chart - TEM-3 S/N-5

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On the ultrasonic test recorder chart for TEM-3 S/N-6, only one predominant ultrasonic indication appeared in the boron nitride sleeve located within the length of the seventh conductor ring from the positive end.

In the preceding test techniques, the ultrasonic pulse echo reflected from the iron conductor ring interfaces was monitored, and gives a typical trace for each couple length. A crack in the inner boron nitride sleeve induces a back reflection with a shorter time interval than that from the iron and produces a recorded pattern typical of that in Figure 2-7. On the basis of these experiments, it appears that an ultrasonic technique has been developed to detect the presence of cracks or lead diffusion in the inner boron nitride sleeves of modules designed to TEM-3 configuration.

Concurrent with the development of ultrasonic shear wave techniques, a method of through transmission using two opposing crystals was considered. A tentative arrangement is shown in Figure 2-8 where two matched 5-mc transducers are angled in a probe such that one transducer sends an ultrasonic wave into the module and its reflection is received by the second crystal. By proper angulation and spacing, the focused beam will be reflected from the inside diameter of the iron conductor rings. The reduction in the pip height will indicate the interruption of the sound path such as would occur when the sound wave traverses a crack in the boron nitride sleeve. The initial through transmission probe is shown in Figure 2-5; a probe that will permit variable angles and spacing of opposing crystals is under development.

Axial cracks in the outer boron nitride sleeve were not detected until TEM-3 S/N-6 was fabricated. An ultrasonic test is being evaluated for this purpose. This test includes an angle transducer mounted on a contact plastic shoe that is contoured to the O.D. of the module. The ultrasonic shear wave will travel circumferentially around the module outer clad penetrating the boron nitride sleeve. Tentative plans are to do this test manually.

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Figure 2-8. Transmission Probe Design

Microwave Systems

The use of microwave systems for non-destructively examining insulating materials could possibly supply information not readily obtainable by other methods. Recent work in this area shows a strong potential for analyzing dielectric materials for moisture content, anisotropy, impurities, dielectric constant, internal flaws and thickness variations. The overall effectiveness of microwave technique measurements in the non-destructive testing field offers the following advantages:

- (1) Microwave systems are normally non-contacting.
- (2) Instrumentation and hardware can be conveniently arranged into various configurations, allowing numerous properties to be observed with the same basic equipment.
- (3) Microwave energy can be focused, reflected and transmitted over distances, and penetration is not dependent on thickness.
- (4) Results can often be correlated with some property which affects the performance of the part; such properties as density, tensile strength, homogeneity and energy absorption can be correlated.
- (5) Microwave absorption is a function of atomic and rotational moments as well as crystal lattice structure, and therefore has a strong frequency relationship. Some frequencies may be entirely absorbed by the material while others may pass through the material with negligible absorption or attenuation. In theory, therefore, minute quantities of certain contaminants (such as moisture) can be selectively detected.

Module insulation (particularly boron nitride) created problems during Phase I. Samples of boron nitride, therefore, were sent to the Microwave Instruments Company for evaluation to determine if any of the aforementioned advantages could be correlated to the problems experienced in module fabrication and testing. Preliminary tests have failed to determine the cause for the fracture in the boron nitride inner sleeve. Although microwave testing of boron nitride has not progressed to a stage where it may be considered acceptable,

certain capabilities of such tests have been reported. For example, microwave testing will:

- (1) Detect impurities to the extent of approximately 1% of the total material.
- (2) Determine moisture content in BN from about 1/2% and up.
- (3) Determine dielectric constant and dissipation factor in BN.
- (4) Provide measurement of tube diameters (accuracy unknown).
- (5) Measure tube wall thickness (accuracy unknown).
- (6) Indicate isotropy of solid material.
- (7) Not detect tight fracture planes in wall tubing.
- (8) Not measure dielectric strength.

Radiation

Penetrating radiation is defined as X-rays or gamma rays or particulate radiation having a wavelength less than one angstrom. Radiation absorption techniques were considered worthy of investigation in the non-destructive testing program. The techniques were based upon an energy-material interaction known as absorption which is basically a function of three factors associated with materials; namely, chemistry, density, and thickness. Absorption is described by the equation:

$$I = BI_0 e^{(-\mu/\rho) \rho t}$$

where: I = transmitted radiation intensity

I_0 = incident radiation intensity

t = thickness of material

ρ = density of material

μ = linear absorption coefficient

μ/ρ = mass absorption coefficient

B = the build up factor

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While the variety of materials and densities found in the module lend themselves to ideal radiation-absorption inspection, the geometry and thickness of the materials prevent the detection of minute discontinuities in an assembled module. The physical measurements that are within the capabilities of penetrating radiation techniques include axial alignment, interface spacing, distortion, thickness variations, and various degrees of internal discontinuities. Various sources of penetrating radiation which have been investigated are presented in the following paragraphs.

Low voltage X-ray up to 300 kv and 20 ma will give excellent resolution of the outer clad, thermal insulation, structural ends of the module, outer boron nitride sleeve, outer iron conductor rings, pins and the spacing between the couples. This energy will not permit resolution through the lead telluride couples. Film placed inside the inner liner failed to give the resolution necessary to see the cracks in the inner boron nitride sleeve.

High energy X-ray techniques included the evaluation of commercial 1 Mev X-ray equipment and several Betatrons rated at 22-24 Mev. The 1 Mev equipment will readily penetrate the module but radiation scattering within the module prevents clear resolution of the boron nitride inner liner. The higher energy from the Betatron served to give higher radiographic contrast, but this advantage was offset by poor definition. The module thickness and radiographic equivalency of approximately 3 inches of steel is at the minimum end of the Betatron range. Steel buildup over the module to bring the total thickness within a more favorable range failed to improve the definition of the inner boron nitride sleeve. A typical radiograph using the Betatron equipment is shown in Figure 2-9; which was made showing TEM-4 S/N-1 and a module from a previous program, serialized HEP 12.

Gamma emitting isotopes including Ir-192 and Co-60 were used in the investigation of penetrating radiation. The resulting radiographs were inferior to those produced by high energy X-ray. The scattering of radiation, the large focal spot size and low contrast resulted in loss of detail. No amount of masking improved the radiographic sensitivity, resolution or contrast.

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Figure 2-9. High Energy X-Ray

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No further results have been obtained from the investigation of neutron radiography reported in Phase I. An investigation of the "transfer method" is being carried out to evaluate the feasibility of producing a neutron radiograph of the mica spacers, the thermal insulation, and the boron nitride parts (without the masking effects) from the structural members and lead telluride. One of the great advantages of neutron radiography is that the relative absorption of thermal neutrons and gamma radiation in materials is very different. To eliminate the interfering gamma radiation, a radioactive screen is exposed to neutron radiation through the object in question. The screen is transferred to a film loaded cassette and allowed to decay. The neutron image will be transferred to the film without the interference from gamma radiation. A second method to produce a neutron image without interference from gamma radiation is to expose the part to a monochromatic neutron beam having an energy in the order of 0.05 ev. This beam is essentially free of gamma radiation. For transfer methods it is expected that 0.010 inch thick dysprosium metal screens will be used to gain the advantage of the high activation cross section for dysprosium.

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

DESIGN

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III. DESIGN

A. MECHANICAL

During February, the experimental program defined to explore the causes and solutions of module shorting was re-evaluated and crystallized. The evaluations resulted in changes in definition and scheduling of various development modules and module experiments.

As initially defined in the Project Plan, the module program for the first six months of Phase II-A was as follows:

<u>Module</u>	<u>Processing Complete</u>	<u>Definition</u>
TEM-3 S/N-5, -6, -7	2-28-66	Same as TEM-3 modules fabricated in Phase I. Objective is to help pinpoint the cause of cracks found in Phase I modules.
TEM-4 S/N-2, -3	3-15-66	Same as TEM-4 modules fabricated in Phase I. Objective is to help pinpoint the cause of cracks noted in Phase I modules.
TEM-5 S/N-1, -2, -3, -4	4-15-66	Redesigned on basis of above program.
TEM-6 S/N-1, -2, -3, -4	5-29-66	Redesigned on basis of above program.
TEM-X-A	4-25-66	Experiment designed to investigate uniform radial flexibility.
TEM-X-B	6-30-66	Experiment designed to investigate improved insulation.

As a result of observations and analyses of the shorting problems described in the Compact Converter Phase I Final Report (WANL-PR(CC)-006), the program has been revised to accelerate the influence of module experiments on the two groups of redesigned modules which

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were initially scheduled for assembly in April and May (TEM-5 and TEM-6). Also, as the result of a program decision to designate all modules (whether of a developmental or experimental nature) with a TEM number to preserve their chronological association, the module experiments previously designated TEM-X have been reassigned numerical code numbers. The design and fabrication schedule is set such that examination of TEM-5 will be possible before TEM-7 assembly is complete, thus enabling the experience gained from TEM-5 to influence the design of TEM-7. Similarly, TEM-6 is intended to influence TEM-8, and so on throughout the first six months of Phase II-A.

The revised program as currently defined is as follows:

<u>Module</u>	<u>Processing Complete</u>	<u>Definition</u>
TEM-3 S/N-5, -6, -7	3-1-66	Unchanged from above.
TEM-4A S/N-1, -2	4-22-66	Based on TEM-4 design except for limited dimensional and processing changes designed to eliminate faults encountered during processing of TEM-4 S/N-1 in Phase I.
TEM-5 S/N-1	3-25-66	Previously TEM-XA. This module experiment has been designed to eliminate the swaging requirement and includes various sealing experiments to prevent shorting to the inner clad.
TEM-6 S/N-1, -2	4-12-66	Previously TEM-XB. This module experiment is based on improvements to the TEM-4 design to investigate the effect of insulation changes on performance as well as the effect of minor design changes on swaging requirements.

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<u>Module</u>	<u>Processing Complete</u>	<u>Definition</u>
TEM-7 S/N-1, -2, -3	4-22-66	Based on TEM-5. This module experiment has been redesigned to incorporate improvements suggested by the TEM-5 experience.
TEM-8 S/N-1, -2, -3, -4	4-27-66	Based on TEM-6. This module has been redesigned on the basis of information developed during the design, fabrication, and testing of TEM-4A and TEM-6 modules.

Descriptions of the five module designs released during the first quarter of 1966 are given in the following paragraphs.

TEM-4A

TEM-4A has been selected as the designation of the TEM-4 modules to be processed through the gas pressure sintering cycle as part of the continuing investigation of the cracks encountered during Phase I in the inner boron nitride sleeve. These modules include several design and processing changes incorporated to determine if the Phase I type module can be fabricated without defects in the BN insulating sleeves. The changes are as follows:

- (1) The inner cladding is being made thicker by reducing the bore size from 0.500 to 0.250 inch. This will reduce the distortions which resulted when the outside diameter was cold-autoclaved and swaged to a uniform size. The bore will be opened to 0.500 inch following final processing of the module.
- (2) A clearance of about 3.5 mils (diametrically) will be provided between the boron nitride insulating sleeve and the inner clad. This will prevent boron nitride insulating sleeve cracking caused by differential expansion of the inner cladding and the boron nitride sleeve before the latter is placed into a state of hydrostatic compressive stress. These cracks were noted during destructive examination of TEM-3 S/N-7, which was destructively examined after the purging operation.

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- (3) The inner cladding will be sealed by welded plugs during the room temperature hydrostatic pressing operation. This change is being made to correct the leakage of the O-rings used for sealing TEM-4 S/N-1 during the pressing operation, which allowed full hydrostatic pressure to be applied to the bore of the inner clad.
- (4) The collector rings will be made more flexible to reduce radial stiffness at the end of the thermoelectric elements, thereby minimizing the need for swaging.
- (5) The dimensions of the mica washers were modified to reduce the gap between the mica and the iron rings.

