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REPORT

Final Report

N123(60530)10049A

30 November 1960

TEST EQUIPMENT FOR AND EVALUATION
OF ELECTROMAGNETIC RADIATION

Volume I

Application of Evaporated Thermocouples to Detection of
RF Power in Bridge Wires of Electro-Explosive Devices

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TEST EQUIPMENT FOR AND EVALUATION
OF ELECTROMAGNETIC RADIATION

Final Report
Covering Period From
15 March 1957 through 30 November 1960

Contract No. N123(60530)10049A

Volume I

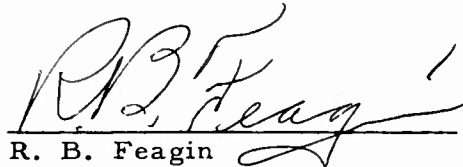
Application of Evaporated Thermocouples to Detection of
RF Power in Bridge Wires of Electro-Explosive Devices

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The final report of U. S. Naval Weapons Laboratory, Contract Number N123(60530)10049A entitled Test Equipment for and Evaluation of Electromagnetic Radiation consists of four volumes.

<u>Volume</u>	<u>Title</u>	<u>Classification</u>
I	Application of Evaporated Thermocouples to Detection of rf Power in Bridge Wires of Electro-Explosive Devices	Unclassified
II	Application of Thermistors to Detection of rf Power in Bridge Wires of Electro-Explosive Devices	Unclassified
III	Methods of Detecting rf Power in Bridge Wires of Electro-Explosive Devices and Instrumentation Problems	Unclassified
IV	Investigations of rf Power Transfer to Bridge Wires of Electro-Explosive Devices	Confidential

ABSTRACT - VOLUME I

A review of the development and a discussion of the present status of vacuum deposited thermocouples are given.

Measurements of thermoelectric powers and resistivities of evaporated films are given as a function of age for various thicknesses. Also presented are the effects of heat on film resistances and thermoelectric powers and studies of "alumel" films. A multiple beam interferometer capable of measuring film thickness to $\pm 20 \text{ \AA}$ has been constructed and a description is included.

Improvements in vacuum deposition methods and thermocouple fabrication are described.

A reference heat source capable of providing steady heat output as well as chopped radiation up to 100 cps is described. Results of tests attempting to determine aging characteristics, effect of humidity and effect of thermocouple coating materials for various types of lead connections and combinations of materials are presented. In addition, tests of the effects of organic solvent vapors are reported. Thin film substrate

thermocouples having time constants of about 10 milliseconds which have been developed are described. Results of measurements of Mk 1 squib bridge wire temperature rise above ambient as a function of current are given with corresponding vacuum deposited thermocouple sensitivities.

ABSTRACT - VOLUME II

Thermistor parameters and uses are described briefly. Theoretical analyses of thermistor bridge circuits and the resulting design equations are presented. Experimental drift data of thermistor bridges have been included and reasons for drift are discussed. Thermal time constant measurements of thermistors are given, and two methods of reducing the response time of a thermistor bridge system are presented.

Techniques for mounting thermistors to detect heat from various bridge wire configurations have been developed, and operating characteristics of thermistor instrumented EED's are given. Both dc and ac thermistor instrumentation units have been developed to indicate temperature rises above ambient in bridge wires of electro-explosive devices.

ABSTRACT - VOLUME III

Problems concerning the application of photoconductors to measuring bridge wire temperature in electro-explosive devices have been investigated, and some data and conclusions are presented. Miscellaneous methods (not including thermocouples or thermistors) for instrumenting electro-explosive devices are discussed.

Results of comparisons of instrumented electro-explosive devices at the 50% firing power as determined by Bruceton tests for dc, 8Mc and 1.2 kMc are given.

Allied problems pertaining to instrumentation required by various temperature sensors and miscellaneous problems pertaining to electro-explosive devices are included.

ABSTRACT - VOLUME IV

A description of the Denver Research Institute of the University of Denver antenna measurements range is given, and results of its use to measure radiation patterns of the FFAR 2.75 Rocket are presented.

Documentations of preparations for specific shipboard and field tests to measure hazards to ordnance by electromagnetic radiation are presented. Procedures and results are included for some tests.

TABLE OF CONTENTS

	<u>PAGE</u>
ABSTRACT OF VOLUME I	ii
ABSTRACT OF VOLUME II	iv
ABSTRACT OF VOLUME III	v
ABSTRACT OF VOLUME IV	vi
LIST OF FIGURES	ix
LIST OF TABLES	xi
I. INTRODUCTION	1
II. THE PRESENT STATUS OF EVAPORATED THERMO- COUPLES	5
III. FUNDAMENTAL STUDIES OF VACUUM DEPOSITED FILMS	11
A. Thin Film Resistivity	11
B. Thermoelectric Power of Evaporated Films	20
C. The Effect of Heat on Resistivity and TEP	28
D. Optical Measurement of Film Thickness	28
IV. EVAPORATION AND FABRICATION	34
A. Thermocouple Materials	34
B. Terminal Connections	36
C. Movable Mask	36
D. Casting Thermocouple Base Plugs	40
V. TESTING AND EVALUATION	42
A. Reference Heat Source	42
B. Aging Test	49
C. Humidity Test	53
D. Effects of Organic Solvents	57
E. Effects of Coating Materials and Thermocouple Constituents	63
F. Thermocouple Sensitivity Measurements Using an Instrumented Mk 1 Squib	66

TABLE OF CONTENTS (Cont.)

	<u>PAGE</u>
G. Time Constant Measurements.	72
H. Output Loading	81
VI. SUMMARY	85
VII. BIBLIOGRAPHY	91
DISTRIBUTION LIST.	92

LIST OF FIGURES

<u>NUMBER</u>		<u>PAGE</u>
II-1	Mk 1 Type Vacuum Deposited Thermocouple, Bismuth-Antimony on Mylar Substrate	9
III A-1	Resistance vs. Thickness of Antimony Films (Group I)	14
III A-2	Resistance vs. Thickness of Bismuth Films	15
III A-3	Resistance vs. Thickness of Tellurium Films	16
III A-4	Resistance vs. Thickness of Antimony Films (Group II)	17
III A-5	Resistance vs. Thickness for Some Thin "Alumel" Films	18
III B-1	Determination of Thermoelectric Power.	22
III B-2	Thermoelectric Powers Relative to Copper vs. Thickness for Some Thin Films	24
III B-3	Thermoelectric Power of "Alumel" Films.	26
III C-1	Dependence of Resistance on Temperature for Some Antimony Films	29
III D-1	Plate Arrangement for Producing Interference Fringe Patterns	31
III D-2	Fringe Pattern Showing the Effect Produced by the Step of the Thin Film	31
III D-3	Optical System for Measuring Thin Film Thick- nesses	32
IV C-1	Insert Plate Showing Movable Mask	38
IV C-2	Vacuum Chamber with Mask in Place	39
V A-1	Reference Heat Source Block Diagram	43
V A-2	Time Constant Measuring System for IR Sensors	44
V A-3	Reference Heat Source	45
V A-4	Reference Heat Source and Associated Equipment	46
V B-1	Resistance Change of Thermocouples in 100 Days	52

LIST OF FIGURES (Cont.)

<u>NUMBER</u>		<u>PAGE</u>
V D-1	Output Change of Uncoated Thermocouples Caused by Exposure to Organic Chemical Vapors	59
V D-2	Resistance Change of Uncoated Thermocouples Caused by Exposure to Organic Chemical Vapors	60
V D-3	Resistance Change of SiO Coated Thermocouples Caused by Exposure to Organic Chemical Vapors	61
V D-4	Resistance Change of Lacquer Coated Thermo- couples Caused by Exposure to Organic Chemical Vapors	62
V E-1	Effects of Lacquer Coating on Thermocouple Outputs	65
V F-1	Experimental Circuit for Determining Resistance Variation of a Mk 1 Squib Bridge Wire with Bridge Wire Current	69
V F-2	Bare Bridge Wire Temperature of a Mk 1 Squib as a Function of Current	73
V F-3	Inert Loaded Mk 1 Squib Bridge Wire Temperature as a Function of Current	74
V F-4	Thermocouple Detector Response as a Function of Mk 1 Squib Bridge Wire Temperature	75
V F-5	Thermistor Detector Response as a Function of Mk 1 Squib Bridge Wire Temperature	76
V G-1	Thermocouple Response to a Step Function of Radiation	77
V G-2	Thin Film Substrate Thermocouple Response to a Step Function of Radiation	80
V H-1	Bi-Sb Thermocouple Loading Characteristic	82
V H-2	Dependence of Thermocouple Output on Thermo- couple Resistance	83

LIST OF TABLES

<u>NUMBER</u>		<u>PAGE</u>
III A-1	Electrical Resistivity of Some Common Thermocouple Materials	12
III A-2	Variation of Thin Film Resistance with Four Months' Aging	19
III B-1	TEP of Some Common Materials at 0° C Relative to Lead	21
III B-2	Thermal Coupling of the Mk 1 Squib Thermocouple to the Bridge Wire	27
IV A-1	Characteristics of Various Thermocouple Combinations	35
IV B-1	Thermocouple Characteristics for Various Types of Connection to the Terminals	36
V A-1	Rise Time of Radiation Beam	48
V B-1	Aging Test on Bi-Sb Mk 1 Squib Thermocouples	51
V C-1	Humidity Test on Bi-Sb Mk 1 Squib Thermocouples	55
V C-2	Comparison of Average Changes in Three Types of Thermocouples During the Humidity Test	56
V E-1	Output Voltages of Mk 1 Squib Thermocouples with Different Coating Materials	64
V E-2	Comparison of Thermocouple Time Constants	64
V E-3	Characteristics of Three Mk 1 Squib Thermocouple Combinations	67
V F-1	Resistance and Temperature Variations of a Mk 1 Squib Bare Bridge Wire with Current	70
V F-2	Resistance and Temperature Variations of an Inert Loaded Mk 1 Squib Bridge Wire with Current	71
V G-1	Comparison of Mk 1 Type Thermocouples Having Different Substrates	80
VI-1	Summary of Sensor Characteristics	86

I. INTRODUCTION

This report describes the work of Denver Research Institute of the University of Denver on the HERO program for the period 15 March 1957 through 30 November 1960.

Denver Research Institute of the University of Denver entered into contract with NOTS/ China Lake in March 1957 for the design of test equipment to be used in evaluating electromagnetic radiation hazards to ordnance items. From this study the need for a detector to simulate electro-explosive devices (EED's) became evident. The first detector used was a thermocouple attached to a one ohm bridge wire by a ceramic bead for simulation of the Mk 1 squib. The detector was enclosed in a glass envelope and evacuated. However, the large time constant due to the vacuum and long leads made this detection method undesirable. In an attempt to reduce the time constant, the vacuum thermocouple was filled with hydrogen to enhance the exchange of thermal energy between the bridge wire and thermocouple. This hydrogen filled detector had a shorter time constant but also less sensitivity than the vacuum detector. These methods of detection did not reduce the time constant to the desired value and other methods of detection had to be found. A serious objection arose to the proposal of removing the ceramic bead and connecting directly to the bridge wire, as little was known about the influence that this operation might have on the rf characteristics of the device. Two very stringent requirements thus dominated the development of new detectors, namely, the necessity of a short time constant, (i. e., one comparable to that of the EED bridge wire), and the isolation of the detector from the bridge wire.

During 1958, three major methods of detection were investigated. Two of these were developed sufficiently to be used in the instrumentation of weapons for field tests. The method which has met with the greatest acceptance and which is still in wide use is that of the vacuum-deposited radiation thermocouple. This technique utilizes thin films forming thermoelectric junctions which are of low mass but wide enough to absorb a relatively large portion of heat from the bridge wire. These thermocouples were used for two shipboard tests in 1958, four in 1959, and several in 1960.

The second detector which has been relatively successful is the thermistor, a small semiconducting solid with a very high negative temperature coefficient of resistance. The first application of thermistors was in the form of printed circuits supplied by Barnes Engineering Co.

These proved to be inadequate for this application, and investigations were performed at Denver Research Institute of the University of Denver to develop thermistor bridges with the desired properties of high sensitivity and low drift. A field test at the Marine Air Station at Cherry Point, North Carolina, April 1959, proved this method of detection to be satisfactory. The third method, and one which has received less attention is the quantum detector or photoconductor.

Approximately four man-years were expended at DRI on the HERO project while under the direction of NOTS/China Lake. In November, 1958, official administration of the DRI contract was transferred to the U. S. Naval Weapons Laboratory. Since that time, but concurrent with the necessary field preparation, the effort has been directed at three major goals:

- (1) To improve the existing thermocouple and thermistor detectors. Some of the vacuum deposited thermocouples had been found to have sensitivities which vary radically with age. There had been little success during fabrication of the thermocouple in determining the controlling reliability factors, but recently some important advancements have been made toward obtaining uniformity and reliability (Volume I of this report.) Improvements of the thermistor detectors have been chiefly in the ability to control noise and drift (Volume II of this report.)
- (2) To seek other methods for detection which will provide the requirement of higher sensitivity and/or faster response. One example is photoconductor instrumentation which is desirable because of its fast response. The problem here is complicated by miniaturization. Radiation choppers are essential to reduce drift and noise, and the cost of developing suitable choppers seems to be excessive. The photoconductor and other detectors such as the electron emission detector, Hall effect probe and toroid detector are described in Volume III of this report.
- (3) To determine whether detectors and instrumentation are susceptible to rf pick up from bridge wires. It is necessary to know whether instrumentation perturbs or is perturbed by the rf excitation of electro-explosive devices and what degree of perturbation exists as a function of frequency. This requires a system for measuring and controlling power into the bridge wire at all frequencies so that dc and rf detector outputs can be compared at some level of excitation (usually the 50% fire level.) Discussion appears in Volume III of this report.

At the beginning of 1960, it was felt that investigations concerning the application of thermistors to instrumenting electro-explosive devices had reached a logical completion considering that the feasibility and practicability of thermistor instrumentation has been demonstrated. The large repertoire of data and knowledge about thermistors which has been accumulated was organized and recorded in a special report. This report has been brought up-to-date and is the substance of Volume II of this report.

Also at the start of 1960 it became apparent that large quantities of instrumented electro-explosive devices were necessary for ordnance testing. The acute demand for hundreds of thermocouples prevented channeling of effort aimed at achieving a basic understanding of the operation and possible means of failure of evaporated thermocouples. Very little progress in perfecting evaporated thermocouples could be expected with these circumstances. For that reason, the U. S. Naval Weapons Laboratory undertook to supply the large number of thermocouples needed for ordnance testing to allow Denver Research Institute of the University of Denver to concentrate on research and development leading toward their perfection.

Evaporated thermocouple development has proceeded in three phases. First, fundamental investigations were directed toward the goal of determining and understanding the basic properties of evaporated films. Second, improvement of the technology of evaporation and fabrication has resulted, including a major achievement of improving thermocouple reliability. Third, testing and evaluation of thermocouples has been accomplished to establish relationships between thermocouple properties and parameters in evaporation and fabrication.

Past use of thermocouple instrumented electro-explosive devices indicated the need for discovering reasons for failure and variation in calibration of some devices. Many tests have been made in an attempt to isolate the major causes of failure and calibration variation. The use of inert squibs as a reference heat source for thermocouple comparison and testing was impractical because of the random spread in bridge wire characteristics due to manufacture and the possible change in characteristics due to repeated heating. In addition, use of small bridge wires as heat sources calls for close thermocouple spacing, thereby making thermocouple placement quite critical. For these reasons it was necessary to use a reference heat source. Use of a reference heat source not only eliminated confusion in discovering how fabrication procedures affect

thermocouple characteristics but also facilitated thermocouple testing. The reference heat source developed for this purpose was made sufficiently versatile to be useful for photoconductor measurements also.

Tests have been designed to determine how various types of thermocouples change in output and resistance as a function of age, humidity and exposure to organic solvents. The results of these tests were used for changing methods of fabrication to obtain optimum characteristics. Time constant measurements have also been made and effort to minimize time constant has been fruitful. Recently thermocouples having thermal time constants equal to those of bridge wires were developed.

II. THE PRESENT STATUS OF EVAPORATED THERMOCOUPLES

The first models of thermocouple detectors were made by machining a plug of plexiglass and machining small terminal connectors or pins which were inserted in holes in the machined plugs. These pins formed terminals to which the vacuum deposited films were attached. Mylar film was stretched over a void machined on the end of the plug and this film served as a base for deposition of the films. This basic idea was quite sound but the construction details were found to be impractical. For example, it was found that the machining procedure took too long and was too costly to produce the number of detectors that would be required on the program. The terminal pins tended to loosen in the plexiglass plug which resulted in open circuit connections. Also the deposited films did not bond adequately to the terminal pins and high contact resistance or open circuits developed.