Following fabrication and processing, the modules will be tested to establish thermoelectric performance, and will then be destructively examined. The results of the testing and examination will determine whether further investigation of TEM-4 type modules will be warranted.

TEM-5

In the Phase I Final Report (WANL-PR(CC)-006) the potential solutions for shorting of thermoelectric material to the inner clad were divided into three categories as follows:

- (1) Seal the PbTe material in place so that it cannot contact the insulating sleeves.
- (2) Eliminate cracking of the boron nitride sleeves.
- (3) Provide additional layers of insulation between the thermoelectric material and the inner clad.

These three broad categories summarize 11 discrete modifications to the TEM devices which were considered desirable near the end of Phase I. Seven of these changes were incorporated into the TEM-4 built at that time in spite of an exceedingly tight schedule. These changes are described in WANL-PR(CC)-006. The four concepts not included in TEM-4 S/N-1 are:

- (1) Increase the mechanical integrity of the boron nitride inner insulating sleeve by increasing its thickness, with a corresponding decrease in thickness of the iron conductor.

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- (2) Increase the mechanical integrity of the inner boron nitride insulating sleeve by reducing the radial stiffness of the end-regions of the module to more nearly match that of the active portion. This minimizes discontinuity stresses in the boron nitride insulating layers caused by physical discontinuities in the claddings.
- (3) Provide a non-conducting coating, such as an oxide film, on inner clad.
- (4) Provide additional layers of boron nitride rings between the inner iron rings and the boron nitride sleeve in order to force the diffusing PbTe to negotiate a tortuous path in order to reach the inner clad.

These four basic ideas, in addition to other proposed improvements, were incorporated into the TEM-5 module experiment as a continuation of the development effort begun in Phase I. The TEM-5 experiment combines several "sealing" arrangements which are designed to eliminate shorting by preventing the flow of lead-rich material radially inward to the inner clad. It is also intended to reduce cracking of insulating sleeves from discontinuity stresses by eliminating the need for mechanical swaging of the assembled module. This is accomplished with a more flexible end structure which will eliminate the sharp variations in radial stiffness experienced in previous TEM-4 type modules. This end structure and that for TEM-7 are illustrated and compared in Figure 3-5. As will be the case for TEM-4A, the TEM-5 module experiment was processed with the thicker inner cladding, and the ends of the inner cladding were sealed during hydrostatic pressing. Further, the outer clad of the TEM-5 was made 23 mils thicker than that for previous units. This is done to add slightly to the strength of the outer clad during operation. Since fabrication of the TEM-5 parts was completed before the problem of differential expansion between the inner clad and the inner boron nitride sleeve was isolated, an increased radial clearance could not be provided, and the inner clad of the TEM-5 was cooled during purging to prevent cracking of the boron nitride.

A brief description of the experimental seals incorporated in the TEM-5 experiment follows. Each sealing arrangement extends for about six couples along the inner cladding. These seals have been designed to help contain the PbTe inside the chambers formed by the iron rings

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and mica washers, thus providing a longer flow path to the inner cladding, if PbTe is not contained, and additional layers of electrical insulation.

Figure 3-1(a) describes a boron nitride seal ring with delta cross-section compressed between chamfered ends of adjacent inner conductor rings. While this arrangement is primarily a seal, the delta ring will also serve to protect the boron nitride insulating sleeve from direct loading by mica washers.

Figure 3-1(a). Seal Ring with Delta Cross-Section

Figure 3-1(b) shows a similar boron nitride ring in the shape of a cylinder fitted into a cavity machined into adjacent iron rings. While this ring is initially loose at assembly, it is expected that module processing operations will take up clearance between the ring and adjacent parts without causing the ring to crack. This configuration provides a seal and creates a longer diffusion path from PbTe washers to the inner cladding.

Figure 3-1(b). Seal Ring with Rectangular Cross-Section

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Figure 3-1(c) is an extension of the arrangement previously discussed. In this configuration, the boron nitride cylinders, which are approximately equal in length to the iron conductor rings, are staggered (as shown) to provide a longer path from the iron ring-mica joint to the inner sleeve of boron nitride. By this arrangement, the inner insulating sleeve is protected from direct loading by mica washers or the edges of the iron rings.

Figure 3-1(c). Seal Ring with Staggered Joints

Figure 3-1(d) shows a modification of Figure 3-1(c) in which thin rings of Armco iron are sandwiched between the boron nitride rings and the inner insulating sleeve. These iron rings guarantee a long path from the iron ring-mica joints to the inner insulating sleeve. In addition, the point at which the lead-rich material could finally contact the inner insulating sleeve is generally away from the location of cracks encountered in Phase I modules.

Figure 3-1(d). Seal Ring with Iron Inner Rings

Figure 3-1(e) shows a different approach taken to prevent shorting in this module experiment. In this case, a plasma spray is used to deposit a thin coating of Al_2O_3 on the inner cladding. In this concept, a short will occur only if penetration of the boron nitride insulating sleeve

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and the Al_2O_3 coincides. While it is not expected that the coating achieved for the TEM-5 experiment will be non-porous, valuable experience will be gained as the module is fabricated and processed with this coating in place.

Figure 3-1(e). Inner Cladding with Al_2O_3 Seal Layer

The TEM-5 module has been successfully cold-autoclaved at room temperature and 45,000 psig, and gas-pressure sintered at 750°C and 10,000 psig. The

results of cold-autoclaving and gas pressure sintering (GPS) shown in Figure 3-2 are of particular significance since the diameter is seen to be uniform within 3 mils after cold-compaction and within 4 mils after GPS over the portion of the module which includes the active and inactive washers of lead-telluride. This demonstrates the validity of the design changes made to decrease the radial stiffness of the regions adjacent to the thermoelectric stack, and makes progress toward the goal of eliminating the swaging of future modules.

After GPS, the TEM-5 end closures were machined away, exposing the Armco iron conductor sleeves. Electrical measurements showed a circuit-clad resistance of 5.5×10^5 ohms to the inner clad and in excess of 2×10^7 to the outer clad. While the resistance to the inner clad is far below the theoretical upper bound of resistance, based on the resistivity of pure boron nitride of about 10^{13} ohms, it is far higher than the circuit-clad resistance typically exhibited by prior modules.

Following these measurements, the module was cut into seven cylindrical pieces, separating the ends and the five individual seal experiments. Circuit-clad resistance was measured after each cut, and one interesting observation was that after one end had been removed, resistance between circuit and clad increased to over 10^8 ohms. This indicates that any significant current-leakage paths lay in one end region of the module, and that the seals were effective, at least through the GPS cycle.

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Figure 3-2. Outer Clad Outside Diameter Versus Distance from Positive End for TEM-5

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Preliminary examination of the end pieces revealed axial cracks in both boron nitride layers adjacent to the conductor sleeve. There appeared to be some contaminant in these fairly wide cracks, and investigation is continuing. Detailed data and photographs of the module sections are given in Section V of this report.

TEM-6

The TEM-6 is the second in the series of experimental module designs intended to investigate solutions to the problem of electrical shorting to the structural clad. It features the seal configuration from TEM-5 which is believed to have the highest likelihood of success (shown in Figure 3-1(d)) and this configuration is used over the entire active length of the module. The module also incorporates one highly significant new feature: an electrically isolated thin tubular iron membrane, sandwiched between the full length of the thermoelectric circuit and the inner clad. While this concept causes an increased temperature drop between hot fluid and thermoelectric material, it also assures positive electrical isolation of the thermoelectric circuit from the inner clad. The array of sleeves and seal rings in TEM-6 is shown in Figure 3-3.

Figure 3-3. Seal Ring Combined with Inner Clad in TEM-6

While the TEM-5 modules were redesigned to achieve more flexible end configurations, the approach taken on TEM-6 is to achieve improved flexibility starting with the TEM-4 concept.

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The principal changes were made in the vicinity of the iron collector ring, which was re-designed for increased flexibility. Thus, analysis of TEM-5 and TEM-6 will permit a comparison of the completely new end concept adopted for TEM-5, with an optimized version of the TEM-4 end design which formed the basis for Phase I modules. One of the two planned TEM-6 modules has been processed along with (and therefore identically to) the TEM-5 module described above. Its outside diameter after cold autoclaving was uniform over the length within the heavy welded flanges to within 7 mils as shown in Figure 3-4.

Because of this relative uniformity it was decided not to swage the TEM-6 modules. Following cold compaction, the first TEM-6 module was gas pressure sintered, after which its contour was found to be comparable to those of swaged Phase I modules. This offers encouragement that even the standard end design can be improved, by adjusting stiffness and initial clearances, to eliminate the need for swaging.

The conductor pins of this module were exposed, and the device prepared and mounted in a hot-test stand. Measurements showed a resistance of 2.4×10^5 ohms between circuit and clad before hot testing commenced.

Both TEM-6 module experiments will be tested for a short period to determine module performance, after which they will be destructively examined to determine the behavior of the new components during processing and testing.

TEM-7

The TEM-7 is the third in the series of development module designs, formerly called TEM-XA, -XB and -XC. The design is 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 will include the more flexible end designed for TEM-5, and the metal membrane for electrical isolation of the thermoelectric circuit tested in TEM-6.

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Figure 3-4. Outer Clad Outside Diameter Versus Distance from Positive End for TEM-6

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Consideration is being given to increasing the contact pressure between internal thermally conducting interfaces at operating conditions by shrinking a high strength sleeve over the outer clad after GPS. It will be recalled that the outer clad is made of annealed austenitic stainless steel so that its strength during GPS is low and the autoclave pressure can act to densify the lead-telluride and boron nitride. A high strength sleeve will increase the elastic strength of the outer shell of the module and permit an increase in internal pressure at operating temperature over the present calculated value of 6000 psi.

To compensate for the increased internal pressure, the thickness of the inner clad wall is being increased by 25 mils. This added thickness will also make the clad sturdier during the fabrication process. The ends of the TEM-7 module (as shown in Figure 3-5) were redesigned to reduce thermal losses below those expected for TEM-5. A new conductor sleeve, insulated thermally from the inner clad, is provided. This sleeve is held in a positive axial location by the axial restraint members welded into the outer clad. Fewer P-type inactive thermoelectric washers are used than in TEM-5, since fabrication experience and analysis of the "wave-length" of the tubular structure show this to be permissible. 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.

Based on experience developed during investigation of Phase I module shorting problems, and analysis of processing effects encountered during examination of TEM-3 S/N-5, -6 and -7, the annular clearances between boron nitride sleeves and adjacent metallic parts have been increased to prevent fracture caused by expansion of the metal during purging. This dimensional change eliminates the need for cooling the inner clad during purging. Three TEM-7 units are being manufactured.

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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	

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Figure 3-5. Flexible End Constructions Used on TEM-5 and TEM-7

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TEM-8

The TEM-8 is a continuation of the TEM-4, TEM-6 design series, utilizing the basic end configuration developed during Phase I. It differs from TEM-6 in that only the insulating membrane and continuous boron nitride sleeves shown in Figure 3-3 will be used; the segmented iron and boron nitride seals will not be incorporated. A thickened inner clad design identical to that of TEM-7 is specified. The outer clad will be thickened 23 mils over that of the TEM-1 to -4 designs (as on the TEM-5) and will have the possibility of utilizing a reinforcing sleeve; however, no sleeve will be specified until its application is well understood.

Insulating Coating Development

A program was initiated to investigate the possibility of utilizing an electrically insulating coating deposited directly on the inner clad. The purpose of the barrier is to prevent electrical shorts from extending from thermoelectric circuits to the inner clad. To achieve its purpose, the barrier material must present a low thermal resistance and a high electrical resistivity. A barrier program has been started to investigate several materials as possible candidates for the thin coating on the inner clad. The following items are included:

- (1) Plasma-sprayed alumina utilizing special powders.
- (2) Plasma-sprayed alumina with a final coat of boron nitride paint.
- (3) Boron nitride paint.
- (4) Plasma-sprayed boron nitride.

In addition, several other materials and/or methods were included in the original scoping phase of the program. One of these was low energy sputtering of boron nitride. (This method of applying boron nitride in a thin coating on the inner clad appears very promising. Investigation is temporarily held up pending receipt and checking out of special RF application equipment at the Westinghouse Research Laboratories.) Another was plasma-spraying of

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commercial-grade alumina. However, commercial grade powder contains considerable impurities which were found to be electrically conducting, thereby defeating the primary purpose of the barrier. Thus the latter effort was terminated.

The plasma-sprayed alumina, utilizing special powders, has also proved disappointing. A very fine alumina powder with 99.99% purity was plasma sprayed on several samples with various coating thickness of up to 0.003 inch. On the very thin coating (<0.001 inch), electrical shorts were noted over considerable areas. On thicker coatings (0.001 to 0.003 inch), at least one electrical short occurred at a very localized area on each sample. It was found that electrically conducting particles were still aligning themselves through the coating to produce an electrical path to the inner clad. This portion of the program was terminated for the time-being.

A very thin coating of plasma-sprayed alumina (0.0005 inch) was applied to an inner clad followed by a thin coating of boron nitride paint (0.0007 inch). This combination of materials has proved very satisfactory in atmospheric testing. The electrical resistance of this pair of coatings is in excess of 200,000 ohms. Several different types of boron nitride paint, supplied by the Carborundum Corporation, were tried, with the best results from types S and V.

Boron nitride paint was applied directly to a roughened inner clad surface. Again very high electrical resistance (> 2 megohm) was determined for coatings of 0.002 to 0.003 inch. The adherence of the coating did not appear to be as good as the adherence to the specimens which had a very thin primary coating of alumina. An order for boron nitride powder for a trial plasma spraying application has been placed. The anticipated initial results for this coating are not too good. It is felt that boron nitride should be sprayed in a dry-box. Presently, no facility of this type exists, but one should be available at the Westinghouse Plant in Lima, Ohio, in the near future.

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Module Inner Clad Replacement

A successful attempt was made to bore the inner clad from a short module section, preparatory to installing a new clad and boron nitride sleeve to repair shorts. This experiment served as a preliminary trial prior to attempting the same operation on a full length module. The clad and inner boron nitride sleeve were removed without damage to the adjacent layer of iron conductor rings. A procedure was devised for performing this removal and replacement operation on a full-length module, and TEM-2 S/N-1 was set aside for this trial. The special saws and extra-length drills required to perform the task have been received, and the trial operation will be performed in the near future.

B. ELECTRICAL

A study was conducted to ascertain the feasibility of determining the presence and location of short circuits that might have developed between the thermoelectric elements and the outer or inner clad of TEM-4 S/N-1 during operation. Because the module was already on test, it was desirable that this be accomplished by using the 20 chromel-alumel thermocouples which existed along the surfaces of the inner and outer clad of the module for the voltage measurements.

A series of voltage measurements between the clad and the various thermocouple leads were taken during the operating tests. After these tests were completed, a series of similar voltage measurements were taken, using a portable voltage probe and a separate d-c power supply to energize the module.

The results of the measurements taken during the operating tests were inconclusive because the thermoelectric emf generated between the clad and the thermocouple leads was of a much larger magnitude than the voltages resulting from any possible short circuits. Therefore, the desired information was masked. However, an analysis of the measurements indicated short circuits between the inner clad and the thermoelectric elements, but no short circuits to the outer clad.

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IV. ANALYSIS

A. THERMAL

Evaluation of Input Power Passing Through Active Portion of Module

Thermal analyses of the TEM-1 test configuration have been performed to determine the fraction of the electrical power input that enters the active length of the module. The analyses were based on the operating conditions for TEM-1 S/N-1 on October 5, 1965. Basically, two different approaches were used. The first consisted of multiplying the 1600-watt input power to the main heater by the ratio of active thermoelectric length to main heater length, and subtracting from this the calculated conduction heat transferred axially through the heater and NaK at the active length ends. This resulted in a value of 1495 watts for the thermal power entering the active length of the module. The second approach consisted of subtracting all of the calculated end losses from the total measured power input to the main heater and the guard heaters, which resulted in a value of 1555 watts. Averaging the results of these two approaches gives 1525 watts for the thermal power entering the active length of the module. Using this value, the correction factor is then calculated to be:

$$\frac{\text{Active Length Power}}{\text{Main Heater Power}} = \frac{1525 \text{ watts}}{1600 \text{ watts}} = 0.9531 \quad (\text{for TEM-1 S/N-1})$$

The variations in the active length of the various TEM modules may thus be accounted for by modifying the above equation as follows:

$$\frac{\text{Active Length Power}}{\text{Main Heater Power}} = 0.9531 \times \frac{\text{Active Length of TEM-X}}{\text{Active Length of TEM-1 S/N-1}}$$

The term to the right of the equal sign is the correction factor by which the power to the main heater must be multiplied to give the power into the active length, or by which the efficiency must be divided to give corrected efficiency. Table 4-1 gives the "correction factors"

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currently being applied as multiplication factors to the input power and overall efficiency of the various TEM series modules which have been, or are currently, on life test. Based on the two methods of thermal analysis described, the uncertainty in the correction factor is about $\pm 2\%$.

TABLE 4-1
TEM Input Power and Efficiency Correction Factors

Module	Active Length (In.)	Correction Factors	
		Main Heater Input Power (watts)	Module Efficiency (%)
TEM-1 S/N-1	7.193	0.9531	1.0492
S/N-2	7.095	0.9401	1.0637
S/N-4	7.086	0.9390	1.0650
TEM-2 S/N-1	7.035	0.9321	1.0728
S/N-2	7.034	0.9320	1.0729
TEM-3 S/N-1	7.043	0.9333	1.0715

$$\text{Efficiency Correction Factor} = \frac{1}{\text{Power Correction Factor}}$$

B. THERMOELECTRIC

Data Processing Code

In anticipation of the large volume of module test data to be generated during Phase II, a data handling code (designated DAHA) has been written for the IBM-7094 computer. With this code, certain preliminary hand calculations are replaced by computer calculations, thereby reducing the likelihood of error. Output from this code is in both printed and punched card form. The punched cards will be stored consecutively and used as source data for a plotting routine to provide, as required, a graphical display of various parameters of interest. This code has been

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written and debugged and will soon be incorporated as a regular part of the data acquisition, processing, and distribution procedure.

Diagnostic Code

A code is being prepared to analyze test data obtained from TEM modules and to diagnose the causes of degradation. The code requires as input that load curve information (load voltage, source temperature, sink temperature, and thermal power input as functions of load current) be obtained at various times during the life of the module. The module is assumed to be operating at nominally constant thermal power input and sink temperature.

The analysis is based on the assumption that the average Seebeck coefficient and module thermal conductance are linear functions of average module temperature and that the module resistance is an exponential function of average module temperature. These functional relationships should be valid for the temperature variations ($\pm 25 \text{ K}^{\circ}$) that will occur due to the varying load current. For each data point on the load curve, four observation equations are obtained. By taking 10 to 20 data points, a total of 40 to 80 equations become available. Since there are eight unknowns to be determined (two each for the three macroscopic thermoelectric parameters and one for each of the thermal contacts to the cladding), the system is overdetermined and can be solved by the Method of Least Squares. The diagnostic code will solve these equations, determine the standard deviation for each parameter, and reject obviously erroneous data points by the application of Chauvenet's Criterion. By correlating the time dependence of these eight parameters with the observed behavior of a module, the causes of degradation can be investigated. The mathematical analysis has been completed and the code is now being programmed.

Improved Design Calculations

In order to determine the range of validity of the analytical approximations used in the presently available thermoelectric design codes, a new analysis is being formulated which includes any arbitrary temperature dependence of the thermoelectric properties, α , ρ and k ,

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and calculates the exact solutions to thermoelectric problems.

The method of calculation is essentially that which was developed by Sherman, Heikes and Ure⁽¹⁾ and modified by Cohen and Abeles⁽²⁾ but with the formulation revised to account for the cylindrical configuration of the tubular module. In addition to the geometric revisions, the new formulation also considers the effect of conductor resistance, conductor-thermoelectric material interface resistance, and shunt heat losses through the inter-element insulators.

Tubular Module Testing

In the testing of a tubular thermoelectric module the parameters which may be measured are limited to hot side and cold side temperatures, load voltage, load current, and thermal power inputs to the main and guard heaters. Parameters used in the design of the module are: the Seebeck coefficient, electrical resistivity and thermal conductivity of both the p- and n-type thermoelectric materials as functions of temperature; the thermal conductivities of the claddings, the iron conductors, and the mica insulators as functions of temperature; the electrical resistivities of the conductors and the conductor-lead telluride interfaces on both the hot and cold sides of the module; and the heat transfer characteristics of the boron nitride electrical insulation and its interfaces with adjacent metallic components.

To effectively use test data to evaluate design methods and/or perform diagnostic analysis of the modules, one must know, or be able to infer accurately from the test data, each design parameter. A review of testing methods has shown the following general ground rules to be applicable:

- (1) Testing of the modules at constant thermal power input is advantageous over testing at constant T_H and T_C because it is experimentally easier to maintain constant power during life tests, and the evaluation of test results as a function

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1. Sherman, B., Heikes, R. R., and Ure, R. U., Jr., J. Appl. Phys, 31, 1 (1960)
 2. Cohen, R. W., and Abeles, B., J. Appl. Phys., 34.

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of time is less sensitive to the techniques of averaging clad temperatures.

- (2) The relative accuracy of the data must be to four significant figures in order to infer from test data small variations of the design parameters as a function of operating time.
- (3) Since only a limited number of modules are to be tested, all should be instrumented such that diagnostic information is available. In addition, sufficient data points should be taken during the test period to evaluate the statistical accuracy of the test data and to avoid irretrievable loss of data.

There are several tests which are typical of those which will be required to achieve an understanding of the behavior of tubular modules. These typical tests are listed in the subsequent paragraphs. As the test program progresses, it may be desirable to add or delete from this listing, or to modify the tests, in order to realize design and performance objectives.

Room Temperature Resistance Measurements

Prior to any appreciable application of temperature to the module after completing fabrication, the internal resistance of the module and the resistance to clad from both the positive and negative terminals should be measured at room temperature isothermal conditions by a-c techniques.

Effective Seebeck Coefficient of Module Near Room Temperature

This test will provide an indication of the as-fabricated state of the thermal interfaces within the module and, after sufficient tests have been performed and the data evaluated, may provide a basis for establishing future Quality Control standards on the fabrication processes.

For these tests hot water, between room temperature and boiling temperature, provides a heat source on the hot side. Cold water passing over the cold side is the heat sink. Measured parameters are the inlet and exit water temperatures for both flow channels, and the open circuit voltage.

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Heater Traverse Test

The purpose of the heater traverse test is to give indication of the as-fabricated state of the module at various locations along the axis. Cold water flowing over the outer clad provides a heat sink. A short heater (recommended to be 1/4 inch long) is used to traverse along the inner clad axis of the module. At each position the open circuit voltage is measured after establishing thermal equilibrium. The axial variation of these open circuit voltage measurements is then an indication of the local as-fabricated state of the module.