Effort was placed on the redesign of the detectors which resulted in the presently used technique of casting the plugs of polyester resin in metal molds. The wire leads and terminals are cast in place and the only machining required normally is to face the end of the plugs before cementing the Mylar film in place. Use of silver conductive ink to make contact with the ends of the lead wires and to act as a broad base for a more positive contact to the deposited films was introduced. The above changes resulted in a practical, easily produced plug on which the vacuum deposited thermocouple elements could be placed. Molds have been made to date to accommodate many different electro-explosive devices.

The first deposited films for thermocouple junctions were bismuth and tellurium. When using tellurium it was very difficult to control the resistance of the deposit and the tellurium film exhibited some very radical changes in resistance due to aging for several hours, and this aging continued for days with no indication that the resistance would ever stabilize. Since tellurium is a semiconductor and impurity doping is important, considerable technology and know-how must be developed before adequate control of evaporation of films can be achieved. It was therefore deemed advisable to temporarily abandon use of tellurium until sufficient time could be spent on the development of its technology. Tellurium is attractive due to its extremely high thermoelectric power of 400 microvolts /°C, but it does have the disadvantage, in common with other semiconductors, of an inherently high resistivity. This latter property conflicts with the need to produce thermocouples with an overall resistance of less than 10 ohms to drive 30 ohm recording

galvanometers directly. Study of literature available on thermocouples indicated that the combination of bismuth and antimony should be quite desirable. This combination exhibits lower resistances and lower voltage outputs for a given temperature, but sensitivity is nearly the same. The lower resistance is important for efficiency in driving low impedance recording galvanometers.

The bismuth-antimony combination can be vacuum deposited easily and many thermocouples were made using these materials. During this period the rate of production was still slow and the demand for thermocouple detectors was intense. As a result the detectors were shipped out immediately after fabrication and preliminary calibration. This proved to be unfortunate, since it was later discovered that the thermocouples exhibited the same aging characteristics as had been observed with tellurium but to a lesser degree. Also control of vacuum deposition was then not adequate and it was not realized that what seemed to be small variations in the manner in which deposition was made had a substantial effect on thermocouple performance and aging characteristics. Therefore, detector reliability in the field suffered.

At this time the project was still under pressure to instrument field tests, so an intensive program was launched to study the literature and to conduct experiments on vacuum deposition, especially bismuth and antimony. There had been two basic types of failures in the detectors. The first was poor adhesion of films to the Mylar. This was particularly true for antimony. Sometimes the film would show cracks immediately after deposition as though rupture were due to mechanical stress and often the Mylar film would be deformed or appear slightly crumpled. Other times the films would stand up for a period of hours or days before developing a "mud cake" effect. This effect consisted of a frost appearance of the film and under a microscope tiny flakes of the film could be seen. These flaked areas could be brushed off readily and these areas resulted in open circuits or very high resistance.

It was determined that the crumpling of the Mylar film and the open cracks immediately after deposition were probably due to overheating of the Mylar film and heat stresses in the deposited film during deposition and subsequent cooling. The source of the excessive heat was the filament used for evaporation. This type of difficulty was minimized by increasing the distance of the evaporating filament from the detector body and controlling the amount of heat applied to the filament to evaporate the material. The poor adhesion characteristics of the

film were eventually traced partially to the lack of proper cleaning of the Mylar film before deposition and partially to the rate of deposition of the material. The cleaning problem, once it was apparent, was solved by consistent and proper use of ionic cleaning by a high voltage glow discharge prior to deposition. The effects of rate of deposition still need further investigation. However, it appears that too fast a rate, which results in a very dense stream of material traveling from the evaporating filament to the detector body, cause the particles to collide with one another and change direction and velocity. The net result is that the deposit is more like the settling of a cloud than a stream of particles. Also when high densities are present during evaporation, chances of particles colliding with oxygen molecules still present in the bell jar are increased. Thus the first particles deposited may be oxidized and form a poor surface for adherence of the rest of the deposit. It is possible to use a shutter operated from outside the bell jar to interrupt the first evaporation particles until oxygen molecules are either driven away or used up and then the shutter can be moved from the path and deposition of clean material can take place. However, control of the rate of evaporation has substantially eliminated the poor adhesion characteristics of the films.

The second major type of failure of the thermocouple detectors was a progressive increase in resistance with time. This is not a catastrophic failure but did result in the characteristics of the detector wandering from the usable range with passage of time. There was also a lack of consistency in characteristics of detectors produced under supposedly identical conditions. Early attempts at the solution of this problem were directed at more accurate control of the amount of material loaded onto the filament for deposition, rate of deposition and distance. During this time it was noticed that the detectors which had the lowest original resistance and changed least with time were those which had thicker film deposits. A series of tests was then performed in which the amount of material in the bell jar for evaporation was increased gradually. Results were affected by the rate of deposition to some extent. But primarily it was found not only that lower original resistances were obtained with films two to three times thicker than previously used but also that the thicker films tended to have much less change of resistance with time. Empirical values for deposition parameters of amount of material, rate and distance were obtained experimentally. This resulted in thermocouple detectors having original resistances ranging from 6 to 8 ohms up to 15 ohms. After 3 to 4 weeks, the magnitude of resistance increase was of the order of 2 to 5 ohms. This was considered to be quite acceptable for the immediate purposes of instrumentation tests and many detectors were produced and made available for field tests.

During 1960, DRI was relieved of the task of supplying vacuum deposited thermocouples for field testing. This enabled concentration on problems heretofore unsolved. Fundamental studies were initiated. These consisted of measurements of resistance and thermoelectric power of vacuum deposited films as a function of thickness. Also the effects of aging and heat on these films of various thickness were studied. It was necessary to develop some specialized equipment for these studies; for example, the thermoelectric power probe and the multiple beam interferometer for measuring film thickness. Results have been quite a valuable asset in thermocouple improvement.

Evaporation and fabrication of thermocouples has steadily been made more efficient. This has been due to feedback of information resulting from fundamental studies and thermocouple testing as well as improved equipment and handling procedure. Thermocouple testing and evaluation has been extensive; aging, humidity and organic solvent tests were performed. Here again special equipment had to be developed; for example, the reference heat source. The reference heat source has enabled large numbers of thermocouples to be measured for output and time constant and quickly compared. Most recent effort has evolved a thin film substrate thermocouple with a time constant approaching that of the bridge wire, which tends to overcome a major problem in thermocouple application.

The DRI vacuum deposited thermocouple has become the most practical sensor of joule heating in the bridge wires of electro-explosive devices. (See Figure II-1) Advantages are compatibility to automatic recording, adequate sensitivity, adaptability to any type or size electro-explosive device and low cost. Three developmental problems are significant. One, there has been the problem of thermocouple characteristics changes and failure in the field. Two, there is need to determine the degree of perturbation in the instrumentation circuit caused by rf excitation in the bridge wire. Three, the time constant should be reduced to a value much smaller than the bridge wire time constant. Investigation of aging and failures has already had considerable attention. Combination of fundamental studies and measurements and thermocouple testing has brought about understanding of possible mechanisms for past failures and verified thermocouple reliability. This information has made thermocouple fabrication more efficient and enabled the number of failures to be reduced to zero by avoiding their cause. Reasons for calibration changes have been investigated and more effort is needed to minimize or eliminate them. Although thermocouple instrumentation has been shown to be unperturbed by rf bridge wire excitation

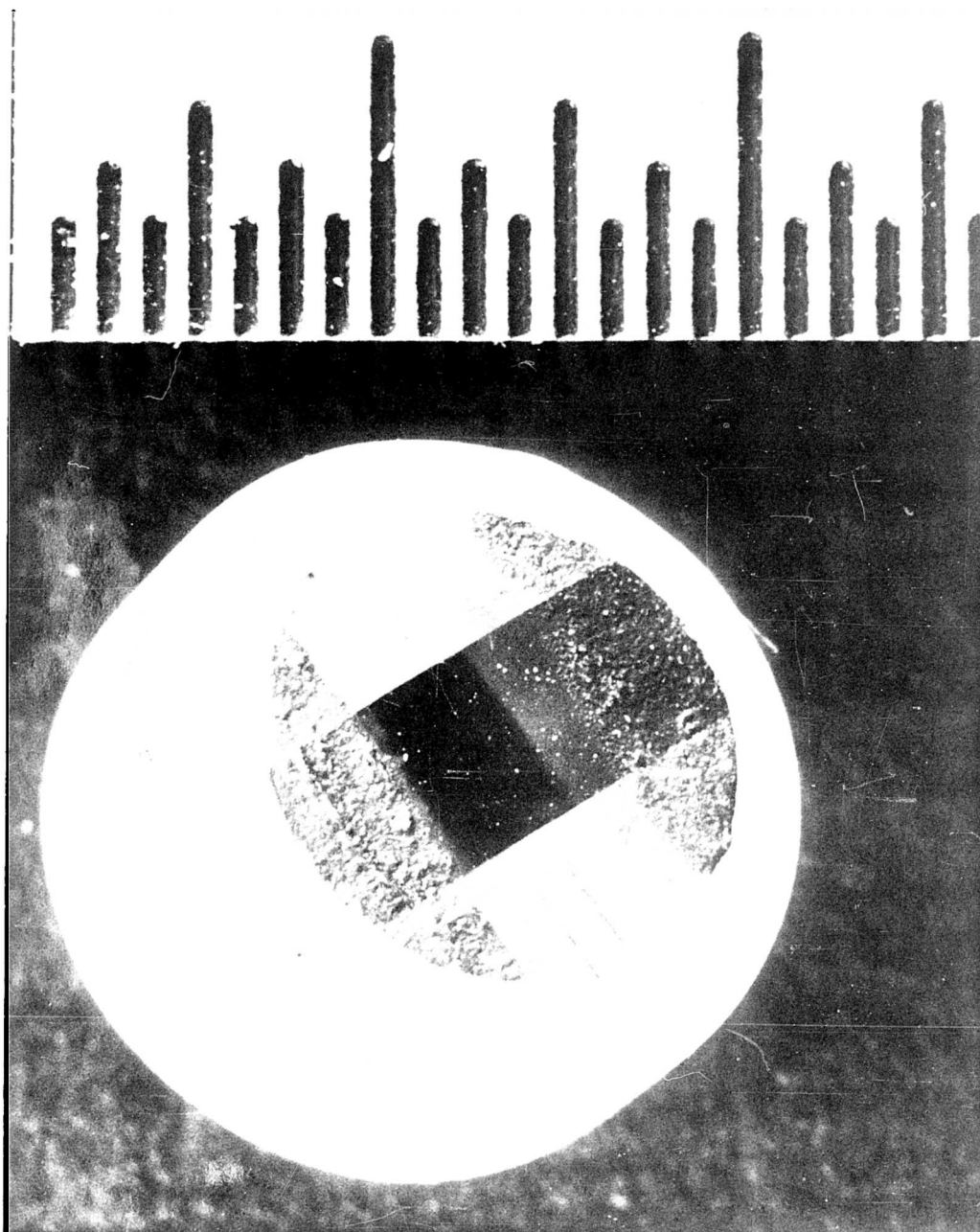


Figure II-1 Mk 1 Type Vacuum Deposited Thermocouple Bismuth-Antimony on Mylar Substrate (smallest division = 1/64 inch)

of 8 Mc and is believed to be unperturbed by rf excitation to the UHF region, at microwave frequencies serious effects may occur. Therefore, it will be necessary to develop and perform calibration tests at microwave frequencies, so that thermocouple instrumentation can be altered to minimize perturbation, if necessary. Recent reduction in thermocouple time constant has given thermocouple instrumentation more favorable status. Indications are that thermocouples can be made to have time constants equal to bridge wire time constants. Here again, more development work is necessary.

III. FUNDAMENTAL STUDIES OF VACUUM DEPOSITED FILMS

Early tests performed with electro-explosive devices instrumented with thin-film thermocouples resulted in occasional malfunctions or complete failure of the thermocouple. Some of the failures were traced to the use of ohmmeters, such as the popular Simpson Model 260 multimeter, for checking continuity through the thermocouple. But for the others, it was apparent that failure was not a result of mishandling or burn-out. In still other instances, instrumented squibs were calibrated and checked from time to time, and the variations noted. Sometimes calibration drifts were observed which were not attributable to changing characteristics of the bridge wire itself. The need for a careful study of the fabrication process, as well as of the metal films themselves, was thus evidenced. This section deals with the research performed on the metal films in an effort to understand and thence improve the characteristics of thin-film thermocouples.

Basic aims in the study of thin-film properties have been the following:

- (1) To determine the variation of film resistivity with thickness
- (2) To learn whether the thermoelectric powers (TEP's) of the films vary appreciably with thickness relative to the bulk values over the range of interest
- (3) To investigate both resistance and TEP as a function of film age
- (4) To study the effects of heat on resistivity and thermoelectric power

The results from this study aid in selecting the proper limits on film thickness for thermocouple fabrication. They also help to point out the causes of thermocouple failure with age by establishing the behavior of the films themselves, apart from small junctions, contacts, and thin substrates.

A. Thin Film Resistivity

1. Background. Occasionally, anomalously high resistances were found in thin-film thermocouples, and were often accompanied by poor response. It was uncertain whether the high-resistance couples were a result of poor films, of poor contact between films, or of poor lead junctions. Since there was no precise knowledge of the various

film thicknesses, it was impossible to say how seriously this parameter affected the thermocouple. Moreover, the resistance of a given thermocouple becomes significant when the read-out device is one of relatively low resistance; therefore, it becomes important to keep the thermocouple resistance at a minimum. * Table III A-1 lists the metals or semiconductors which have been used for constructing thermocouples. The bulk electrical resistivity is listed for these at room temperature.

TABLE III A-1

Electrical Resistivity of Some Common Thermocouple Materials

<u>Material</u>	<u>Resistivity (micro-ohm-cm)</u>
Tellurium	200,000
Antimony	39.0
Bismuth	106.8
Chromel	100
Nickel	6.84
Iron	9.71
Constantan	49.0
Silicon	100,000 (?)
Germanium	89,000

Those materials with high resistivity generally have a very high absolute thermoelectric power, so the relative usefulness of one combination of films over another will be determined by the read-out equipment.

2. Technique. An important study is determining the variation of thin-film resistivity from that of the metal in bulk form. For this purpose, several depositions were performed of each of the above metals onto glass microscope slides as substrates. The slides were prepared by dipping in aqua regia, washing with distilled water and acetone. Glow discharge was used in the vacuum chamber for final cleaning. Generally, for each evaporation, three glass microscope slides were used. One of

*If the open-circuit output voltage of a thermocouple is e_0 and the resistance of the thermocouple and of the read-out device are R_t and R_r , respectively, it is easily shown that the apparent output as seen by the device will be

$$e = e_0 \left(\frac{1}{1 + R_t/R_r} \right).$$

these was prepared by evaporating a thick layer of copper on each end, with a one-centimeter gap remaining bare in the center. A mask was then used across the gap to form a 1 cm by 3 cm film of the metal to be studied. The resistance to be measured was then that of a 1 cm square. Film contacts with the copper were also 1 cm square, insuring low contact resistance.

Eccobond solder was applied at the ends of the slide to form lead contacts from the copper strips to a General Radio impedance bridge. Total extraneous resistance was in each case about 1.2 ohms, generally small compared with the film resistance.

A second glass slide was carefully weighed before placing in the vacuum system. After evaporation this slide was weighed again, and the film thickness calculated from the gain in weight, assuming the film to have a density equal to that of the bulk metal. The weight difference was usually but a few milligrams, making this method rather unreliable, especially for the thinner films. The third slide was partially masked, so that a smooth "step" was formed in the evaporation. This slide was used later to measure the thickness directly by optical methods. (Cf. Part D of this section.)

3. Results. Figures III A-1 to -5 represent the results of the resistivity measurements on antimony, bismuth, tellurium and alumel. For a check on reproducibility, antimony was studied twice (Figures III A-1 and -4.) Agreement was fairly good, although the results in Figure III A-4 show a more rapid increase of resistance with decreasing thickness below 1000 angstrom units. Slopes of these curves vary considerably with film thickness especially for bismuth and antimony. Tellurium, which has a much greater resistivity even in bulk form, does not vary in resistance as rapidly. Reasonable lower limits of thickness appear to be about 2000, 3000, 8500, and 700 angstroms for antimony, bismuth, tellurium and alumel, respectively. However, the results on alumel are still indefinite. Thicker films should be studied, but difficulties are experienced when trying to evaporate this alloy. (Cf. Part B, "Thermoelectric Power.")