Warm Up Test

After the module is installed in its life test fixture with appropriate heaters, fins and instrumentation, it is warmed up to the operating test conditions in a controlled manner. The change in temperature between the points at which the module comes to equilibrium should be no more than 25 K^o. After equilibrium is established at each of these steps, the temperatures, open circuit voltage, internal resistance, resistance to clad, and thermal power inputs are recorded. This test should be performed under conditions of open circuit so that the internal resistance of the module can be measured by a-c techniques. Discontinuities and changes in slope of the open circuit voltage, the internal resistance, and the thermal impedance of the module during escalation of temperature will indicate changes which may occur in contact interfaces and within the module.

Life Tests

A life test can be performed in a number of ways; the test procedure selected is determined by the information desired from each particular test. Two specific life test procedures have been considered. Both procedures will assume that the test is performed under constant thermal power and constant cold side temperatures.

Open Circuit Tests

In an open circuit life test, the module is run continuously with no power being drawn from the module. Data to be recorded include open circuit voltage, thermal power input, temperature

distribution, internal resistance, and the leakage resistance to clad. Changes in the open circuit voltage will be indicative of changes in the thermal contacts within the module, changes in the bulk temperature of the thermoelectric material, or multiple shorts to clad. Changes in the internal resistance can be attributed to cracking of the thermoelectric materials, change in bulk temperature of the thermoelectric material, or changes in the electrical contact resistivity between the thermoelectric material and the conductors. If multiple shorts occur to clad, their occurrence should be reflected in decreased internal resistance of the module. In addition, changes in thermal contacts will be reflected as changes in thermal impedance. Although power output and efficiency are not measured directly, they may be computed from these data with an accuracy sufficient for engineering evaluation of module performance. The major advantage of open circuit testing is that the electrical and thermal properties of the module are much less strongly coupled to each other and may be analyzed separately for diagnostic purposes.

Load Tests

After warmup, load may be applied by introducing a load resistance into the open circuit and increasing the thermal power to compensate for Peltier heat transfer. Matched load is realized by adjusting the load resistance to exactly the internal resistance of the module. Data recorded include thermal power input, temperature distribution, voltage to load, load current, and leakage resistance to clad. Load curve data points must also be taken at various times during the test to obtain diagnostic information to determine the tubular module design parameters as a function of time.

Curve Fitting of Efficiency Degradation Data

An attempt was made to fit the life test data of TEM-1 (S/N-1 and -2) modules to an equation of the form

$$\eta = \eta_1 + \eta_2 e^{-\frac{t}{\tau}}$$

where:

η_1 = asymptotic efficiency approached at high operating times

$\eta_1 + \eta_2$ = efficiency at beginning of life ($t = 0$)

τ = time constant for $\frac{1}{e}$ degradation

t = time at life test conditions

By using a least squares method, and rejecting data on the basis of Chauvenet's Criterion, the equation was found to be a reasonable fit for data obtained on modules tested prior to the Compact Converter program.

Three attempts were made to fit the TEM-1 data to the curve after 500, 1300 and 2400 hours of operation. Since the standard deviations showed increasing divergence with time, it can be concluded that the TEM-1 degradation does not fit this curve form.

Because the TEM-1's were tested at constant temperature, another attempt will be made to fit the curve to data obtained during Phase II-A testing of modules at constant input power.

Comparison of Performance with Different Materials in P-leg

The material being employed in the P-leg of the TEM-series thermoelectric couples is 3M type TEGS-2P. Because this material is purported to be somewhat unstable at high temperatures, preliminary calculations were made to determine the relative performance of this material and the more stable 3M type TEGS-3P. Using TEM-1 series couple dimensions and 1100°F hot clad temperature, the overall efficiency, electrical power output and open circuit voltage were calculated as a function of cold clad temperature. This information is presented graphically in Figures 4-1 through 4-3 for modules identical except for the different P-type materials. Optimizing of internal dimensions with respect to efficiency was also done for couples using the two different P-type materials; this information is presented in Table 4-2. As can be seen, the use of -3P material would entail about a 7-1/2% reduction in overall efficiency and about 15% reduction in power output.

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Figure 4-1. Overall Efficiency Versus Average Temperature of Outer Clad

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Figure 4-2. Open Circuit Voltage Versus Average Temperature of Outer Clad

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Figure 4-3. Total Electrical Power Output Versus
Average Temperature of Outer Clad

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TABLE 4-2
Comparison of Single Couple Performance Using
TEGS-2P and TEGS-3P with $\bar{T}_H = 1100^\circ\text{F}$ and
 $\bar{T}_C = 400^\circ\text{F}$

<u>Parameter</u>	<u>TEGS-2P</u>		<u>TEGS-3P</u>	
	<u>TEM-1 Dimensions</u>	<u>Optimized Dimensions</u>	<u>TEM-1 Dimensions</u>	<u>Optimized Dimensions</u>
Overall Efficiency (%)	6.159	6.742	5.679	6.240
Open Circuit Voltage/Couple (volts)	0.170	0.172	0.150	0.155
Matched Load Current (amps)	35.40	73.11	34.71	68.43
Internal Resistance/Couple (ohms)	0.00237	0.00118	0.00215	0.00113
Electrical Power Output/Couple (watts)	2.974	6.304	2.601	5.297
Thermal Power Input/Couple (watts)	48.29	93.50	45.81	84.89

Thermoelectric Module Performance

Some detailed calculations have been made with the computer code, CPBTE2, to determine the improvements which could be expected in overall efficiency of thermoelectric modules due to certain changes in dimensions and operating temperatures. A summary of the efficiencies obtained from these calculations is given in Table 4-3. It is important to realize that the cases presented were optimized with respect to efficiency; thus, other parameters such as voltage, current, power output, and power input would not be the same as those predicted in other studies for a fixed set of dimensions. The values are for "beginning-of-life" and no attempt was made to project the efficiencies to some later time.

TABLE 4-3

CPBTE2 Computer Code Efficiencies

<u>Item</u>	<u>Module</u>	<u>Operating Temperature ($^{\circ}$F)</u>	<u>Design Change</u>	<u>Efficiency (%)</u>
1	TEM-1	$\bar{T}_H = 1000^{\circ}\text{F}$ $\bar{T}_C = 392^{\circ}\text{F}$	Baseline	5.61
2	Redesigned TEM-1	$\bar{T}_H = 1000^{\circ}\text{F}$ $\bar{T}_C = 392^{\circ}\text{F}$	Same as Item 1 except 0.0025 mica in place of 0.005 mica	6.34
3	Redesigned TEM-1	$\bar{T}_H = 1200^{\circ}\text{F}$ $\bar{T}_C = 425^{\circ}\text{F}$	Same as Item 2	6.94
4	Redesigned TEM-1	$\bar{T}_H = 1200^{\circ}\text{F}$ $\bar{T}_C = 425^{\circ}\text{F}$	Same as Item 2 except couple dimension optimized	7.20
5	Reference TEM	$\bar{T}_H = 1200^{\circ}\text{F}$ $\bar{T}_C = 425^{\circ}\text{F}$	Increased diameter module based on Item 4	7.57

C. STRESS

Stress analysis effort initiated during the first quarter of Phase II-A included the following:

(1) analyses to provide qualitative explanations of the cracking of the boron nitride, (2) analyses to provide a background and to assist in improved material selections and fabrication processes, and (3) a seven-layer stress analysis using an available computer code. Details of several tasks in these areas are presented in the following paragraphs.

Cracking of the BN Sleeve

The circumferential cracking of the inner BN sleeve at the mica washer region was analyzed in relation to the stresses in this sleeve during fabrication and operation. In Table 4-4, the

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strength properties of BN are given for the fabrication temperatures.

TABLE 4-4

Test Ultimate Strength for BN

<u>Test Direction</u>	<u>Module Direction</u>	<u>Tensile Strength (psi)</u>			<u>Compressive Strength (psi)</u>	
		1292°F	932°F	68°F	1292°F	68°F
Longitudinal (parallel to pressing direction)	Axial	570	1400	6200	3400	—
Transverse (perpendicular to pressing direction)	Tangential Radial	620	2100	—	4000	36,000

The results of the analysis show the following stresses in the sleeve for comparison with Table 4-4:

- (1) During swaging at room temperature, the mica washers between couples are displaced radially, and press the inner BN sleeve against the inner cladding to cause the following stresses: 24,000-psi tangential compression and 6,500-psi axial bending. These values were obtained for the instant that the 0.001-inch inner radial gap of the sleeve was closed. Since this axial bending can cause failure, the design of modules after TEM-3 was modified to provide greater radial clearance for the mica washers.
- (2) During sintering at 1382°F and 10,000-psi external pressure, the compaction introduces high compressive stresses of 7,500-psi radial compression and 2,500-psi axial and tangential compression. These stresses cause the BN sleeve to flow and conform to the mating surfaces. Relaxation of the BN at the sintering temperature causes these stresses to equalize at about

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5000-psi compression. This isostatic pressure is retained when the module is brought to ambient conditions after the processing.

- (3) When the module is heated to operating conditions (the BN sleeve being approximately 990°F), stresses of 1,800-psi axial tension and 1,500-psi tangential tension are superimposed on the residual stresses. These stresses are above the tensile strength and would result in fracture if the residual processing compression stresses were completely relaxed. Measurements of interface pressures after processing show that this is not the case.

It was noted in the compression tests of BN specimens that failure normally occurred by separation of the sample in layers perpendicular to the pressing direction (the weaker direction as shown in Table 4-4). The preceding analysis shows that the relative tensile stress is higher in the axial direction. The BN sleeves are currently being machined with the pressing direction coinciding with the axial direction (the weaker direction as given in Table 4-4).

Radial Stiffness of TEM-1 Type Modules

To increase interface pressures, a modification is contemplated in which a strong stainless steel sleeve will be shrunk over the outer clad. To provide a guide in this design, the radial stiffness of a fully compacted TEM-1 was calculated for external pressure. The results are shown in Figure 4-4. The relationship between radial deformation and external pressure is nearly linear for the range of interest. At room temperature the pressure required to reduce the outer radius elastically by 0.0005-inch is 7,700 psi, or an equivalent spring constant of 15.4×10^6 psi/in. for the radial direction over the active length. The analysis further indicates that at the inner radius of the outer clad the interface pressure increases by 70% of the external pressure applied by the additional sleeve.

Clad Strength Versus Contact Pressure at Operating Conditions

The capability of holding the contact pressure by action of the inner and outer clads during operation is of primary importance to the design. Calculations have been made using the

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Figure 4-4. Reduction of Outer Radius Due to External Pressure

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three-cylinder simplified model for various combinations of operating temperature and clad dimension on the basis of TEM-1 design. Figures 4-5 and 4-6 show typical results. The primary aim in developing the data was to give criteria for inner and outer clad yielding and contact pressures for various thickness clads. To better understand the effect of clad thickness, it was useful to assume perfect material elasticity in the clads. Then they were interpreted in an elastic-plastic analysis using the yield strengths of the material.

In summary, these curves show the following:

- (1) Contact pressure increases rapidly with the hot side temperature due to differential thermal expansion of the components. At a given temperature, the ratio of the outer radius to the inner radius of the outer clad (b_7/a_7) has greater effect on the contact pressure than that of the inner clad. At a given temperature and radius ratio, a larger contact pressure can be developed with the inner clad in the axially restrained condition than in the free condition.
- (2) At a given temperature, the yielding pressure of one clad increases with the increase of the radius ratio of the same clad. The effect of increasing the ratio of one clad upon the other is different in the axially restrained condition (Figure 4-6). A larger ratio of the outer clad makes the yielding pressure of the inner clad also larger, while a larger ratio of the inner clad reduces slightly the yielding pressure of the outer clad. These conclusions are due to the effect of axial restraint on the stress state of the clads. In the axially free condition, the change in ratio of one clad has no effect upon the yielding pressure of the other.
- (3) The relations represented in Figure 4-6 can also be used as a criterion to determine when outer and inner clad yielding occurs. If the contact pressure line lies above the clad yield pressure line, the clad has yielded. Therefore, for the material constants and dimensions given in Figure 4-6, the outer clad has yielded for all ratios of outer clad thickness shown. The inner clad, however, yields only for the larger outer clad thicknesses and thinner inner clad values.