4. Effects of Aging. The resistances of the slides prepared as described above were monitored as they aged to determine if any major changes occurred. In general, it was observed that those films having a high initial resistance exhibited the greatest proportional changes with time. These films were usually the thinner ones, so that surface oxidation would be most likely to affect the observed resistance.

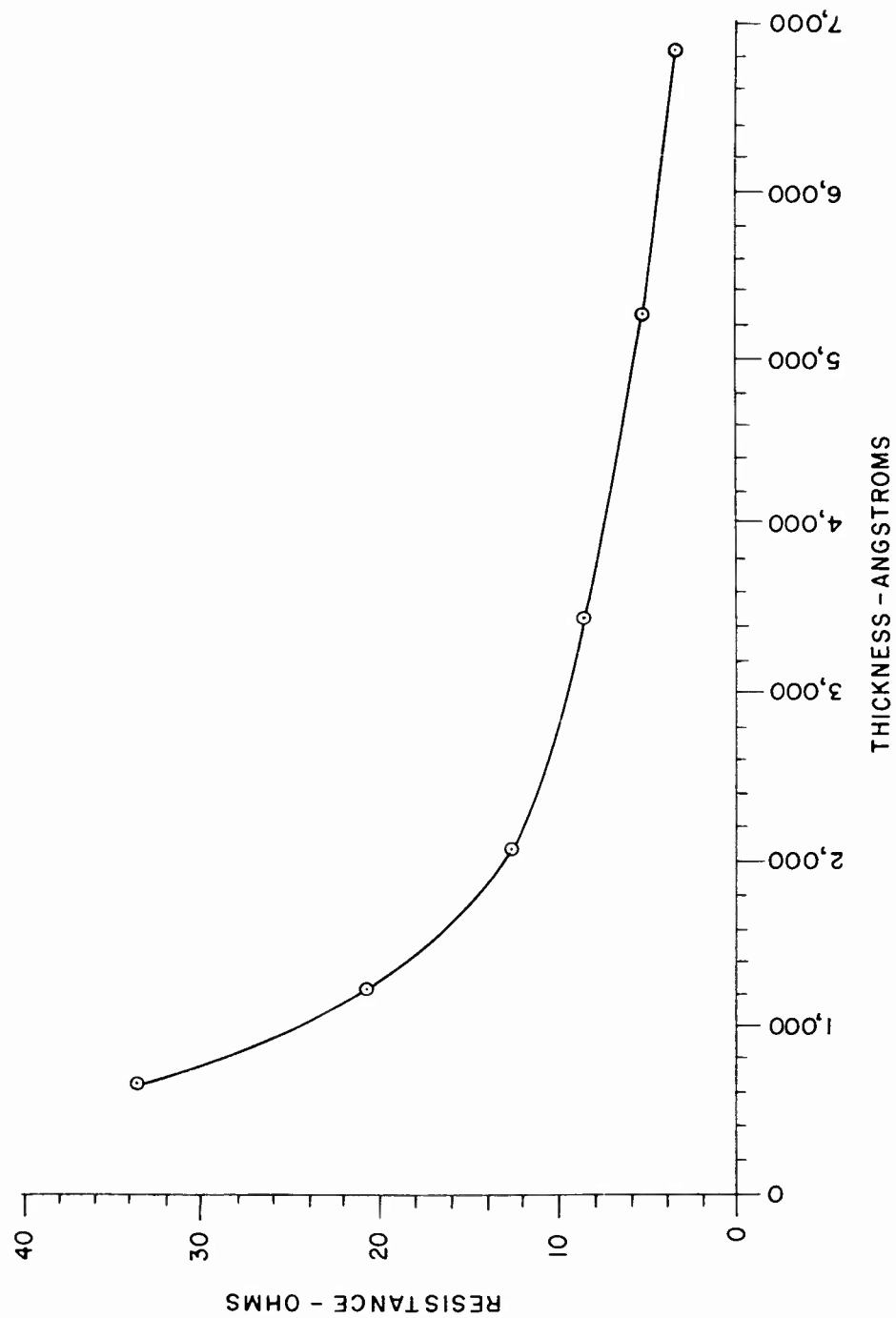


Figure III A-1 Resistance vs. Thickness of Antimony Films
(Group I)

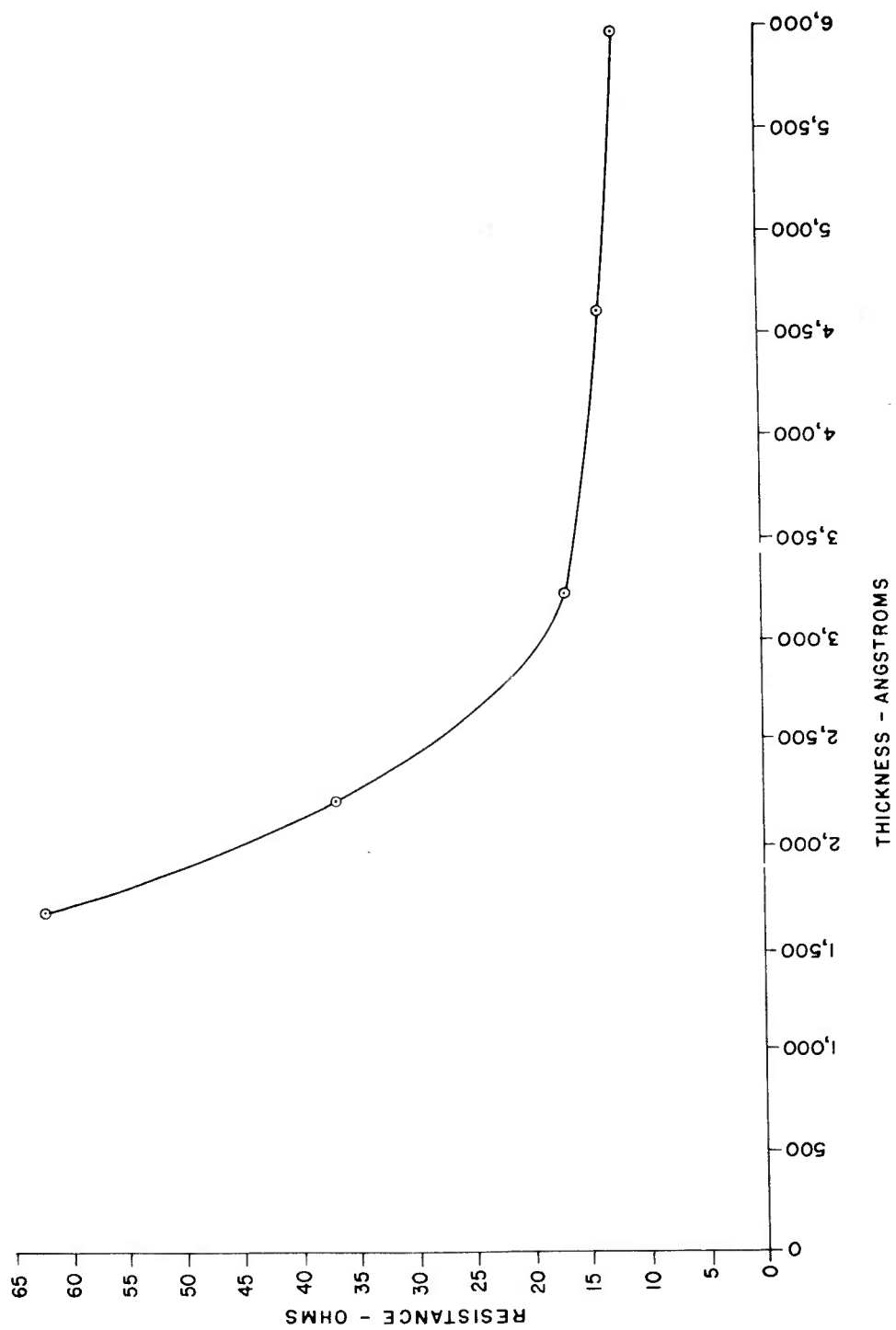


Figure III A-2 Resistance vs. Thickness of Bismuth Film

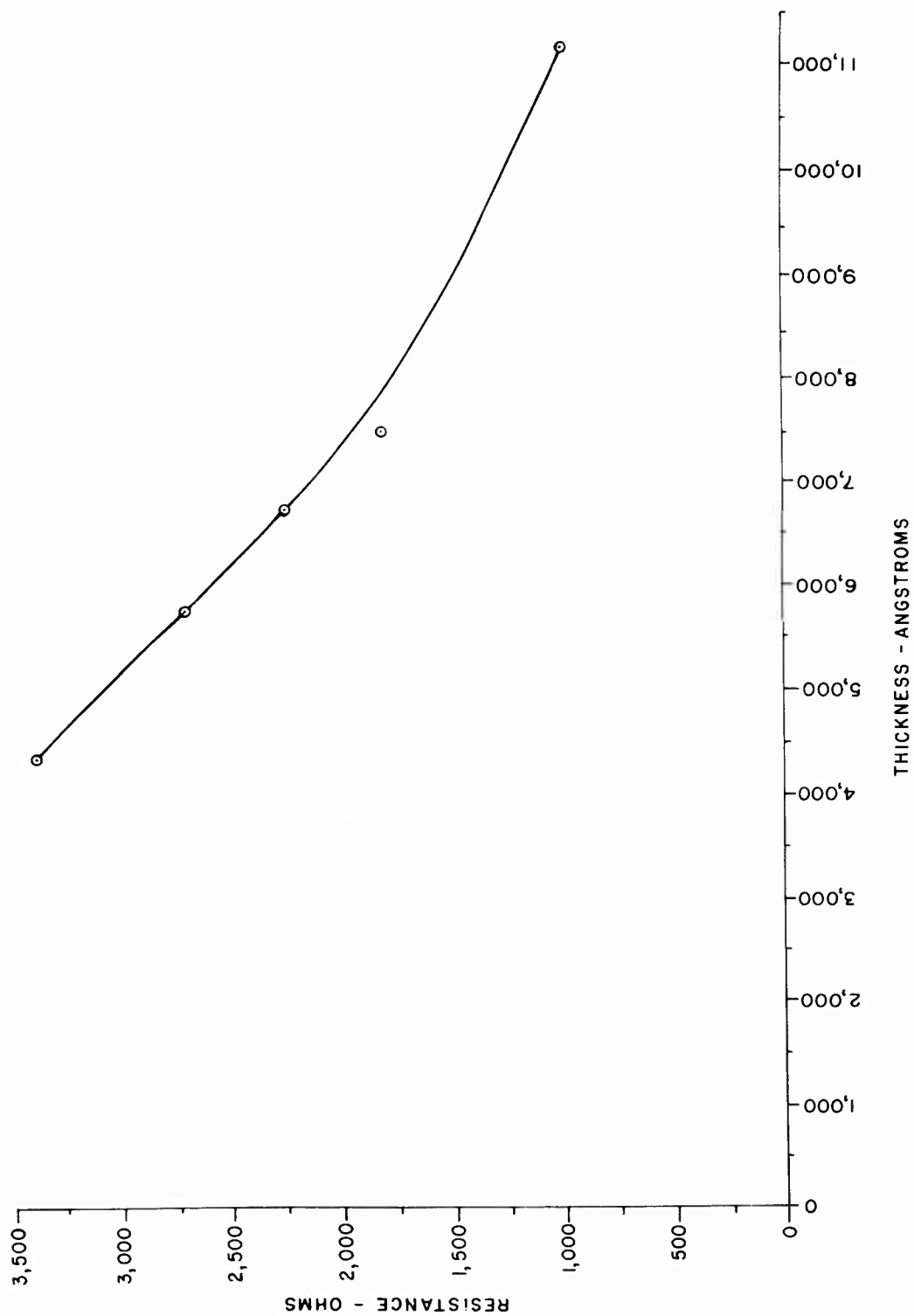


Figure III A-3 Resistance vs. Thickness of Tellurium Film

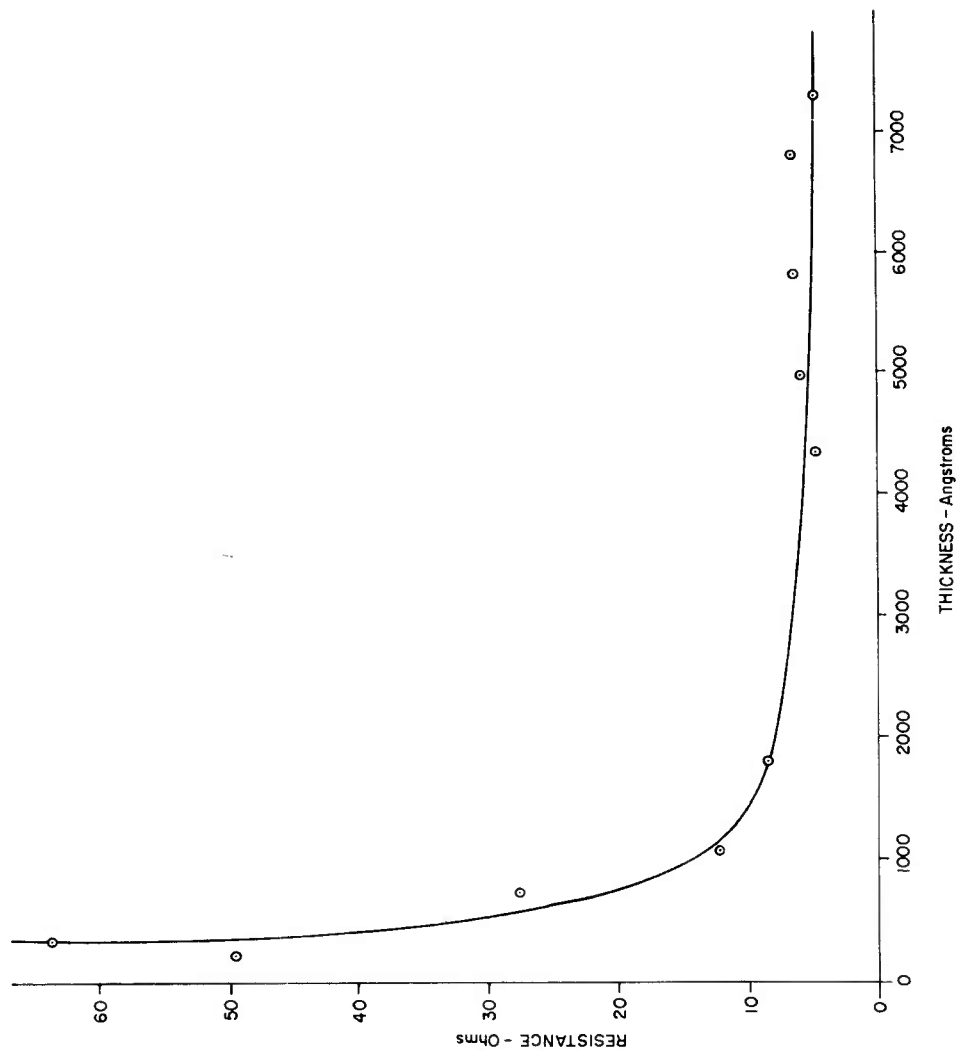


Figure III A-4 Resistance vs. Thickness for Some New Antimony Films (Group II)