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Figure 4-5. Contact Pressure Versus Inner Clad Temperature for $b_1/a_1 = 1.199$

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Figure 4-6. TEM Contact Pressure and Clad Yielding Pressure at Operating Temperature, $T_H = 1025^\circ\text{F}$ and $T_C, 395^\circ\text{F}$ Axially Restrained

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The contact pressure lines, which are based on the assumption that both clads are elastic, serve as a reference to evaluate changes in outer cladding dimensions. Actually, the contact pressure can never exceed the outer clad yield pressure. The yield pressures have been ratioed from the elastic calculations to account for the clad yield strength, while the contact pressure has not been changed. For the curve shown in Figure 4-6, when yielding of the outer clad occurs, the contact pressure is somewhat below the outer clad yield pressure lines. This curve, together with others developed for other temperatures, can serve as design guides.

Lower Bound Interface Pressure During Operation

The three-cylinder simplified method predicts only the average contact pressure on the composite material contained between the two clads and the average stresses in each clad. During operation, however, the contact pressure at each interface should be sufficient to form a good thermal and electrical contact. An attempt has been made with a seven-layer model to evaluate the initial interface pressure distribution across the radius during operation at 1025° and 395°F of inner and outer clad temperature, respectively. The results are shown in Figure 4-7. The calculations were based on the condition that the residual interface pressures resulting from the sintering process are neglected. Therefore, the results may be considered as the lower bound initial stress pattern in the module during operation. It is noted that the contact pressure peaks at the iron-lead telluride hot interface and decreases to the lowest value at the boron nitride outer clad interface.

A line has been drawn on Figure 4-7, showing the contact pressure previously calculated for the three-cylinder simplified model when the effect of outer clad yielding and residual stress from the sintering process are included.

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Figure 4-7. Radial Interface Pressure of TEM-1 at Operating Temperature,
 $T_H = 1025^{\circ}\text{F}$, $T_C = 395^{\circ}\text{F}$

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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. STATUS OF MODULE FABRICATION

A discussion of the fabrication status of each of several TEM series modules is presented in this section. Component fabrication and processing as well as module assembly and processing are, in general, the same as described in the Phase I Final Report, NYOO-3584-1, WANL-PR(CC)-006, with specific exceptions as noted.

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

In an attempt to identify the roles of various process steps on the development of short circuits to clad and other internal defects, three TEM-3 modules (WANL Drawing 909E682) were assembled and fabricated through various stages of the overall sequence. S/N-7 was fabricated through the hydrogen purge-vacuum degassing process; S/N-6 through the swaging process; and S/N-5 through gas pressure sintering at 10,000 psi and 750°C. Destructive examination and analysis of these modules is in progress and is discussed later in this section.

TEM-4A S/N-1 and -2

This design (WANL Drawing 909E684) represents a slight modification to the TEM-4 module fabricated and tested at the end of the Phase I program. The TEM-4A S/N-1 and -2 modules will be completely fabricated, non-destructively examined for defects, tested, and then destructively examined. All components for these modules have been fabricated and processed, and module assembly has been completed.

TEM-5 S/N-1

This module experiment (WANL Drawing 910E055) contains a number of design changes intended to eliminate shorting between the thermoelectric circuit and the module inner clad. It also includes an experimental end assembly, intended to make the response of the assembled

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module to radial deformation more uniform along most of its length. After the components were fabricated and processed, the module was assembled, axially compressed and welded. It then was given a modified hydrogen purge-vacuum degassing treatment, in which the module bore was water-cooled during the entire cycle. This was done to limit the radial thermal expansion of the inner clad, because the results of the destructive examination of TEM-3 S/N-7 indicated that such expansion may cause cracking of the inner boron nitride insulating sleeves. Due to equipment limitations, it was necessary to limit the module average outer surface temperature to about 325°C with a bore temperature in the range 80° to 250°C during the cycle. The decision was made that this deviation from the standard process was not of major importance for this module, since it is essentially a physical and geometric experiment and is not intended for performance testing.

The module was cold hydrostatically compacted at 45,000 psi with the pressure applied only to the outer surface and the ends. The resultant outer diameter profile is shown in Table 5-1. Overall elongation was about 0.008 inch. No swaging was performed on this module. After cold hydrostatic compaction, it was gas pressure sintered at 10,000 psi and 750°C. Non-destructive testing and destructive analysis of the module is in progress.

TABLE 5-1
Outside Diameter Profile of TEM-5 S/N-1 After Room Temperature
Hydrostatic Compaction at 45,000 psi
(Initial Uniform OD = 1.670 In.)

<u>Distance from (+) End (In.)</u>	<u>Average OD (In.)</u>	<u>Distance from (+) End (In.)</u>	<u>Average OD (In.)</u>
0	1.643	6	1.647
1	1.647	7	1.648
2	1.648	8	1.648
3	1.646	9	1.648
4	1.647	10	1.646
5	1.647	11	1.645

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TEM-6 S/N-1 and -2

The TEM-6 design (WANL Drawing 910E056) contains innovations in the inner insulation layer to eliminate short circuiting between the thermoelectric circuit and the module inner clad. All components for these modules were fabricated and processed. S/N-1 was assembled, but one of the inner insulating members was damaged during axial compression of the assembly prior to welding. The module could not be disassembled at this point, and it was necessary to delay further fabrication while some special fixtures and tools for repairing the damage were designed and manufactured. After fabrication is completed, S/N-1 will be tested to establish the effect of the anti-shortening experiments on module performance, after which the module will be destructively examined.

S/N-2 was assembled, axially compressed, welded and given a modified hydrogen purge-vacuum degassing treatment, similar to that for TEM-5 S/N-1. In this case, the average outer surface temperature was about 400°C and the bore temperature in the range from 25° to 175°C. The module was cold hydrostatically compacted at 45,000 psi with the pressure applied only to the outer surface and the ends. The resultant outer diameter profile is shown in Table 5-2. Overall elongation was 0.010 inch. This diameter profile compares extremely well with profiles of previous modules after swaging, and it was therefore decided that swaging was unnecessary. This is very encouraging because the elongation which results from cold compaction is almost negligible compared with that usually experienced as a result of swaging. It is anticipated that defects arising from axial shifting of conductor rings with respect to PbTe washers and from axial elongation of PbTe washers during densification will be substantially reduced. After cold compaction, the module was gas pressure sintered at 10,000 psi and 750°C. Non-destructive testing and analysis are in progress and preparations for the performance check prior to destructive analysis are under way. As noted above, during assembly of TEM-6 S/N-1 the inner insulating members were damaged by an assembly tool. Unfortunately, S/N-2 had already been assembled when the damage was discovered in S/N-1 and it is possible that S/N-2 has damaged insulation.

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TABLE 5-2
Outside Diameter Profile of TEM-6 S/N-2 After Cold Hydrostatic
Compaction at 45,000 psi
(Initial Uniform OD = 1.625 In.)

<u>Distance from (+) End (In.)</u>	<u>Average OD (In.)</u>	<u>Distance from (+) End (In.)</u>	<u>Average OD (In.)</u>
0	1.603	7	1.600
1	1.604	8	1.600
2	1.603	9	1.601
3	1.601	10	1.604
4	1.600	11	1.608
5	1.599	12	1.606
6	1.599	12.4	1.597

TEM-7 S/N-1, -2, and -3

This design (WANL Drawing 910E057) is based on the TEM-5 design primarily, which makes use of a flexible end assembly configuration to improve the uniformity of response to radial deformation along the module length. Three modules are being fabricated, with two intended for performance testing and one to be removed from test and destructively examined after a short performance run. The PbTe washers for these modules have been fabricated, processed and placed in vacuum storage. All other components have been ordered and are in various stages of fabrication and processing.

B. DESTRUCTIVE EXAMINATION OF MODULES

TEM-3

Three TEM-3 modules have been fabricated and destructively examined at various stages of processing to investigate the cause of boron nitride insulating sleeve cracks observed during Phase I. The modules have been assigned serial numbers as follows:

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- (1) S/N-7 — assembled, welded and hydrogen purged (400°C)
- (2) S/N-6 — assembled, welded, hydrogen purged (400°C) and swaged
- (3) S/N-5 — assembled, welded, hydrogen purged (400°C), swaged and gas pressure sintered (750°C , 10,000 psi).

The principal steps and methods of destructive examination for these modules are illustrated in Figures 5-1 through 5-3. S/N-7, since it had not progressed beyond purging, was disassembled axially, component by component.

Examination of the inner insulating sleeve of this module revealed one long axial crack extending from one end almost to the midpoint of the active thermoelectric length, and a short axial crack extending about $1/2$ inch inward from the opposite end. Both ends of the inner insulating sleeve had some angular cracks extending around the periphery from the axial crack. All of the metallic module components were clean and bright with no traces of discoloration from impurity gases or PbTe transport during the purging cycle.

Visual examination of the inner boron nitride insulating sleeve and the inner clad revealed a pattern of discoloration. This discoloration indicates that differential thermal expansion during the purging cycle caused the inner cladding to expand to a degree where it compressed the boron nitride insulating sleeve against the inner iron rings. This is sufficient differential thermal expansion to cause axial cracks in the boron nitride insulating sleeve, which is one mode of failure that had been suggested as a result of the analysis made of the Phase I program.

TEM-3 S/N-6, with the outer BN sleeve removed and some of the inner components peeled away (as shown on Figure 5-2), was observed to have a continuous helical-type crack in the outer sleeve and some PbTe-like surface contamination of that sleeve. The remaining inner components appeared clean and contamination free. At this point of the destructive examination, the inner BN sleeve does not appear to be cracked. Destructive examination of this module will be completed with the examination of the axial section of the remaining thermoelectric couples and end assembly.

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Figure 5-1. TEM-3 S/N-5 Module

Figure 5-2. TEM-3 S/N-6 Module

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

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Laboratory

TEM-3 S/N-5 has been processed through the entire fabrication cycle and sectioned (as illustrated in Figure 5-1) into one axial and several transverse cuts for special tests. One test included cutting open the full cross-section with strain gages attached. The axial view indicates the presence of radial cracks in the inner BN insulating sleeve and slight penetration (without radial cracks) in the outer BN insulating sleeve after gas pressure sintering, as was observed previously on the TEM-1 and TEM-3 type modules of Phase I. Work is being continued on the module sections to classify the movement of PbTe-type materials.

Three significant observations have been made in relation to the TEM-4 S/N-1 unit now undergoing examination after destructive testing of one section of the module. These are as follows:

- (1) The inner boron nitride sleeve contains one axial crack, filled with conducting material. Additionally, several radial cracks (located within two inches of the single end examined) are filled or partially filled with conducting material. The incidence of radial cracks appears to be considerably less than in the TEM-3 modules previously examined.
- (2) The PbTe washers do not appear to be fully densified, and bonding between concentric washers is not good. Accordingly, density measurements are being made on washers extracted from the module.
- (3) Axial and radial relaxation measurements taken during destructive machining of the module indicated that the internal interface pressures were much lower than in previous TEM modules.

Examination of TEM-4 is continuing, and results will be summarized in later reports. Points (2) and (3) above confirm the earlier conclusions which indicated that the modified gas pressure sintering cycle (650°C , 7,500 psi) did not achieve full compaction of this module. Consideration of this fact has served as the basis for continuing to use the 750°C , 10,000 psi cycle until conclusive evidence can be gained from the capsule tests being conducted in the materials program.

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TEM-5

TEM-5 S/N-1, an experimental module described in detail in the Design section of this report, was sectioned by making radial cuts across the interface of each array shown in Drawing 910E055, and across the end (inactive) sections. The ends of each section were then machined perpendicularly to the module axis and metallographically ground for macro-photos. These macrophotos are used for preliminary examination for detection of cracks present in the BN, or movement of PbTe-type materials. Considerable effort remains before examination of this module is complete, but some of the preliminary observations are typified by Figures 5-4 and 5-5(a) and (b).

Figure 5-4 shows a cross section through the positive end of Array B (Drawing 910E055). No cracks are observed in the outer BN sleeve. Although there is one defect present in the inner BN sleeve (see arrow), there is no evidence of general penetration of the BN.

Figure 5-5(a) (Array H, Drawing 910E055) shows a clean crack-free outer insulating sleeve, but a double inner BN layer with the innermost layer having a small group of cracks (top arrow), filled with a PbTe-type material. In addition, there appears to be a penetration of the surface of the innermost layer (right arrow) that is typical of PbTe-type penetration. Figure 5-5(b) is an enlargement of a segment of Figure 5-5(a). On this photo, the cracks and penetration are more obvious. Further detailed examination is required to verify material identification and the exact nature and location of all such observed defects. It is interesting that in several instances of observed cracks in a double BN sleeve the cracks did not appear to propagate across the BN/BN interface.

After the examination of these radial cuts is completed, the next step will be to prepare axial cuts through each section to examine the alignment of parts, and to look for additional evidence of cracking and PbTe transport in the plane normal to the ones currently being examined.

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Figure 5-4. TEM-5 Cross-Section (Array B, Drawing 910E055)

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(b)

(a)

Figure 5-5. TEM-5 Cross-Section (Array H, Drawing 910E055)

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TESTING

VI. TESTING

A. STATIC PERFORMANCE TESTS

The results of continuing tests on the TEM-1 S/N-1, -2 and -4 modules are shown in Figure 6-1. The time indicated is the total hours of continuous operation after the initial 500-hour seasoning period. Hence, the total time on test is actually 500 hours more than that plotted in the figure. The efficiencies shown have been corrected by factors listed in Section IV of this report. It should be noted that TEM-1 S/N-4 is operating at 1100°F hot side temperature after having been thermally cycled as shown, whereas TEM-1 S/N-1 and -2 are operating at 1000°F without having been cycled. The present electrical characteristics of the modules on test are listed in Table 6-1.

TABLE 6-1

The Electrical Output Characteristics of TEM-1 Modules

	<u>S/N-1</u>	<u>S/N-2</u>	<u>S/N-4</u>
V_{oc} (volts)	5.76	5.88	6.37
V_L (volts)	2.89	2.936	3.175
I_L (amps)	20.53	21.05	19.45
P_e (watts)	59.3	61.8	61.8
R_g (ohms)	0.140	0.139	0.165

NOTE: Conditions at last data point of Figure 6-1.

B. NON-DESTRUCTIVE EXAMINATION

The non-destructive testing of TEM-3 S/N-5 was completed as part of the examination of a module which has been through the entire fabrication process used on previous modules, i.e., like the TEM-1 modules on test. The measured room temperature resistances of this module are as follows: (1) circuit resistance 0.050 ohms and (2) resistance to clad 1.18 ohms.

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Figure 6-1. Efficiency Versus Time: TEM-1 S/N-1, -2 and -4

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The heater probe test, which gives a qualitative indication of the performance of incremental thermoelectric sections along the length of a module, showed that the positive end of the module was not performing as well as the negative end. However, the overall performance of the module during this test was better than that of previous modules. This suggests that a shorting problem existed between circuit and clad, which was noticeably worse near the positive end. Also, it implies that the overall effect of the shorting on the module was less than on the previous modules (i.e., the resistance from clad to circuit for this module should be more than for previous modules). The measured value of the resistance between circuit and clad was 1.18 ohms; this is high in comparison to the previous modules having this problem.

The hot to cold water test, which gives open circuit voltage versus a varying $\bar{T}_H$ from 10° to 100°C with constant $\bar{T}_C$ (10°C), also indicated a slightly higher V_{oc} for the same test than for previous modules.

When a current was passed through the thermoelectric circuit and the potential profile along the clads was measured, the module experienced shorts to the inner clad only. An indication of a low resistance shorting was found in the length of the module extending from the positive collector ring and through the six couples adjacent to it. Indications of a higher resistance short from inner clad to circuit were also found between the twelfth and sixteenth couples, counting from the positive end. Sectioning of the module later proved that these indications were accurate.

The above tests were also performed on TEM-3 S/N-6 as an example of a module which has been through the processing cycle including swaging, but excluding gas pressure sintering. The measured resistance of the thermoelectric circuit was 1.43 ohms and the circuit-to-clad resistance was 4×10^{10} ohms, both at room temperature. Both the heater probe test and the hot to cold water test resulted in open circuit voltage values higher than any other TEM module. The voltage profile test was not performed on this module since it had a high resistance from circuit to clad.

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C. HEATER TESTING

Two heater performance tests have been operating at power input and $\bar{T}_H$ levels above normal TEM test conditions. The HEP-11 heater has been on test for a total of 3216 hours at $1320 \pm 20^\circ\text{F}$, and the finned heater for a total of 3312 hours at the same temperature and under the same conditions. The heaters used in these tests are the bayonet-type cartridge heater described on WANL Drawing 978D841. The first test is a heater installed in a HEP module (S/N-11) in the same manner as is used on the TEM modules. The second test consists of a heater installed in a finned body designed to simulate the TEM module heat transfer condition. Both tests are described in detail in the Phase I Final Report (WANL-PR(CC)-006). The test conditions maintained are well above the present module test conditions of 1000° to 1100°F , and serve as a measure of performance capability for this type of heater.

D. DYNAMIC LOOP TESTS

A test section for operation in one of the NaK loop test legs is being fabricated in preparation for insertion of modules later in Phase II-A. The test section, shown in Figure 6-2 is of multipurpose design. Thermocouple penetration design, materials, and thermal characteristics will be evaluated during operation of the test section in the NaK loop.

The unique temperature measurement requirements for thermoelectric module operation in a liquid metal loop necessitate introduction of clad thermocouples directly into the flowing liquid metal. Two techniques are proposed to accomplish this objective. First, a conventional "Swagelok" thermocouple seal will be used on the end of a length of small diameter tubing. The extension tubing will permit the thermocouple seal to be located away from the hot flowing liquid metal and thus permit the seal to be at a lower temperature. A second type of penetration design is also shown in Figure 6-2. A flange is electron-beam-welded directly to the thermocouple sheath. This assembly is then welded to the adapter section.

Included in the test section are lengths of Inconel 718 and X750. The relative corrosion resistance of these materials to flowing high temperature NaK will be determined although

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Figure 6-2. NaK Loop Test Section

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no problems are anticipated. The Inconel 718 was solution treated and aged prior to welding to the Type 316SS adapters. The Inconel X750 was heat treated after welding, per the standard fabricating procedure for manufacturing TEM inner clads. One end of the Inconel 718 section was electron-beam-welded and the other end was TIG welded to the Type 316SS adapters. The effect of exposure to flowing high temperature NaK on these welds will also be investigated.

Thermocouples will be arranged along the outside diameter of the test section and a correlation of external wall temperature with internal NaK temperature will be attempted. The test section is currently being assembled and will be installed in the loop. The loop will be operated for about 1000 hours at conditions approximating those expected for module operation.

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VII. MATERIALS DEVELOPMENT

A. THERMOELECTRIC MODULE

The module development program is supported by a materials program with the objective of establishing the following:

- (1) A basis for understanding degradation phenomena in thermoelectric performance
- (2) The physical and mechanical properties of module materials as required for analytical and design purposes
- (3) Criteria for fabrication process control
- (4) Materials which may be introduced to module application for extended, improved performance at optimized conditions.

In this respect, therefore, the most significant need for experimental effort is in the thermoelectric materials being used in the tubular modules. Hence, the initial Phase II-A effort has placed the primary emphasis on materials studies related to module problems associated with PbTe. One of the most elementary problems has been the characterization of the thermoelectric material being used in the modules so that long-term effects could be adequately evaluated, and phenomena related to degradation of performance could be separated on the basis of mechanical design, or metallurgical, chemical, thermoelectric or physical attributes. The following sections present the work accomplished, initiated or planned in this respect during the first quarter of Phase II-A.

Lead Telluride Materials

Metallography

In order to develop the proper basis for understanding the morphology (or phase-equilibrium changes) of lead telluride materials during the complex module fabrication process, it is necessary to place the metallography of the thermoelectric material on a sound, reliable basis,

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because reliable interpretation of metallurgical phases will be required to explain and control property changes. The 3M PbTe materials are being thoroughly examined in order to characterize the microstructure after various processing treatments. The problem associated with excess lead in the n-type material has been demonstrated many times in electrical shorts through the boron nitride. This problem merely emphasizes the need for accenting studies on the metallographic phase relationships and the necessity for correlation with concurrent X-ray analysis of structure and thermoelectric properties.

Samples of 3N, n-type PbTe were cold pressed, heat treated in H_2 at $550^\circ C$ for two hours and further treated as shown in Table 7-1 to simulate and bracket, initially, the processing exposure of the material. This material was examined metallographically using standard cold mounting techniques and vacuum cathodic etching. Precautions were taken during all sectioning operations to ensure that the PbTe was maintained at room temperature. Vickers hardness readings were taken on the as-polished specimens in an attempt to correlate the microstructure, hardness and thermal treatments.

TABLE 7-1
Oxygen Analysis of PbTe

Lot-Type Condition	Oxygen Content (ppm)				Number of Samples from Each Lot
	Lot 1051 3N	Lot 1050 2N	Lot 1052 2P	Lot 1053 3P	
As Received	36, 139	66, 65	119, 105	330, 365	2
Heat Treated	20*, 18*	21*, 38*	21*, 32*	259**, 269**	2

*Heat treating temperature = $550^\circ C$

**Heat treating temperature = $450^\circ C$

The photomicrographs shown in Figures 7-1 through 7-4 reveal the changes produced by temperature and pressure. H_2 treated material showed numerous large void areas and a randomly dispersed spherical type of precipitate as shown in Figure 7-1. The VHN of 31.2 showed a

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100X Magnification

100X

100X

100X

500X Magnification

500X

500X

500X

Figure 7-1. 3N PbTe Pellet 369P
Pressed and Hydrogen Tested at 550°C
for 2 Hours

Figure 7-2. 3N PbTe Pellet 369P
Pressed and Hydrogen Tested at 550°C
for 2 Hours, 650°C for 15 Minutes,
Water Quenched

Figure 7-3. 3N PbTe Pellet 369P
Pressed and Hydrogen Tested at 550°C
for 2 Hours, 750°C for 15 Minutes,
Water Quenched

Figure 7-4. 3N PbTe Pellet 369P
Pressed and Hydrogen Tested at 550°C
for 2 Hours, Gas Pressure Sintered at
650°C, 7500 psi

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relatively uniform value across the face of the sample. Examination of the indentation revealed little, if any, strengthening from the precipitate particles.