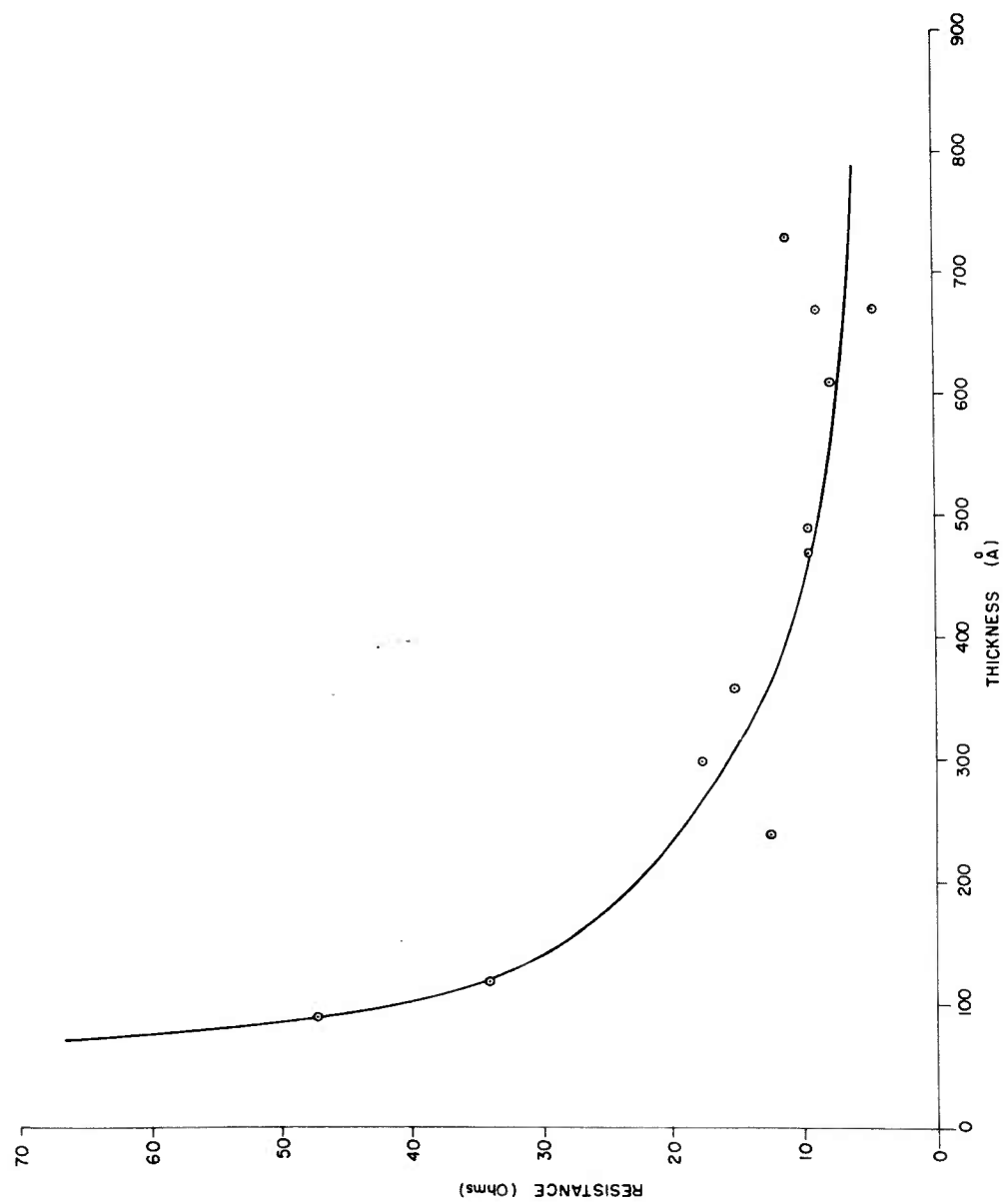


Figure III A-5 Resistance vs. Thickness for Some Thin "Alumel" Films

Representative of these measurements are those made on the antimony and alumel films. The following table gives the change in resistance of these slides, together with the percentage change, for a period of four months. It is concluded that if the resistance variations of these films represent the variations which would be observed had the films been deposited on Mylar, then the aging of the films themselves is not likely to cause failure of a thermocouple unless the initial film resistance is inordinately high.

TABLE III A-2

Variation of Thin Film Resistance with Four Months' Aging

Antimony	Slide #	Final Resistance		Percent Change
		Initial Resistance (Ohms)	(Ohms) (as of 11/15/60)	
	2A	6.3	6.5	+ 3
	3A	6.7	6.0	- 10
	4A	6.5	6.1	- 6
	5A	5.64	5.5	- 5
	6A	4.2	3.64	- 13
	7A	4.45	3.95	- 11
	8A	12.05	11.95	- 1
	9A	7.69	8.6	+ 11
	10A	8.34	8.55	+ 2
	11A	62.0	76.5	+ 23
	12A	27.5	31.7	+ 15
	13A	49.5	58.5	+ 18
	14A	4.67	3.07	- 34
	15A	6.79	7.0	+ 3
Alumel	16A	1200	2400	+100
	17A	47.3	57.2	+ 21
	18A	65.4	73.0	+ 11
	19A	9200	40000	+334
	20A	52.3	52.3	--
	21A	1620	2950	+ 82
	22A	12.7	13.1	+ 3
	23A	77	76.5	- 1
	24A	11.4	9.25	- 19
	25A	4.4	4.3	- 2
	26A	105	125	+ 19
	27A	9.7	9.6	- 1

TABLE III A-2 (Cont.)

Variation of Thin Film Resistance with Four Months' Aging

Alumel	Slide #	Initial Resistance	Final Resistance	Percent Change
		(Ohms)	(Ohms) (as of 11/15/60)	
	28A	9.6	9.85	+ 3
	29A	4.5	4.3	- 4
	30A	11.2	12.2	+ 9
	31A	67	83.5	+25
	32A	34	40	+17
	33A	7.8	7.7	- 1
	34A	15.2	15.7	+ 3
	35A	17.6	19.6	+11
	36A	79	98.5	+25
	37A	9.5	9.75	+ 2
	38A	8.7	8.9	+ 2
	39A	6.0	5.55	- 7
	40A	8.8	8.85	+ 1
	41A	34.5	39.2	+14

B. Thermoelectric Power of Evaporated Films

1. Description of Seebeck Effect. When two dissimilar wires are joined and the junctions held at different temperatures, a thermal emf arises, whose value depends on the materials and on the two temperatures. The open-circuit emf is called the Seebeck effect, and the rate of increase of the Seebeck voltage with temperature is called the Seebeck coefficient. It is often referred to as the thermoelectric power (TEP). Thermoelectric powers vary widely with the two materials chosen. Usually, pure lead is arbitrarily taken as the reference material, and other materials are reported relative to it. Thus, the relative TEP of a given material may be either positive or negative. Tables exist which list the TEP of most of the common metals and alloys in bulk form. Some of the more important for this work are listed below.

TABLE III B-1

TEP of Some Common Materials at 0°C. Relative to Lead

Material	Thermoelectric Power (microvolts per °C.)
Tellurium	+ 370
Antimony	+ 35.58
Bismuth	- 74.42
Alumel	- 10
Chromel	+ 30.30
Nickel	- 19
Iron	+ 16
Constantan	- 38.10
Silicon	-408.5
Germanium	+302.5
Copper	+ 3.1

2. TEP Measurements on Thin Films of Sb, Bi, and Te. The actual TEP of the various films used in making radiation thermocouples was initially unknown. It had been assumed that the TEP for an evaporated film was substantially less than that of the bulk metal, but there was no assurance that the TEP did not vary greatly with film thickness. To investigate the TEP behavior of thin films, a hot copper probe was devised from a small soldering iron which could be used to determine the TEP relative to copper. The probe was first used against lead in order to check its purity. The observed thermoelectric power was +2.9 microvolts per degree centigrade at 30°C for the copper, in close agreement with the value of +3.1 microvolts per degree listed in the Handbook of Chemistry and Physics. This value must be added to the observed TEP to obtain the power relative to lead. Figure III B-1 illustrates the method. Both cold junctions are at room temperature, and it is presumed that the difference in temperature between the two cold junctions is negligible. Thermoelectric emf's were measured at probe temperatures of 13°C, 30°C and 49°C above ambient for films of bismuth, aluminum, tellurium, antimony and copper. The results were found to vary only slightly with temperature. Therefore, because of possible damage caused by higher probe temperatures, the 13°C level was used in most of the thin film investigations.

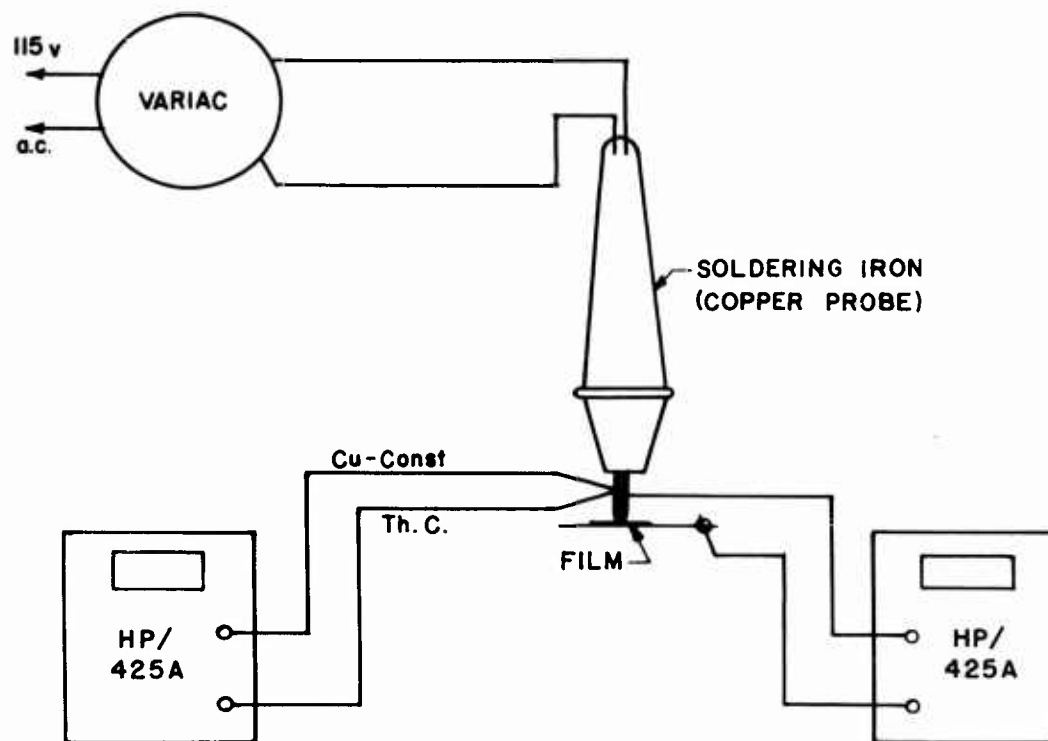


Figure III B-1 Determination of Thermoelectric Power

The first results showed a definite correlation of the film TEP with that of the bulk metal. It was found that in general a film has a thermoelectric power which is a fairly large fraction of that for the bulk form.

The dependence of TEP on film thickness was investigated using one of the three slides prepared (as described in Part A) from each evaporation. The results for bismuth, antimony and tellurium are summarized in Figure III B-2. Throughout the range studied, it thus appears that the TEP's are not a very sensitive function of thickness, and in fact do not vary significantly beyond the minimum limits of thickness set above by the resistance measurements. The resistance is therefore the decisive factor in setting lower limits to film thickness.

3. TEP Measurements on Thin Films of Alumel. Because of the relatively high output obtained from some thermocouples made of alumel and tellurium films, some measurements have been made on the TEP of alumel films. Alumel is a nickel alloy having the following composition:

<u>Metal</u>	<u>Boiling Point</u>
Nickel: 95%	2800°C
Aluminum: 2%	1600
Manganese: 2%	1800
Silicon: 1%	2600

Alumel has a lower resistivity (34.3 micro-ohm cm) than either antimony (44.8) or bismuth (128). However, judging from the TEP of a chromel-alumel couple (41 microvolts per °C) relative to chromel-constantan (63 microvolts per °C), the thermoelectric power of alumel wires must be approximately $-16\mu\text{V}/^\circ\text{C}$ relative to the standard, lead. This would not place it in a more desirable position relative to bismuth, which is closer to $-55\mu\text{V}/^\circ\text{C}$ in the film form. Nevertheless, the alumel-tellurium thermocouple has shown higher sensitivity than the Bi-Te couple. (Ref. 1)

In studying evaporated films of alumel, it must be remembered that the film composition may not necessarily be that of the bulk alloy. This is because of the different boiling points of the metals constituting the alloy; those with lower boiling points, or higher vapor pressures, begin to come off the filament sooner than those with low vapor pressures.

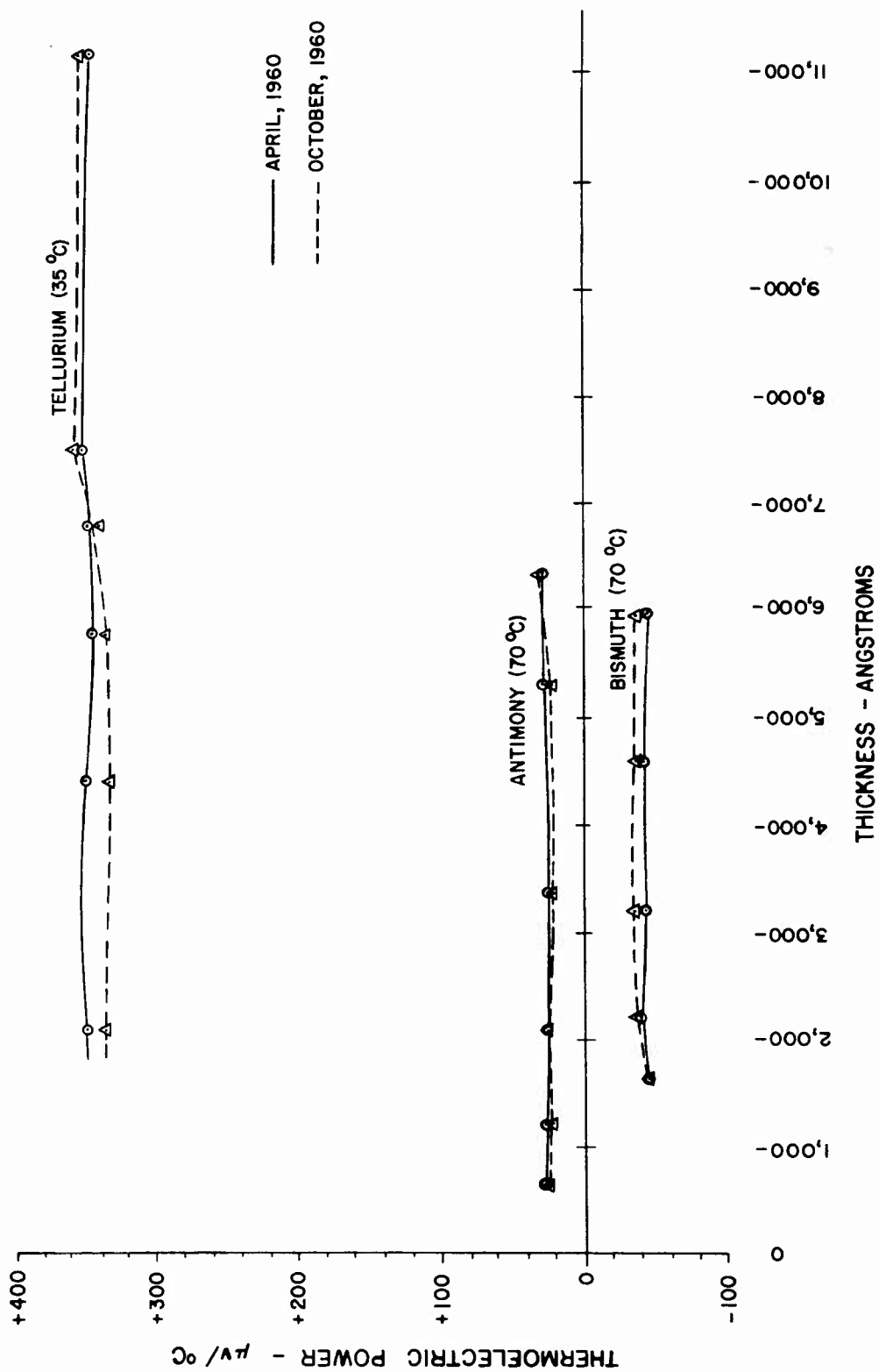


Figure III B-2 Thermoelectric Powers Relative to Copper vs. Thickness for Some Thin Films.

Thus, for alumel, aluminum and manganese are probably more abundant in the vapor during early stages of evaporation. The exact composition is difficult to control, and one may expect considerable variation in any property (such as thermoelectric power) which depends sensitively upon composition. Figure III B-3 shows some of the wide variation obtained in the thermoelectric power of thin alumel films. However, it is clear that in none of the films measured did the TEP appear to be as large as the above estimated value of bulk alumel, and the small, negative values obtained certainly do not account for the sensitivity of the alumel-tellurium thermocouple. Further investigation is necessary.

4. Aging Effects on Thermoelectric Power. The dotted lines in Figure III B-2 represent the results of measurements on the same films after six months' exposure to the air. The overall change in TEP appears greatest for the bismuth films, with an average decrease of 14%. Antimony films have shown a decrease of only 5%, while tellurium has changed, on the average, less than 1%. Not one of all the films studied exhibited a change in TEP of sufficient magnitude to suggest a failure due to this cause.