Figures 7-2 and 7-3 show the respective microstructures for samples heated for 15 minutes at 650 and 750°C, followed by a water quench. As the quenching temperature increases, the precipitate becomes more dense. In addition, at higher temperatures, there is a tendency for the precipitate to thicken into what appears as rods in the two-dimensional view of the microstructure. The hardness of the material changes with increasing temperature, decreasing from a VHN of 31 for the H₂ treated sample to 29 and 25 for the 650° and 750°C quenched samples, respectively. Whether the decrease is associated with the precipitate or is an "annealing" of the stresses produced by cold pressing has not been determined.

Figure 7-4 shows a sample that has been gas pressure sintered at 650°C and 7500 psi. The microstructure shows an almost completely dense condition with a considerable reduction in the density of the precipitate inside the grains. The gas pressure sintering has also increased the hardness to a VHN of 32, approximately that of the H₂ treated material.

From the preceding observations, it appears that the kinetics of the PbTe at this thermal treatment are extremely sluggish. Heating to 750°C in the absence of pressure failed to get the precipitate into a condition approaching a single-phase solid solution. However, gas pressure sintering for two hours altered the microstructure and much of the precipitate disappeared from the grains.

Selected pellets from various modules will be examined after testing to completely characterize both n- and p- material precipitate morphology. In addition, a concentrated effort will be made to enable a more complete understanding of the time-temperature-pressure relationships of the system phases.

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Oxygen Concentration

Degradation in the electrical performance of PbTe thermoelectric generators has been attributed to oxidation at the metal-semiconductor junction and/or internal oxidation of the dopant. The problem of accurate, reliable oxygen determinations was investigated during Phase I. A bromine trifluoride analysis produced an oxygen content an order of magnitude higher than that determined by neutron activation analysis. The discrepancy was resolved in favor of neutron activation on the basis of results obtained on comparable material by Battelle Memorial Institute, and by analyses performed on identical samples by the Westinghouse R & D Center and the AEC New Brunswick Laboratory.

In the present program, it is planned to

- (1) Measure the oxygen content of 3M incoming material
- (2) Determine the effectiveness of the storage system in preventing additional oxygen pickup, and
- (3) Establish the effectiveness of H₂ heat treating in reducing the oxygen content of the lead telluride.

The results shown on Table 7-1 were obtained on pellets pressed from as-received material as well as on pellets that were H₂ treated for two hours at the indicated temperature. These results indicate that H₂ heat treating at 550°C reduces the O₂ level in the PbTe samples to the 20 to 30 ppm range regardless of the original content. The Pb-Sn-Te alloy which was treated at a lower temperature* (450°C) has been reduced in oxygen content to a lesser extent. Whether the small reduction is due to the lower heat treating temperature or is inherent in the material cannot be answered at this time. It should be emphasized that these observations are not complete and require considerable supplementary experimentation for confirmation.

*Previous work under other programs disclosed that some 3P material was covered with a golden brown coating after H₂ treatment at high temperatures (550° to 600°C); hence, the first treatment was performed at a reduced temperature.

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Chemical Characterization

The composition of the thermoelectric materials purchased from the 3M Company is being investigated in order to characterize the material. The information obtained will be used in other investigations, e.g., penetration of BN by free lead extruded from the material during processing⁽¹⁾ and dopant diffusion in a temperature gradient. The preliminary results are summarized in the following paragraphs.

The materials designated TEGS-2N, -3N and -2P are suitably doped lead telluride. They are lead and tellurium rich for the n- and p-type materials, respectively. The p-type dopant has been identified as sodium and is present at a level of approximately 0.062 weight percent. The n-type dopant, as yet, has not been identified in the materials currently being used. Lead iodide has been used by 3M as the n-type dopant; however, no iodine has been detected either in a hollow cathode survey for halogens or by wet chemistry methods. Work in this area is being continued and previous results are being checked.

The feasibility of determining dopant levels in PbTe by activation analysis is currently under investigation. Various facilities are being contacted to determine the sample size, degree of accuracy, level of concentration and probable delivery schedules. The initial objective will be for bulk analysis, and this later will be extended to surface concentrations.

Chemical and X-ray analyses performed in investigating the short circuiting problems in thermoelectric modules disclosed that the probable cause is the n-type material.⁽²⁾ It appears that some of the excess lead, which comprises about 0.43 weight percent of the material, is released from the PbTe at high temperatures and pressures. PbTe containing one-half the usual excess lead has been received from the 3M Company and will be chemically analyzed and tested.

(1) WANL-TME-1342, Investigation of the Short Circuit Problem in TEM Series Tubular Thermoelectric Modules, Phase I Status, December 31, 1965.

(2) R. W. Fritts, et al., U. S. Patent 2,811,571, dated October 29, 1957.

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TEGS 3P is a Pb-Sn-Mn-Te alloy. The manganese content (approximately 3 mol percent) would appear to be too high for an impurity dopant, particularly since manganese is reported as an n-type dopant for lead telluride.⁽¹⁾ The alloy also contains about 0.08 weight percent sodium as the acceptor impurity. The 3P material is currently used by 3M in their thermoelectric programs, because of its stability at high temperatures. It does not appear that this stability is achieved by deleting sodium as a dopant as has been stated by other investigators.

Electrical and Thermal Properties

Apparatus is being built and tested to provide measurements of the electrical resistivity and thermal conductivity of individual samples after H₂ treating, gas pressure sintering and long-time "soaking" intervals (600°C) for determination of stability of properties. If large changes in the Seebeck coefficient or the electrical resistivity are determined, it will probably be necessary to examine the materials for carrier concentration changes, changes in the Pb/Te ratio or metallographic changes. The isothermal stability studies will be supplemented by investigation of thermoelectric material from modules removed from test in order to establish whether dopant diffusion has occurred in the thermal gradient of test operating conditions.

After test samples in the form of right circular cylinders (1/2 inch diameter by 5/8 inch long) are H₂ treated, measurements are made on five of them. The remainder are encapsulated in test capsules as shown schematically in Figure 7-5. After gas pressure sintering, another group of five is removed from a capsule and measured. The remaining capsules are heat treated at 600°C. The test schedule calls for capsules to be extracted and measurements made on the contents at 500, 1000, 3000, 5000, 7000 and 9000 hours. Seebeck measurements have been made on 3N material, Lot 1051. The Seebeck coefficient was calculated from the slope (over an interval of 25°C) of the Seebeck voltage versus temperature curve. The results are shown in Figure 7-6. Each data point from the program represents the average value of five samples. It is apparent from this curve that gas pressure sintering at 750°C and 10,000 psi produces negligible changes in the Seebeck coefficient of 3N material.

Figure 7-5. Schematic of Test Capsules Used in Gas Pressure Sintering of Lead Telluride

Figure 7-6. Seebeck Coefficient 3N Material, Lot 1051

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Dense Lead Telluride

Increased density (greater than 91 to 92%) of the PbTe washers is desirable so that final fabrication of the thermoelectric module will require less reduction to achieve full compaction. Work is planned to determine the maximum density that can be obtained while retaining the precise measurements of the washers after heat treating. Initial experiments have begun by pressing 95% dense p-type material in the form of one-piece washers instead of the normal two-piece (inner and outer) combination.

Methods other than cold pressing and H_2 heat treating are being considered for the fabrication of thermoelectric washers. Requests were sent to the 3M Company during Phase I and again this quarter for supplying low oxygen content, high density washers. A recent reply from the 3M Company indicates that they may be doing experimental work in order to determine what kind of a proposal could be made to this Laboratory for supplying the requested washers. They have indicated a proposal may be forthcoming early in the next quarter.

The most dense thermoelectric material, other than gas pressure sintered PbTe, is cast ultrasonically on a research basis by Tyco Associates. Their material is purported to be 100 percent dense, oxygen free (approximately 10 ppm O_2) and, in the case of n-type, free of excess lead. Attempts have been made through the AEC to obtain some of this material for evaluation. No definite commitments for provision of the material have been made as of this time.

Density samples of hot pressed material were obtained from the penetration experiments described in Section VI, and the results are shown in Table 7-2. The theoretical density of stoichiometric PbTe, calculated from X-ray measurements, is 8.251 gm/cc.

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TABLE 7-2
3N Material After Hot Pressing

<u>Sample</u>	<u>Pressure (psi)</u>	<u>Temperature (°C)</u>	<u>Density (gms/cc)</u>
12680	6,500	650	8.19
12678*	7,500	650	8.26
12679	10,000	650	8.24
12676	4,000	750	8.23
12677	6,500	750	8.23

*Sample size was small and could have resulted in measurement error.

The pressures and temperatures used in Table 7-2 are lower limit determinations of those necessary to achieve full compaction of the n-type material. These experiments show the pressure directly applied to the thermoelectric material. In a module or test capsule the pressure will be attenuated to some extent by the clads and/or other cylindrical shells.

Additional work of this type will be performed on p-type material which is more difficult to compact at room temperature.

Boron Nitride

Penetration by Lead Telluride Material

The non-destructive and destructive examination of tubular thermoelectric modules in the Phase I program established the fact that some had electrical short circuits through the inner boron nitride insulating sleeve to the inner clad. This phenomenon was observed as being caused principally by cracks filled with a Pb-rich material, but also in the form of BN porosity filled with the same material, although the latter manifestation was usually not electrically shorted. As this penetration has been observed to be much more pronounced in BN for n-type PbTe, experiments were designed to determine qualitatively the time-temperature-pressure parameters affecting this penetration. As shown schematically in Figure 7-7, 3N-PbTe was

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

cold pressed (approximately 97% dense) into 0.500 inch diameter x 0.500 inch long cylinders, hydrogen treated for two hours at 575°C, and fitted into mating BN 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 7-3, 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 BN/PbTe, BN/graphite interfaces as well as the cross section of the PbTe and BN. Detailed analysis remains to be completed.

TABLE 7-3
Temperature Pressure Data for PbTe/BN Interpenetration Study

Sample No.	Pellet No.	Temperature (°C)	Pressure (psi)	Time (min)
1	136P	650	6,500	30
2	137P	750	4,000	30
3	138P	750	6,500	30
4	139P	650	7,500	30
5	140P	650	10,000	30

In addition to these experiments, which simulated the actual fabrication exposure conditions, two boron nitride insulating sleeves (one standard, hot pressed boron nitride; the other with a Ca additive) were examined metallographically to determine the degree of penetration encountered during gas pressure sintering. These boron nitride sleeves were in contact with 3N-PbTe pellets separated by mica discs. The 3N-PbTe pellets were 1/2-inch diameter x 5/8-inch long right cylinders that had been cold pressed and H₂ treated for two hours at 550°C. The PbTe, mica and boron nitride were encapsulated in AISI 304 of 0.050-inch wall

thickness and gas pressure sintered for two hours at 650°C and 7500 psi during a special auto-clave cycle which was run to help establish the lower temperature/pressure relationships for module fabrication. Undoped boron nitride showed localized penetration to depths of 0.0073 inch. Examination of the calcium containing boron nitride showed the penetration to be much more uniform along the inner wall with a maximum penetration depth of 0.0012 inch. This observed difference in behavior of the two types of boron nitride is being followed up by continuing experiments.

Thermal Expansion

The lack of reliable thermal expansion data on boron nitride has restricted the theoretical stress analysis program. In order to obtain more complete data, a 4 inch diameter x 8 inch long cylinder of boron nitride (the standard hot pressed source of insulating sleeve material) was sectioned as shown in Figure 7-8 to provide samples from the top, center and bottom of the "as pressed" cylinder. Samples were machined to 0.750 inch diameter x 2 inch and 3 inch long cylinders, with the long axis parallel and normal, respectively, to the axis of pressing. Some of each configuration were encapsulated in thin walled AISI 316 stainless steel, heated to 350°C under vacuum and then sealed by electron beam welding. The capsules were gas pressure sintered (GPS) at 750°C and 10,000 psi. After GPS the stainless steel was removed and the boron nitride machined to 3/8 inch diameter x 1.750 x 2.500 inch long for the 2 and 3 inch long samples, respectively. In addition, samples of the "as-pressed" material were also machined to similar dimensions. These samples are currently being prepared for thermal expansion measurements.