5. Estimate of Thermal Coupling for Mark 1 Instrumented Squibs. If we assume that the TEP of a thermocouple may be represented by the results in Figure III B-2, it becomes immediately apparent that a bismuth-tellurium thermocouple has a TEP equal to 90% of that for a couple made from the bulk metals, or nearly 400 microvolts per degree. This is in contrast to such common thermocouples as copper/constantan and iron/constantan, which exhibit thermoelectric powers in the neighborhood of 50 microvolts per degree centigrade. Using the values in the figure for the bismuth/antimony combination, the thermoelectric power is 90 microvolts per degree, which is about 80% of the value for the bulk materials.

We next examine the temperature rise of some typical thermocouples in a Mark 1 squib as the bridge wire current is increased. Table III B-2 lists the calibration of two instrumented squibs, showing the thermocouple output in microvolts as a function of current in the Mark 1. Using the above thermoelectric powers, the apparent temperature rise of the thermocouples has been calculated and entered in the third column. Thermocouple No. 256, a bismuth/antimony couple, appears to undergo a slightly larger increase in temperature than the bismuth/tellurium. This may be due to different spacing, a difference in the heating of the bridge wire, or perhaps a larger thermoelectric power than that which has been used. At any rate, the discrepancy is not serious.

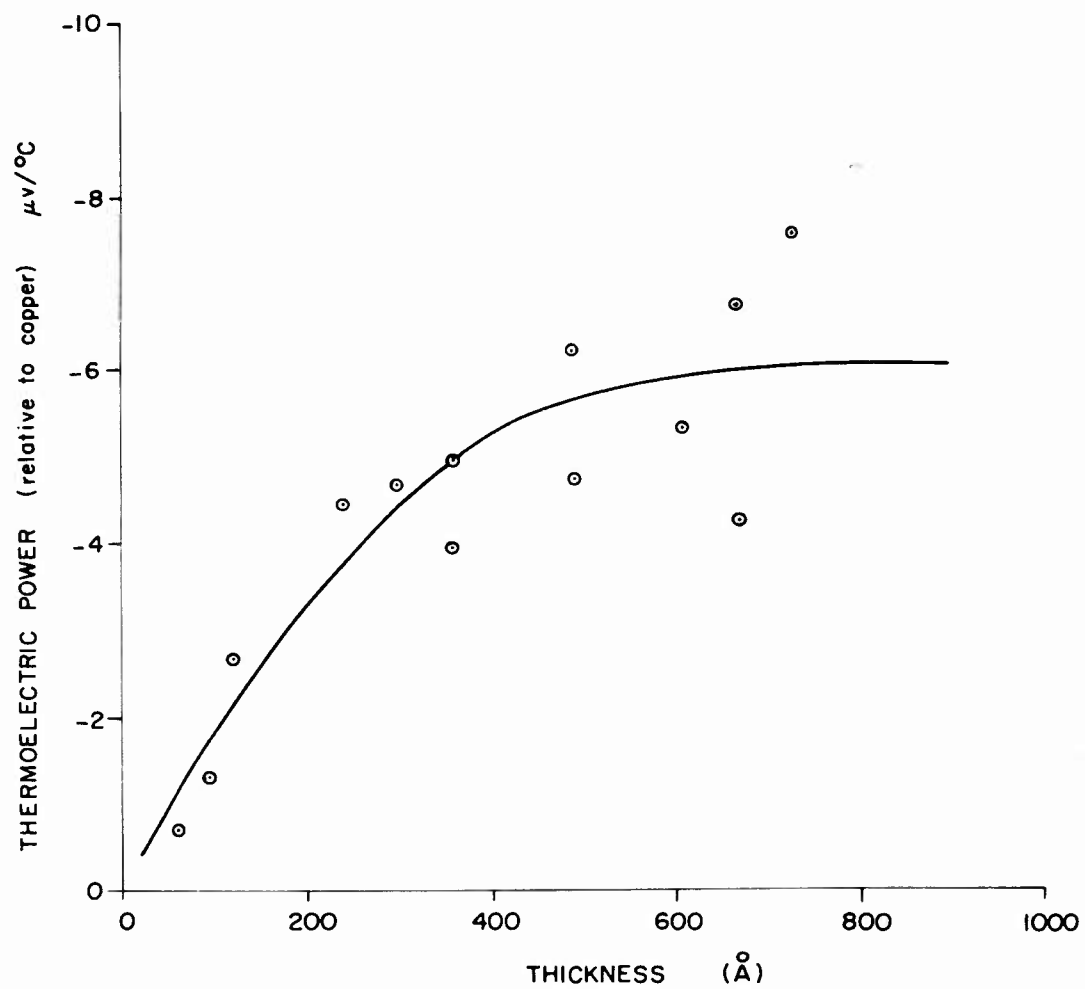


Figure III B-3 Thermoelectric Power of Alumel Films

Finally, using the results of Section V F, a thermal coupling coefficient has been computed, which is defined as the ratio of the apparent temperature rise of the thermocouple to the temperature rise of the bridge wire. In doing this, we are assuming the rise of the wire to be the same in both squibs for a given current level. It is found that the average apparent temperature rise of thermocouple No. 256 is 0.110 times that of the bridge wire, while for No. 265 the average ratio is 0.132. We may thus tentatively conclude that the temperature rise of a Mark I thermocouple represents about one-eighth the rise of the bridge wire.

TABLE III B-2

Thermal Coupling of the Mk I Squib Thermocouple to the Bridge Wire

Squib No. 256 (Bi/Te)				
I (ma)	Output (μ v)	Th. C. Temp. ($^{\circ}$ C)	$\Delta T(\text{wire})(^{\circ}\text{C})$	$K = \frac{\Delta T(\text{Th. C.})}{\Delta T(\text{wire})}$
25	190	0.48	4.17	0.115
50	610	1.52	14.9	0.102
75	1400	3.51	32.0	0.109
100	2450	6.12	57.5	0.106
125	4000	10.0	91.	0.111
150	6000	15.0	127.	<u>0.118</u>
				K=0.110

Squib No. 265 (Bi/Sb)				
I (ma)	Output (μ v)	Th. C. Temp. ($^{\circ}$ C)	$\Delta T(\text{wire})(^{\circ}\text{C})$	$K = \frac{\Delta T(\text{Th. C.})}{\Delta T(\text{wire})}$
25	47	0.52	4.17	0.125
50	165	1.83	14.9	0.123
75	380	4.22	32.0	0.132
100	700	7.78	57.5	0.135
125	1100	12.2	91.	0.135
150	1650	18.3	127.	<u>0.145</u>
				K=0.132

C. The Effect of Heat on Resistivity and TEP

1. Effect on Antimony Film Resistivity. Antimony slides were selected for this study because of the relatively low melting point of this metal (630°C). However, the absence of any deleterious effects on these films does not preclude possible effects on other films, which should be studied in the future.

Figure III C-1 shows the results of measurements on the resistance of four antimony slides as the temperature was increased. The measurements were made in air on films having a thickness of 410, 1900, 3400, and 6800 angstrom units.

Resistance of the thinner films was found to be very sensitive to temperature above 300°F , and after cooling, it was found that the resistance did not return to its initial value. The thicker films did return to lower values, and the 6800 Å film returned to its initial resistance. The jump in the 3400 Å film at 280°F is not understood, but is probably due to a poor initial contact.

From these measurements it is concluded that on glass substrates the films are not damaged by exposure to air at 300°F . At higher temperatures, migration probably occurs, with a resulting "agglomeration" of the metal. When this happens, the resistance change becomes irreversible.

2. Effect on Antimony Film TEP. The four slides described above were subjected to thermoelectric power tests after six months, as were the other antimony slides. The net average change in TEP for these four slides was only about one percent, as against an overall average of five percent. Thus, it is concluded that the exposure to air at 300°C has no observable effect on the thermoelectric power of antimony films. Effects on other types of films have not yet been studied.

D. Optical Measurement of Film Thickness

Early work in which the thickness of evaporated films was required made use of the weighing method. However, because of the necessity of assuming that the density of evaporated films is equal to the bulk density, and because of the large uncertainties in weighing very thin films, such a procedure is subject to some question.

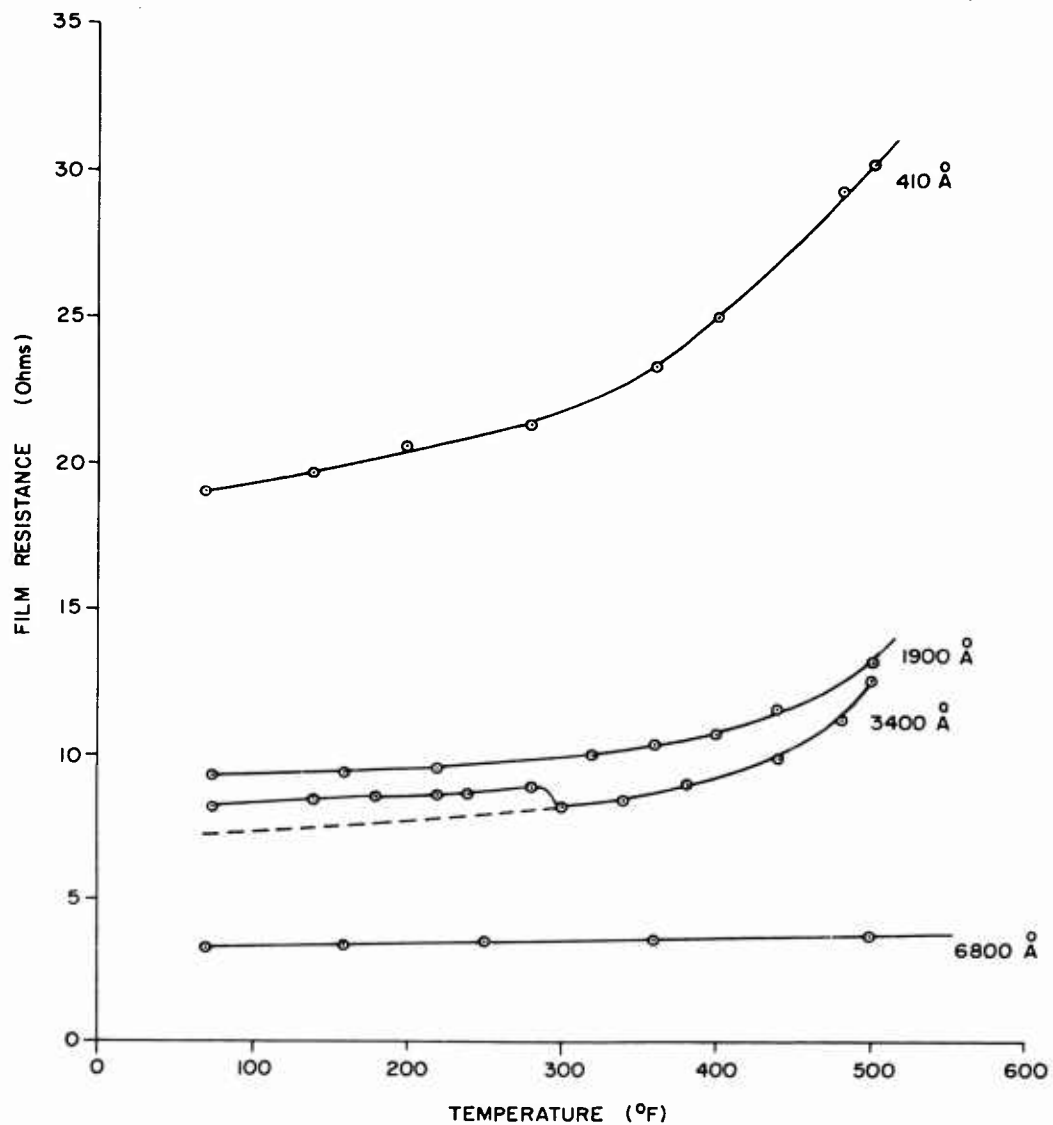


Figure III C-1 Dependence of Resistance on Temperature for Some Antimony Films

An optical method making use of a multiple beam interferometer can be used to measure directly the thickness of a thin film to an accuracy approaching 20 Å. The fringe system utilized is known as "Fizeau fringes." (Ref. 2) Without going into elaborate detail concerning the theory of Fizeau fringes, the experimental conditions necessary for the production of sharply defined multiple-beam Fizeau fringes are that:

- (1) the surfaces must be coated with a highly reflecting film of minimal absorption;
- (2) the coating film must contour the surface exactly and be extremely uniform in thickness;
- (3) a monochromatic source of light must be used;
- (4) the interference-producing surfaces must be separated by only a few wavelengths of light;
- (5) the incident light should be a parallel beam (within 1° to 3°);
- (6) the incident beam preferably should strike the interference surfaces normally.

A procedure for determining the thickness of a thin film developed by W. K. Donaldson and A. Khamsavi is described. (Ref. 3) In Figure III D-1 the film AB to be measured is deposited on part of a flat glass surface, ABC. The uncoated portion of the glass slide is masked off during the deposition and the mask removed after the film has been deposited. A reasonably heavy coating of silver, KLMN, is then deposited uniformly over the entire glass plate. The height of the step, BB', in the film is reproduced on the surface of the coating at LM. Another glass plate, GP, silvered to a thickness of about 500 Å, is brought close to the prepared slide, ABC, and illuminated in such manner as to produce Fizeau fringes. The appearance of the fringes is shown in Figure III D-2. The break in the fringe lines is due to the change in the thickness of the air film between the two reflecting surfaces, KLMN and GP, produced by the step, BB', made by the film whose thickness is being measured.

The arrangement used for measuring the thickness of deposited metal films is shown in Figure III D-3. A sodium lamp was found to be sufficiently monochromatic for the purpose. Lens #1 is located so that the aperture placed in front of the source is at the focal point. Thus, the light falling upon the thin film plate is nearly parallel. The image of the fringes which are formed at the plates is focused by lens #2 so that it can be observed with a microscope. The microscope is equipped with

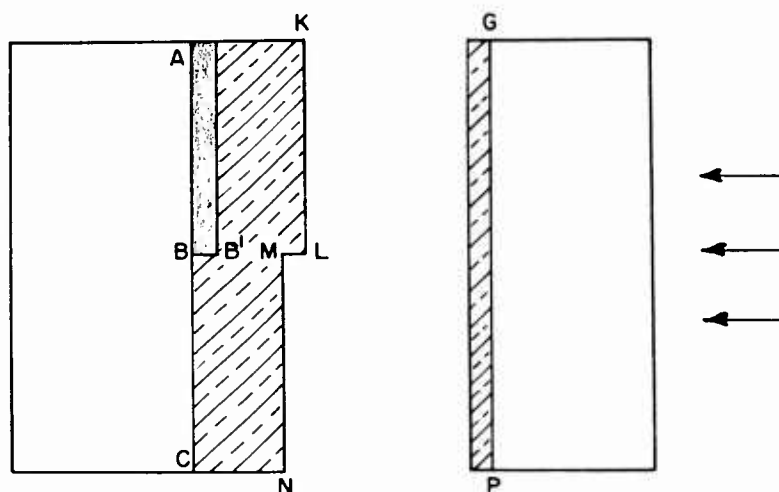


Figure III D-1 Plate Arrangement for Producing Interference Fringe Patterns



Figure III D-2 Fringe Pattern Showing the Effect Produced by the Step of the Thin Film

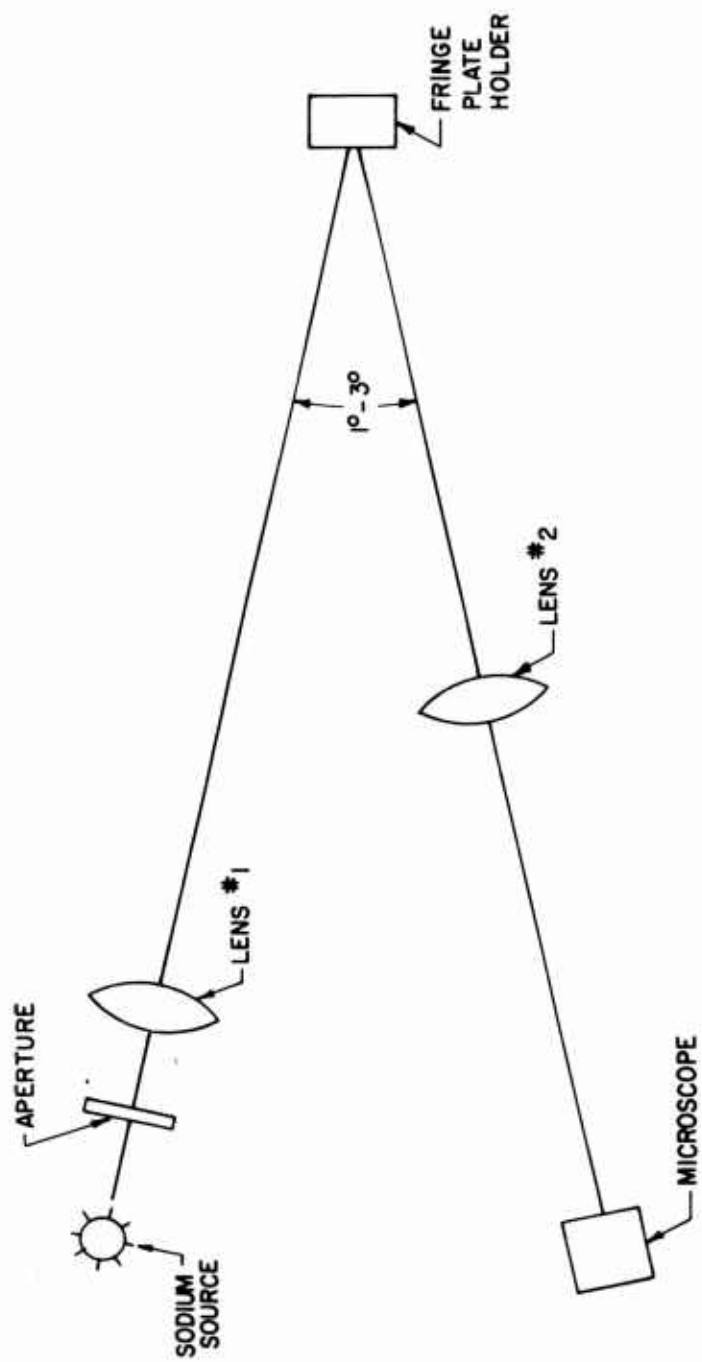


Figure III D-3 Optical System for Measuring Thin Film Thickness

a comparator head for measuring the distance between fringe lines. To obtain the thickness, t , of the deposited metal film, we proceed as follows: 1) Adjust the spacing between the mirror and the step plate, shown in Figure III D-1, to produce a good set of fringes; 2) measure the distance, D , between successive fringes and also the fringe displacement, d , produced by the step; 3) calculate the thickness from the equation

$$t = (d/D)(\lambda/2) \text{ where } \lambda = 5890 \text{ \AA}.$$

The fringe separation and displacement are shown in Figure III D-2.

Actual equipment for making optical measurements has been assembled and put into operation. It serves as a valuable aid in determining the thickness of the thinner films.

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IV. EVAPORATION AND FABRICATION

A. Thermocouple Materials

Three combinations of evaporated films for thermocouples have appeared most promising. These are bismuth-antimony, bismuth-tellurium and alumel-tellurium. Other combinations of materials which have been eliminated are antimony-tellurium, bismuth-chromel, chromel-alumel, chromel-constantan, iron-constantan, copper-constantan, nickel-antimony and nickel-chromel. The most desirable characteristics for a thermocouple are highest possible output in volts per degree C and lowest possible resistance. Table IV A-1 indicates why bismuth-antimony and bismuth-tellurium make the best thermocouples.

Bismuth and antimony are easily evaporated metals, whose properties are relatively insensitive to contamination compared to semiconductors. In addition they make a low resistance thermocouple capable of driving galvanometers directly. For these reasons and because of demonstrated reliability bismuth-antimony thermocouples have become most prevalent.

Tellurium, being a semiconductor, has properties which are sensitive to contamination, thereby necessitating more precise control in evaporation. In addition junctions of other materials with tellurium can exhibit noticeable non-linearity and anisotropy. Tellurium does have a higher conductivity than most semiconductors and because of its high thermoelectric power it is a promising thermocouple constituent. Bismuth-tellurium thermocouples have resistances around 1000 ohms and outputs much greater than bismuth-antimony thermocouples. Although many Bi-Te thermocouples have been made, they have not been used so extensively as Bi-Sb thermocouples.

Alumel-tellurium thermocouples have shown promise of having nearly the same output as Bi-Te but half the resistance. One large difficulty is evaporation of alumel, which is an alloy consisting mostly of nickel. First, as the alloy melts, different constituents boil off according to their boiling point temperatures leaving the nickel, which has the highest boiling point, until last. Second, the boiling point of nickel is so high that frequently the tungsten filament burns out before a significant quantity of nickel can be evaporated. The resulting "alumel" film has an unknown consistency and is necessarily very thin (to prevent filament burn out.) Therefore, much developmental work is necessary before the alumel-tellurium thermocouple can become practical.

TABLE IV A-1

Characteristics of Various Thermocouple Combinations

(November 1959)

Thermo- couple	Bismuth Tellurium		Antimony Tellurium		Bismuth Chromel		Chromel Alumel		Chromel Iron		Copper	
	8.7 Ω	1000 Ω	33,400 Ω	81 Ω	119 Ω	8000 Ω	---	---	---	---		
Resistance												
Squib Current (milliamperes)												
25	.063	.10	0.080	.034	.015	0.008	---	---	---	---	---	---
50	.245	.49	0.300	.135	.053	0.025	.009	---	---	---	---	---
75	.595	1.18	0.720	.320	.125	0.065	.017	.003	.005	.009	.003	.005
100	1.08	2.15	1.330	.580	.230	0.122	.028	.005	.028	.044	.009	.009
125	1.68	3.40	2.200	.920	.395	0.195	.044	.009	.044	.068	.013	.013
150	2.48	5.3	3.200	1.38	.555	0.310	.068	.013	.068	.013	.013	.013
			Output Voltage (millivolts)									

REMARKS:

Chromel-Constantan: Chromel deposit thin. Output unstable on low values of current input.

Iron-Constantan: Iron deposit was very thin due to filament burning through.

B. Terminal Connections

Connection between the output wires and the evaporated films of thermocouples is a difficult problem. Silver ink has almost always been used for the connection; however, at one time this method was suspected as the cause of much of the change in characteristics due to aging and unreliability. For this reason tests of thermocouples using various schemes of connections were made. Table IV B-1 shows the results of these tests.

TABLE IV B-1

Thermocouple Characteristics for Various Types of Connection to the Terminals

Thermocouple	Silver Ink	Copper Deposit	Gold Deposit
	resistance (ohms)	resistance (ohms)	resistance (ohms)
1	3.64	4.74	16.15
2	3.04	4.22	136.0
3	6.45	9.5	43.4
4	3.24	2.87	43.1

The silver ink connection provides the lowest resistance and least variation in resistance. Copper and gold films do not provide satisfactory connections. This is most likely due to unavoidable cracks between the wire and the polyester base plug and scratches and grooves in the plug. The silver ink bridges all of these gaps whereas evaporated films of copper and gold may not be able to accomplish reliable bridging of larger gaps.

C. Movable Mask

A new mask has been developed for the deposition of the thermocouples making it possible to deposit the films without breaking vacuum. Steps which are possible under continuous vacuum include: (1) glow discharge of the entire face surface of the polyester base, silver ink and Mylar; (2) bi-metallic deposit of thermocouple; (3) silicon-monoxide deposit for protection. By changing various types of inserts in the top of the insert plate, thermocouples for different squibs can be deposited simultaneously. The mask contains eight inserts, which when filled double the number of thermocouples that may be produced at one time.

These inserts are made to allow the polyester block to fit snugly against the target face. Two brass plates then form a slit across the insert which is the width of the thermocouple.

The movable mask will block deposition on one-half of the thermocouple. Moving the mask position to the reverse stop will then complete the bi-metallic thermocouple. At present, the stops on the mask are placed to allow an 0.008 inch overlap at the hot junction. This overlap dimension can be varied by changing the position of the stops. See Figure IV C-1. Positioning the mask midway between the two stops exposes the entire thermocouple surface for treatment. A simple pointer and scale outside the vacuum system is used to align the mask properly since earlier deposits cover the surface of the bell jar with an opaque film. Glow discharge and deposition of a protective coat are accomplished through the circular opening. Movement of the mask while inside the bell jar is accomplished through a rotary motion seal. See Figure IV C-2.

The thermocouple squib is placed in the mask so the films will be centered over the copper terminals formed by the wire leads. This step may be done at a work bench since the insert plate can be detached from the turning mechanism. After placing this plate in position, the mask is centered to expose the thermocouple area to the glow discharge. When the vacuum reaches 0.03 microns of mercury, the mask may be turned against a stop and one of the metals forming the thermocouple deposited. After allowing a short time for this filament to cool sufficiently, the mask is moved against the other stop, and the second metal deposited. Each deposit requires a separate filament and set of electrodes inside the bell jar. Care must be taken in placing these filaments so that metal from one deposit cannot contaminate the filament containing the second. Finally the mask is positioned back to center by the external pointer and scale for a protective covering over the thermocouple. The insert plate and thermocouples may be removed from the system after the filaments have had sufficient time to cool.

In using this mask nearly all contamination of the first deposit due to air, moisture, etc., has been eliminated. This advantage is shown by nearly a 20% increase in output and a lower resistance compared with thermocouples produced in the older type masks. It also eliminates two pump-downs from atmospheric pressure thereby reducing the previously required time to complete a thermocouple to less than one-half.



Figure IV C-1 Insert Plate Showing Moveable Mask

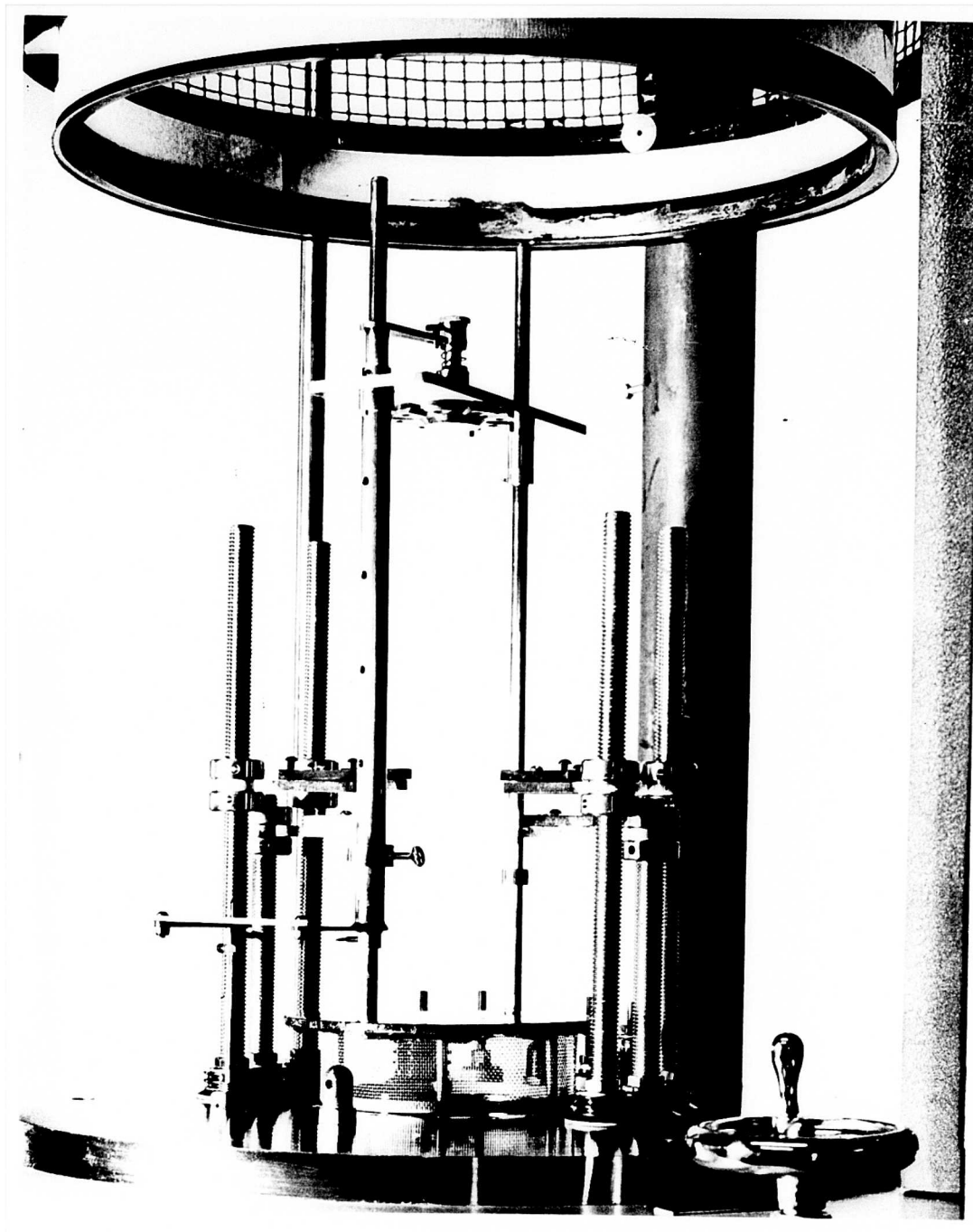


Figure IV C-2 Vacuum Chamber with Mask in Place

D. Casting Thermocouple Base Plugs

A major advance in casting thermocouple base plugs has resulted from developmental efforts during this quarter.* The plug material must have good machining properties, a low rate of outgassing in vacuum and must also be quick setting. The first type of polyester resin used for casting thermocouple base plugs required over two hours' setting time before removal from the mold and two additional hours before machining could be accomplished. In addition, some large and complex plugs had to be cast in parts because of difficulty in removal from the mold. The present type of polyester resin and catalyst requires about ten minutes' setting time for removal from the mold and machining can be accomplished immediately thereafter. This means a 3-3/4 hour saving in plug fabrication.

The new plastic used is a polyester casting resin which is supplied as a liquid plus a liquid catalyst. The resin is Laminac #4110 by the American Cyanamid Company, New York 20, N. Y. The catalyst is a solution of methyl ethyl ketone peroxides and hydroperoxides in dimethyl phthalate. It is Lupersol Delta, a product of the Lucidol Division of Wallace & Tiernan Inc., 1740 Military Road, Buffalo 5, N. Y. Curing time required for complete setting of thermocouple plugs can be changed by varying the mixture of catalyst and resin and by controlling the amount of heat applied to the mixture. The removal of plugs from molds is facilitated by coating the insides of molds with wax from a spray can. This product is 5% Vydex A, a Fluorocarbon Telomer Wax, from E. I. duPont, Organic Chemicals Dept., Wilmington, Delaware. Even large and complex plugs can be removed from their molds when this wax is used, and one coating lasts for many castings.

The present procedure for casting thermocouple plugs is to mix a convenient ratio of one drop of catalyst to one milliliter of resin in a disposable 1/2 ounce paper cup, thereby eliminating the need for cleaning semi-hard plastic from glass vessels. The mixture is then poured into molds. The molds in turn are placed on a large metal block, which is kept near 200°F by means of a hot plate. Plugs can be removed from their molds in approximately six minutes, and machined almost immediately thereafter. Usual procedure, however, is to allow ten minutes' curing time.

*This work was accomplished through consultation and direction of Dr. D. N. Gray, Chemistry Division, Denver Research Institute of the University of Denver.

It has been found that the aluminum coating, which was vacuum deposited on the trough of the base plug to enhance infrared radiation received by the hot junction of the thermocouple, has negligible effect on thermocouple sensitivity. Averages of outputs of several thermocouples with and without the aluminized troughs have been compared on the reference heat source and found to be equal within experimental variation. Therefore, aluminizing of base plugs is no longer a part of the thermocouple fabrication process.

V. TESTING AND EVALUATION

A. Reference Heat Source

The problem of being able to compare the response characteristics of various infrared sensors has been of concern for some time. One of the major difficulties has been obtaining a source of sufficient intensity which could be controlled accurately. Another difficulty has been the control of the "on" and "off" time of the source so that time constant measurements may be made. A method of achieving these controls has been designed and developed using a heavy nichrome wire as a source of heat and a motor driven chopper wheel. Figure V A-1 is a block diagram showing the reference heat source. Figure V A-2 is a sketch showing the system for measuring time constants, and Figure V A-3 is a photograph of the reference heat source. Figure V A-4 is a photograph showing the heat source in use with a Tektronix scope or Hewlett-Packard microvoltmeter.