Fracture of Boron Nitride

To further define the mechanical properties of boron nitride, two 5/8 inch cubes of "as-pressed" material were machined. The first was compressed at R. T.; the second will be tested at 650°C. Strain gages were attached to yield the strains shown in Figure 7-9. The original hot pressing direction of the ingot was parallel to the "Z" direction. Compressive stress was therefore applied transverse to the pressing stress. The sample was stressed and relaxed at

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Figure 7-8. Location of Boron Nitride Cylinders for Thermal Expansion Measurements

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9,000, 18,000 (3 times) and to failure at 38,300 psi. Selected properties are tabulated in Table 7-4. The final fracture (Figure 7-10) occurred across the X and Y planes or in the direction of the pressing longitudinal axis. These data are currently being analyzed and will be compared with the 650°C results.

TABLE 7-4
Compressive Data Obtained at R. T. on 5/8 Inch
Cube of Boron Nitride

	<u>Plane Normal to Hot Pressing Direction</u>	<u>X Plane*</u>
Failure strain	10.93 mils/in.	11.54 mils/in.
Poisson's ratio	0.06 ± 0.02 to 20,000 psi	0.16 ± 0.01 to 20,000 psi
Tangent Young's modulus measured over stress range, 0 to 20,000 psi	7.7 to 3.3×10^6 psi	5.8 to 3.9×10^6 psi
Total lateral strain	2.42 mils/in. at 38,300 psi	2.90 mils/in. at 35,000 psi

*See Figure 7-9 for orientation of planes.

Mica

A previous literature summary of thermoelectric module materials properties⁽³⁾ disclosed the lack of thermal conductivity data on phlogopite mica. This absence of information presents major difficulties in the theoretical thermal analysis of module performance. Three approaches are being taken to obtain the necessary thermal conductivity data as a function of temperature. In the first two approaches, mica strip (0.5 inch wide x 2 inch long x 0.005 inch thick) and mica washers (1.333 inch OD x 0.7930 inch ID x 0.005 inch thick) were cut or pressed to size and heat treated for 1/2 hour at 800°C in $< 5 \times 10^{-5}$ torr pressure. These two shapes of mica

3. WANL-TME-1306, Literature Summary of Thermoelectric and Thermoelectric Module Material Properties, September 21, 1965.

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Figure 7-9. Strain Gage Orientation on Boron Nitride at Room Temperature

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Figure 7-10. Fractured Boron Nitride at Room Temperature

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will be used in conventional measuring techniques to determine the thermal conductivity.

The third approach utilizes a somewhat less orthodox method and simulates the actual laminar orientation of the mica in a tubular module application. Mica washers will be encapsulated in a hollow tubular stainless steel container and swaged to eliminate any processing voids. The entire capsule will then be heated by a contained heater to various temperatures to raise the temperature of the ID of the mica approximately 10°C higher than that at the OD. Accurate measurements of the power supplied to the heater for various ambient furnace temperatures will be used to calculate the thermal conductivity. The components have been fabricated, the thermocouples attached and the composite capsule assembled; thermal measurements are the next experimental phase.

Potential Insulation Materials

The tubular thermoelectric module utilizes several electrical insulation materials and contains numerous potential areas for the utilization of improved or unique electrical and thermal insulation. It is generally desired that the electrical insulation be a good thermal conductor; therefore, materials such as BN, BeO, SiN and, in particular, unique methods of applying thin, dense layers will always be of interest. The first application of an "applied" layer of such a material used Al_2O_3 as a possible approach to preventing thermoelectric circuit shorting to the inner clad. This material was incorporated as an experiment by plasma spraying a deposit of Al_2O_3 on the inner cladding of TEM-5 S/N-1. For a quick evaluation of the process, samples were obtained and a metallographic examination was used to select the method producing the best adherence qualities. A 2-inch section on the inner clad of TEM-5 S/N-1 was plasma sprayed with a coating consisting of a nickel-aluminide base of 1 to 3 mils and an Al_2O_3 thickness of 4 to 6 mils over the base.

In addition to the microscopic examination, the following checks were performed:

- (1) Specimens were heated to 700° in 100°C increments, removed from the hot

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furnace and allowed to cool in still air. No cracking or chipping of the coating was observed.

- (2) Room temperature electrical resistance measurements ranged from 5×10^9 to 2×10^{12} ohms when 500 V d-c were applied across the coating.
- (3) Room temperature electrical resistance did not change significantly after heating specimens in air to 250°, 350° or 450°C.
- (4) A calculated density measurement based on the composite of nickel-aluminide and Al_2O_3 coating averaged 3.92 gm/cc. The coating was satisfactory for an initial experimental module not going into a performance test, but considerable work will have to be done to use it successfully in a regular test module. In addition, Al_2O_3 was used only because of experience and convenience in coating with that material. Other materials with better thermal conductivity will be considered on a continuing basis, at a low level of effort, in the event that a need arises in the future.

Structural Material Tests

Mechanical testing of the clad materials, AISI 316 and Inconel X750, was carried out to provide data for stress analysis. The effort provided information on both materials as to their load carrying abilities as a function of temperature. The data were used to construct flow stress curves so that changes in materials properties at constant strain could be calculated as a function of temperature.

AISI 316 stainless steel and Inconel X750 were machined into 2.00 inch long x 0.357 inch diameter gage, threaded tensile bars. The tensile bars were cleaned and sealed in quartz under a partial pressure of argon. The AISI 316 was annealed for 1 hour at 1025°C to produce a fully recrystallized structure. The Inconel X750 was solution-annealed for 1 hour at 1050°C, and double-aged at 840° and 705°C to produce an optimized creep-rupture structure.

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The AISI 316 stainless steel was then tested at R. T., 200° and 750°C, through cyclic load-unload tensile tests to 0.2, 0.5, 1.0, 3.0 and 5.0% strain. The Inconel X750 was similarly tested at 538° and 649°C to 0.2, 0.5 and 1.0% strain.

All tests were conducted in air at a strain rate of 0.015 in./in./min. Typical mechanical property data are listed in Table 7-5.

TABLE 7-5
Stress Data for AISI 316 Stainless Steel and Inconel X750

<u>Specimen No.</u>	<u>Temperature (°C)</u>	<u>Stress at P.L. (psi)</u>	<u>Stress at 0.2 Y.P. (psi)</u>	<u>Uniform Strain (%)</u>	<u>Elongation (% in 2 in.)</u>
AISI 316-1	R. T.	29,300	32,200	9.23	9
AISI 316-2	200	20,000	22,100	4.90	5
AISI 316-3	750	13,600	15,500	4.25	4
Inconel - 1	538	96,000	103,000	1.14	1
Inconel - 2	649	71,000	87,000	1.12	1

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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 demanded by module configuration and construction. Where possible, existing tooling has been modified to meet the requirements of development hardware without destroying the capability of usefulness on existing designs.

A preliminary survey of power requirements for an emergency power supply for loop and test stands has been completed.

B. EQUIPMENT

The 60,000-psi isostatic compacting system with a chamber 4 inches in diameter and 20 inches long was scheduled for delivery in March. However, delivery has been delayed until May because of late delivery of a forging.

A liquid metal transfer chamber has been obtained and is undergoing modification. The chamber will be used for NaK filling and sealing of modules. In addition to the liquid metal chamber, a Blickman atmosphere chamber was obtained and is presently being integrated into the system in the module assembly room. The Blickman chamber contains a transfer lock which will be tied to the existing chambers in the near future.

The Whitaker vacuum storage system was delivered during the first quarter of Phase II-A. The system was installed in the module assembly room and put into operation in early February.

Additional activity for this reporting period in the area of Facilities and Equipment is as follows:

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- (1) Special carbide dies design for compacting powder on the 100 ton compaction press were delivered in early March.
- (2) Quotations have been obtained and reviewed for a gas pressure sintering auto-clave, and site locations are being investigated.
- (3) Equipment specifications for the thermoelectric module test stand data acquisition system were prepared, and quotations are expected in early April.
- (4) An additional H₂ purge and vacuum degassing system was installed, allowing experimental samples to be processed in parallel with modules, using the original H₂ purge and vacuum degassing system.

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APPENDIX A

PERT DATA

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TABLE A-1

Summary of PERT Network Events Completed
During the Report Period

<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
232 0014	Start Material Evaluation Program	1-15-66	1-15-66
212 0002	Complete Phase II-A Project Plan	1-31-66	1-28-66
232 0080	Complete Manufacturing TEM-3	2-7-66	2-16-66
234 0104	Complete Spec Test Equipment Design	2-15-66	2-21-66
232 0082	Complete Assembly TEM-3	2-15-66	2-23-66
232 0084	Complete Processing TEM-3	2-22-66	3-1-66
232 0088	Complete Preliminary Destruct Examination TEM-3	3-1-66	3-28-66
231 0064	Complete Design TEM-5	3-3-66	2-14-66
231 0066	Complete Design TEM-6	3-9-66	3-1-66
233 1000	Complete Manufacturing TEM-5	3-11-66	3-11-66
231 0034	Complete Design TEM-4A	3-16-66	3-23-66
233 2000	Complete Manufacturing TEM-6	3-17-66	3-19-66
233 1110	Complete Assembly TEM-5	3-18-66	3-18-66
231 0068	Complete Design TEM-7	3-23-66	3-18-66
231 0070	Complete Design TEM-8	3-29-66	3-28-66
234 0094	Complete Preliminary Performance Test Evaluation TEM-1	3-30-66	3-30-66

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TABLE A-2
Phase II-A Compact T/E 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	Phase II-A Project Plan Submitted to AEC	1-31-66	1-31-66
232 0088	Complete Destruct Examination TEM-3	3-1-66	3-28-66
234 3128	Start Performance Tests - TEM-6	4-19-66	
213 0004	Submit First Quarterly Progress Report	4-15-66	
234 4128	Start Perform Tests - TEM-7	4-29-66	
232 0028	Complete Evaluation of Cold Isostatic Pressing	6-1-66	
232 0020	Complete Mid-Term Material, Fabrication and Process Evaluation	6-30-66	
234 0110	Complete C/O of Special Test Equipment	7-14-66	
211 0006	Submit Mid-term Progress Report (2nd Qrty.)	7-15-66	
211 0008	Phase II-A Mid-Term Briefing	7-21-66	
232 0016	Complete PbTe Densification Report	8-15-66	
234 0094	Complete Initial Performance Test Evaluation	9-15-66	
213 0010	Submit 3rd Quarter 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, Evaluations	12-15-66	
213 0000	Submit Phase II-A Final Report (Draft)	12-31-66	

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Figure A-1. PERT Network

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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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Figure C-2. Assembly, Thermoelectric Module, TEM-5

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Technical drawing of a nuclear fuel element assembly, showing various views and details. The drawing includes a top view (SECTION C-C), a side view (SECTION A-A), and a detail view (DETAIL B). The assembly consists of a central fuel element (GROUP 1) surrounded by a coupling assembly (GROUP 3) and a negative end assembly (GROUP 4). The drawing is labeled with various dimensions and part numbers, including P294666-1, P294666-2, and P294666-6. The drawing is titled "FINAL MACHINING AFTER PROCESSING".

Figure C-3. Assembly, Thermoelectric Module, TEM-6

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Figure C-5. Assembly, Thermoelectric Module, TEM-8

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