The source of heat is a one-eighth inch diameter nichrome wire connected directly to a heavy duty filament transformer by means of heavy copper buss bars. The transformer is capable of delivering 30 amperes at 6.3 volts. The amount of heating is controlled by means of a type V2 Variac connected to the transformer. A chromel-constantan wire thermocouple is welded to the portion of the nichrome heater wire which irradiates the sensor being tested. This thermocouple used in conjunction with a millivoltmeter monitors the temperature of the heat source. The baffle immediately in front of the heat source has a one-eighth inch aperture to allow a narrow beam of radiation to pass through to the sensor under test. Adaptors have been made to hold the various infrared sensors. For thermocouples the adaptor has an aperture just large enough to allow radiation to strike the "hot" junction but shield the "cold" junctions from being heated.

With the thermocouple under test in place, current to the nichrome heater is adjusted until the monitoring wire thermocouple registers a predetermined setting on the millivoltmeter. The output from the thermocouple under test is measured with the Hewlett-Packard 425A microvoltmeter. By maintaining the temperature of the heater at the desired value, the output of a number of thermocouples can be compared quickly. Since it is possible by means of the monitoring thermocouple to obtain the same temperature of the heat source from day to day, this arrangement can be used to follow any possible changes in output for a group of

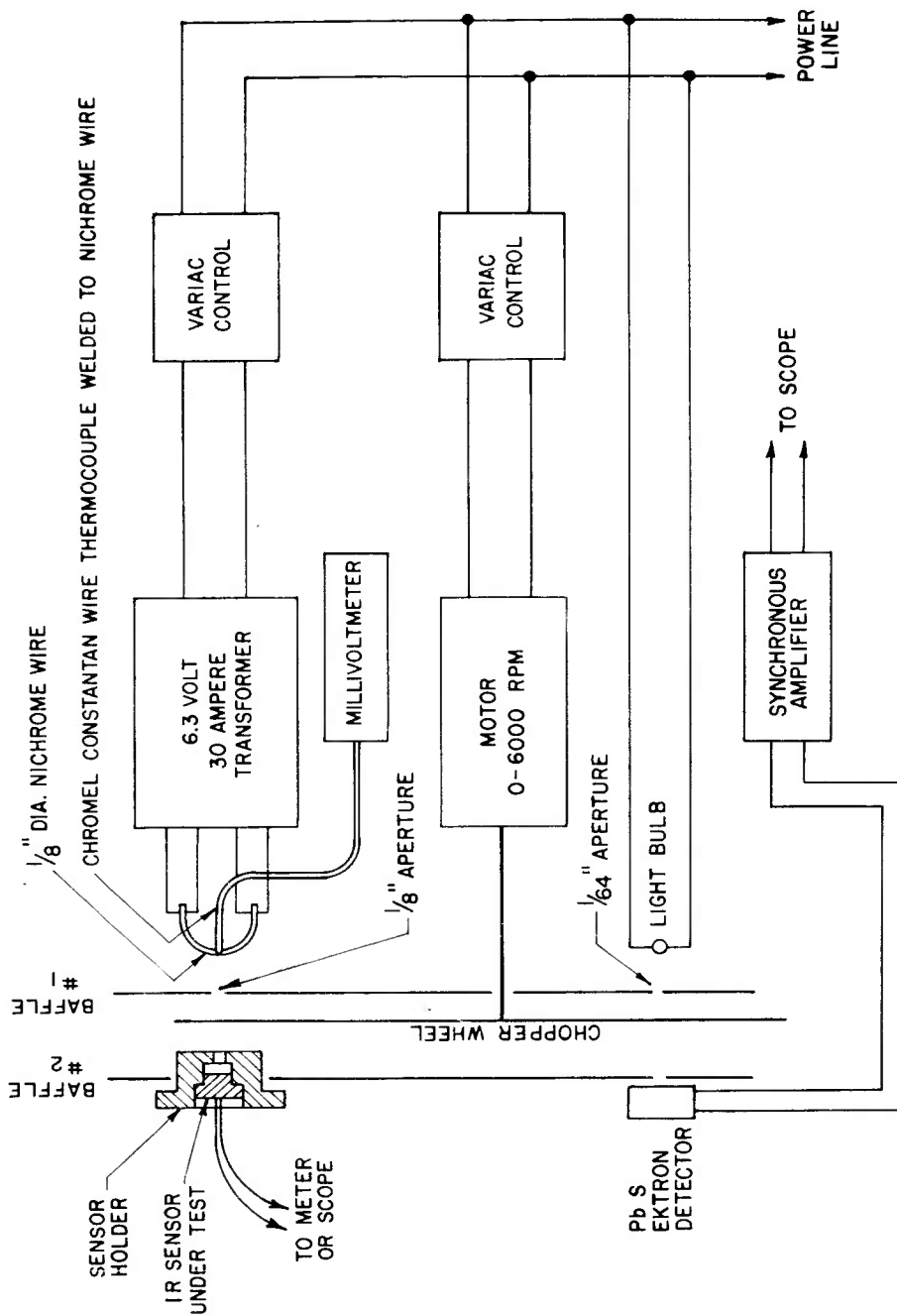


Figure V A-1 Reference Heat Source Block Diagram

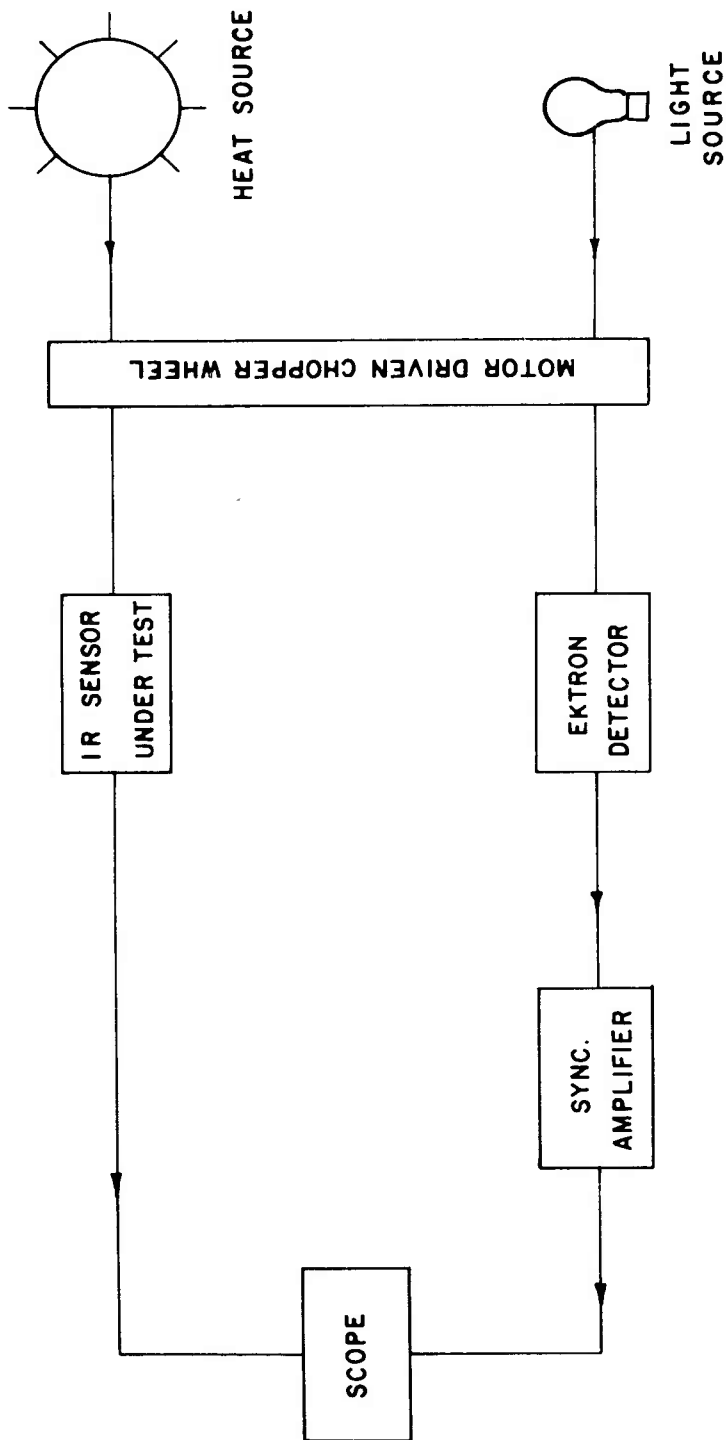


Figure V A-2 Time Constant Measuring System for IR Sensors

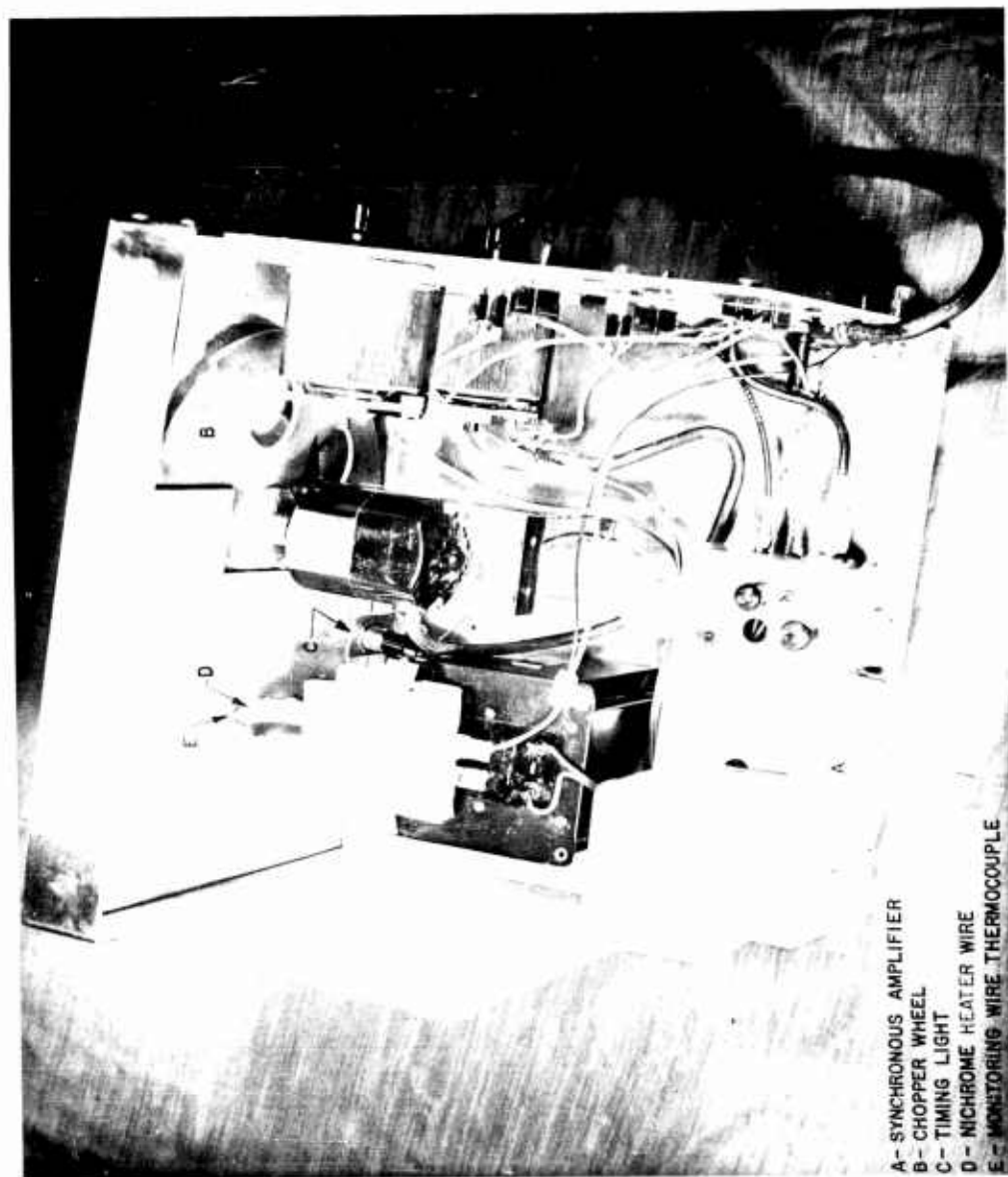


Figure V A-3 Reference Heat Source



Figure V A-4 Reference Heat Source and Associated Equipment

thermocouple detectors over a period of time. Used in this manner the apparatus provides a reference heat source for thermocouple comparison and development.

This system has proved to be a far more reliable and meaningful method of comparing detector thermocouples than did the practice of using an EED for the source of heat. With this apparatus, which has an intense heat source at a greater distance from the detector, small variations in source to sensor spacing of the order of a few mils are insignificant whereas the same variation in spacing when a bridge wire is used for the heat source produces drastic differences in the output of the thermocouple. In addition this method is far more economical of time and materials.

This heat source has replaced the equipment described in DRI Quarterly Report No. 33 (Ref. 4). It was found that the projection lamp was not a suitable source to be used for the development and improvement of infrared sensors. Detectors which showed improved output when tested with this lamp showed very little increase when illuminated with longer wave length radiation. In addition no satisfactory provision had been made to measure the intensity of the projection lamp, and it was difficult to be sure of the amount of illumination from test to test. For these reasons the nichrome wire heat source described above was developed and has been used for reference work in testing and developing infrared sensors.

In order to provide a means of making time constant measurements on infrared sensors a motor driven chopper is arranged to operate in the space between first baffle and the holder of the infrared sensor. The chopper is a circular metal disc having an outside radius of five inches. The wheel is turned by a variable speed ac motor. The speed of the motor can be varied by a type V2 Variac between 0 and 6000 rpm. To measure time constants of sensors which have a slow response time it is desirable to have the "on time" of the chopping cycle long enough to enable the sensor to reach its maximum response before the signal is shut off. Such sensors are also slow in cooling so it is equally important that sufficient time be allowed for cooling before a signal is applied again. Yet it is necessary to make radiation turn-on and turn-off time as small as possible. For this reason the chopper wheel diameter was made relatively large and the wheel gives only one interruption of radiation per revolution. This gives a very large ratio of period to turn-on time, which is necessary for accurate time constant measurements. The wheel is dynamically balanced to operate at speeds up to 6000 rpm.

Measurements of the response time of rapidly responding sensors such as the semiconductor photo sensitive detectors require that the rise time of the radiation beam be short. For this reason the aperture to be chopped by the wheel should be as small as is consistent with keeping the intensity of the beam at a detectable level. The size of aperture finally chosen was approximately one-eighth inch in diameter. The time required to open the aperture is the diameter, d_a , of the aperture divided by the velocity with which the edge of the slot in the chopper wheel passes the opening. This velocity is determined of course by the rate at which the wheel is turned,

$$V = 2 \pi fr$$

where r is taken as the mean radius of the slotted section of the wheel and f is the frequency in revolutions per second. Then turn-on time of the radiation beam is

$$t = d_a / 2 \pi fr$$

Using the dimensions for the aperture and mean radius of the slot we find the approximate rise time for the source to be $t = \frac{4}{f}$ milliseconds.

Table V A-1 shows the rise times corresponding to different speeds of the chopper wheel.

TABLE V A-1

Rise Time of Radiation Beam	
Chopping Rate (rpm)	Approximate Rise Time (milliseconds)
60	4
240	1
480	.5
960	.25
2400	.1
4800	.05
6000	.04

These values are considered to be quite adequate. In order to achieve a shorter rise time it would be necessary to reduce the size of the radiation beam aperture and/or increase the radius of the chopper wheel or its speed. If the radius of the wheel were increased, it would make the apparatus unwieldy and require a larger motor to turn it. Any

significant increase in speed would require special motors or bearings and could prove hazardous to operate if the wheel should fly apart. It is also to be noted that as the speed of the chopper is increased the heat source element is cooled by convection currents of air flowing through the radiation beam aperture. In fact, at the highest speed the potential measured by the monitoring thermocouple is reduced to approximately one-half the value obtained when the chopper is not turning. In addition to cooling the source these convection currents also produce a cooling effect on the sensor being tested thus leading to erroneous readings at the high chopping rates.

Reducing the size of the aperture results in a decrease in intensity at the detector being tested. If the intensity of the source is increased in order to compensate for this decrease at the receiver, it is necessary to elevate the temperature of the source. This results in radiation predominant in the visible spectral region which is not the region which is of interest; i. e., the infrared region characteristic of lower temperatures. Therefore, the present choice of parameters appears to be a very workable combination.

The "on time" or exposure time is, of course, in all cases, approximately 125 times the rise time of the beam. Thus, if we choose an exposure time to allow a particular type of sensor to reach its maximum response, we find the rise time of the beam is sufficiently short to be used for determining the response time or time constant of the sensor under test. The results of tests conducted with this apparatus are presented in other sections of this report.

B. Aging Test

PURPOSE: The purpose of the aging test was to determine the changes in Bi-Sb Mk 1 Squib thermocouple characteristics during a 100 day period.

In the past large variations in the resistance of thermocouples in instrumented EED's have been measured. In many cases the resistance continued to increase until, after a few days or weeks, the thermocouple was an open circuit. The thermocouple failures were believed to have been caused by the deterioration of the silver ink that is used as the terminal material to connect the thermocouple films to the copper leads. Two types of protective coatings have been used on thermocouples in attempts to reduce the aging. They are deposited SiO coatings and

painted lacquer coatings. Deposited copper was used instead of silver ink as the terminal material on several groups of thermocouples. The two types of protective coatings were also applied to these thermocouples.

Lacquer coated, SiO coated and uncoated thermocouples were tested for a period of 100 days to determine the effects, if any, of these coating materials on thermocouple characteristics. Thermocouples with copper terminals and silver ink terminals were tested to compare stability using these two materials.

PROCEDURE: All the thermocouples were kept at room ambient conditions for 100 days. Four resistance measurements were made on each thermocouple. They were:

- (1) the initial resistance of the thermocouple the day it was made.
- (2) the resistance when the thermocouple was 20 days old.
- (3) the resistance when the thermocouple was 50 days old.
- (4) the resistance when the thermocouple was 100 days old.

Only Bi-Sb Mk 1 Squib thermocouples with initial resistance less than 10 ohms were used in the test. Variations in the output voltage of the thermocouples during the test period could not be determined because no reliable reference heat source was available at the beginning of the test. Since thermocouple failures are accompanied by large changes in resistance, the measurement of thermocouple resistance is an indication of its stability.

RESULTS: The results of the aging test appear in Table V B-1. The total resistance change during the 100 days of each thermocouple is shown in Figure V B-1.

Two failures occurred in the 35 thermocouples tested. Both failures were thermocouples with copper terminals and they occurred in less than 20 days. The remaining 10 copper terminal thermocouples had an average increase in resistance of 2.1 ohms compared to an average increase of 0.9 ohms for the 23 silver ink terminal thermocouples. Another method of comparison shows that 58% of the copper terminal thermocouples and 9% of the silver ink terminal thermocouples has a resistance change greater than 2 ohms.

A comparison of resistance changes and the coating materials on thermocouples made with silver ink terminals shows that one uncoated

TABLE V B-1

Aging Test on Bi-Sb Mk 1 Squib Thermocouples

Terminal Material	Coating	Thermo- couple No.	Resistance			
			Initial	20 days	50 days	100 days
Ag ink	none	235	2.9	-	2.9	2.9
"	"	157	3.1	5.6	5.5	5.2
"	"	288	4.1	3.6	3.8	4.1
"	"	287	4.5	4.3	5.5	6.2
"	"	286	4.7	4.3	5.0	4.4
"	"	289	4.7	4.1	4.2	4.2
"	"	167	5.1	5.1	-	5.5
"	"	103	7.1	-	8.3	8.4
Ag Ink	SiO	240	3.0	5.1	7.2	9.5
"	"	242	3.2	3.3	3.3	3.5
"	"	239	3.6	3.7	3.8	4.0
"	"	216	4.1	4.2	4.3	4.5
"	"	209	4.7	4.8	5.2	5.4
"	"	210	4.7	5.1	5.3	5.5
"	"	215	4.7	4.9	5.3	5.5
"	"	300	5.0	5.0	5.3	5.4
"	"	299	5.2	5.6	6.3	6.6
"	"	213	5.3	5.5	5.8	6.4
"	"	234	6.4	6.4	-	7.0
"	"	241	6.5	7.5	6.7	7.2
"	"	214	10.0	10.2	10.3	10.3
Ag ink	Lacquer	297	6.7	6.5	6.5	6.6
"	"	298	6.8	7.1	7.3	7.3
Copper	none	220	3.2	∞	-	-
"	"	227	4.2	-	4.8	4.7
"	"	226	4.8	-	6.2	7.2
"	"	295	8.7	9.7	12.7	12.4
"	"	291	9.8	∞	-	-
Copper	SiO	249	2.9	3.0	3.1	3.2
"	"	247	4.2	6.0	6.9	8.9
"	"	248	9.5	6.6	5.1	5.3
Copper	Lacquer	290	6.0	5.5	15.6	15.8
"	"	293	6.0	6.0	6.0	6.2
"	"	292	6.3	7.3	7.4	7.5
"	"	294	6.5	6.5	8.2	8.1

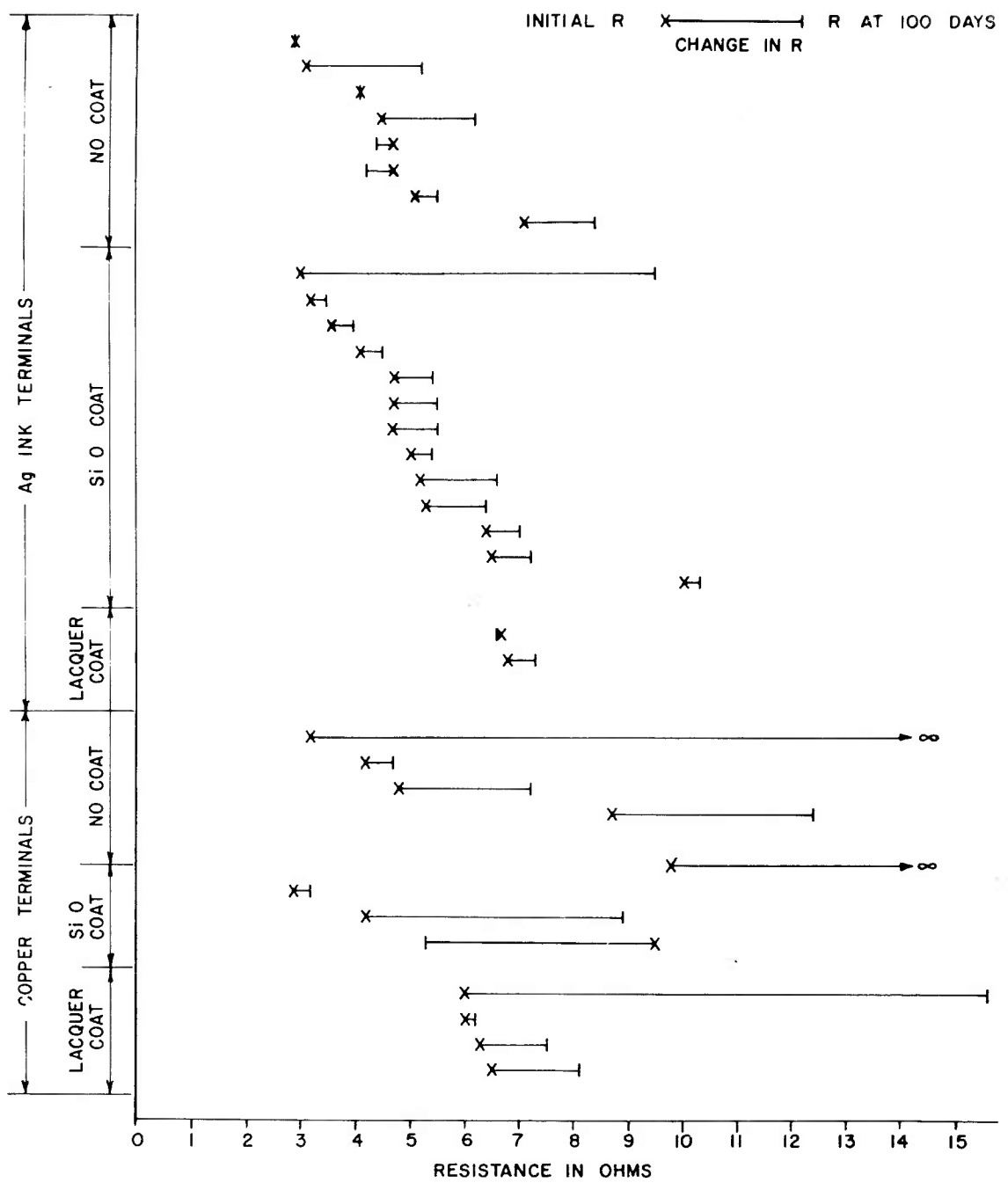


Figure V B-1 Resistance Change of Thermocouples in 100 Days

and one SiO coated thermocouple had a change greater than 2 ohms. The lacquer coated ones exhibited less change. Two ohm resistance changes occurred in all three kinds of thermocouples with copper terminals. Four of the thermocouples had resistance decreases for the complete test period and several others had decreases from one test period to the next. These decreases occurred at different times with no set pattern.

CONCLUSIONS: Bi-Sb Mk 1 Squib thermocouples with silver ink terminals are stable when they are at ambient room conditions for a period of 100 days. Only two out of twenty-three had a resistance change greater than two ohms. The thermocouples with copper deposited terminals were not as stable. In the group of twelve, there were two failures and five of the remaining ones had resistance changes greater than 2 ohms. The two coating materials, lacquer and SiO, had no measurable effect upon the resistance changes in the thermocouples. The result of this aging test indicates that the failures of Bi-Sb thermocouples with silver ink terminals in instrumented EED's were not caused by the aging of the materials in the thermocouples.

C. Humidity Test

The humidity test was based on Method 103A, Mil-Std-202A.

PURPOSE: This test was performed to evaluate the characteristics of Denver Research Institute's thermocouples as they are influenced by the absorption and diffusion of water and water vapor to determine if humidity caused failures. This was an accelerated environmental test, accomplished by the continuous exposure of the thermocouples to high relative humidity at an elevated temperature. These conditions impose a vapor pressure on the thermocouples under test which constitutes the force behind the moisture migration and penetration. Absorption of moisture by many materials causes loss of physical strength and changes in other important mechanical and electrical properties.

Since the aging test showed that the Bi-Sb Mk 1 squib thermocouples with silver ink terminals were stable at room ambient conditions for 100 days, this test was to determine if humidity could have caused the failures that occurred in instrumented EED's. Lacquer coated, SiO coated and uncoated thermocouples were tested to evaluate the ability of these materials to protect the thermocouples against high humidity.

PROCEDURE: Twenty-one Mk 1 squib Bi-Sb thermocouples with silver ink terminals were deposited the day the test was started. There were three types of thermocouples:

- (1) Uncoated
- (2) SiO coated
- (3) Lacquer coated

Two measurements were made each time the thermocouples were tested. The thermocouple resistance was measured on the General Radio Impedance Bridge. The output voltage of the thermocouple, when it was placed in the DRI reference heat source, was measured by a Hewlett-Packard 425A microvoltmeter.

Test 1 was made within two hours after the thermocouples were deposited. Due to a failure in the monitoring circuit of the heat source, the output voltages were not obtained in this test. The thermocouples were conditioned in a dry oven at a temperature of 110°F for 24 hours. At the end of this period they were removed from the oven and allowed to cool to room temperature. Test 2 was made at this time. Then the thermocouples were placed in the humidity chamber and subjected to a relative humidity of 80 to 100 percent and a temperature of 99° to 104°F for 96 hours. The chamber was arranged in such a manner as to avoid condensation dripping on the thermocouples under test. Upon completion of the exposure period Test 3 was made. Following the measurements at high humidity the thermocouples were conditioned at room ambient conditions for a period of 16 hours after which Test 4 was performed.

RESULTS: The results of the Humidity Test appear in Table V C-1. There were no failures in the 21 thermocouples tested. The largest change in resistance occurred during the initial twenty-four hour drying period. The average change for this period was a decrease of 1.6 ohms. The average change in resistance for the 96 hour high humidity period was an increase of 0.4 ohms. After the thermocouples were conditioned at room ambient conditions a slight decrease in resistance was measured. The average decrease between Test 3 and Test 4 was 0.2 ohms. No thermocouple had a resistance change greater than 2 ohms after the drying period.

TABLE V C-1

Humidity Test on Bi-Sb Mk 1 Squib Thermocouples

Type	Test 1	Test 2		Test 3		Test 4	
	R ₁	R ₂	E ₂	R ₃	E ₃	R ₄	E ₄
Not Coated	6.4 Ω	5.4 Ω	205μ _v	5.9 Ω	240μ _v	5.4 Ω	235μ _v
	6.7	4.9	155	5.5	165	5.1	170
	7.0	6.5	165	6.4	220	6.9	180
	6.2	4.7	220	5.1	255	4.8	245
	9.8	8.9	205	9.4	280	9.4	240
	9.8	6.3	155	6.1	210	6.9	190
	6.7	4.7	235	5.5	265	5.6	260
	Average	7.5	5.9	190	6.3	235	6.3
SiO coated	11.0	10.3	215	9.9	275	10.1	225
	12.0	9.6	230	10.2	265	9.8	265
	10.2	8.6	240	9.1	320	9.1	280
	10.9	8.0	190	8.0	245	7.6	220
	9.9	7.9	235	8.4	275	7.8	265
	12.0	10.0	160	10.3	220	10.4	180
	9.2	6.9	195	7.7	200	7.2	200
	Average	10.7	8.8	210	9.1	255	8.9
Lacquer coated	9.2	8.1	230	8.0	280	7.4	270
	8.7	7.9	170	9.2	250	9.6	230
	6.9	5.7	260	5.8	350	5.3	260
	8.6	7.2	205	7.6	270	6.9	240
	9.4	6.7	305	7.5	400	6.8	380
	8.2	7.2	230	7.5	325	7.9	300
	7.4	6.6	250	7.0	305	6.5	300
	Average	8.3	7.1	235	7.5	310	7.2

The measured output voltages showed larger variations than the resistance. For each thermocouple tested the output voltage increased in Test 3 and decreased slightly in Test 4. There are two possible reasons for these changes: the heat source and the thermocouple. A reliability test on the heat source was made by measuring the output voltages of a group of thermocouples several times during one day. Ten percent variations in the average output voltages were present. Therefore, a 10% change in the outputs could be caused by the heat source. This means the decrease in the outputs between Test 3 and Test 4 could have been caused by the heat source. The larger increase that occurred between Test 2 and 3 must have been produced by some effect of the humidity exposure period. The output voltages of dry and damp thermocouples were compared in another test and the outputs did not change. The fact that the thermocouples were damp in Test 3 could not have caused the higher outputs. A plywood holder was in the test chamber and the bases of all the thermocouples were discolored by the holder. It is possible that the thermocouple junction was also discolored and the increased output was caused by better absorption of the short wavelength portion of radiation from the heat source. The outputs did not return to their initial values after the test was completed indicating the change was permanent.

A comparison of the effects of the coating materials is shown in Table V C-2. The average change in resistance between Test 1 and Test 2 is represented by ΔR_{12} and the changes in outputs are represented in the same manner. The resistance changes and the output changes were about the same for the three types. The output change, ΔE_{23} , was slightly higher for lacquer coated thermocouples. Each time the outputs were measured, the lacquer coated thermocouples had the highest outputs followed by SiO coated and uncoated ones in that order.

TABLE V C-2

Comparison of Average Changes in Three Types
of Thermocouples During the Humidity Test

Type	ΔR_{12}	ΔR_{23}	ΔR_{34}	ΔE_{23}	ΔE_{34}
Not coated	-1.6 Ω	+ .4 Ω	0 Ω	+25%	-10%
SiO coated	-1.9 Ω	+ .3 Ω	- .2 Ω	+20%	-10%
Lacquer coated	-1.2 Ω	+ .4 Ω	- .3 Ω	+30%	-10%

CONCLUSIONS: The exposure of thermocouples to high relative humidity at an elevated temperature for 96 hours caused no deterioration in the thermocouple properties. The only noticeable resistance change was the decrease that occurred during the initial drying period. Resistance decreases also had occurred at various times during the aging tests with copper terminal and silver ink terminal thermocouples. The reason for this is unknown. The increase in the outputs after the humidity period cannot be explained satisfactorily because of the number of variables. However, it is believed to be caused by the variations in the heat source and the discoloration of the thermocouple by the plywood holder.

The coating materials had no significant effect on the thermocouples under test. Since the uncoated thermocouple does not appear to deteriorate with age or under high relative humidity, the coating materials seem unnecessary. However, they produced higher outputs, a characteristic that is reported in Section V E.

The results of the aging and humidity tests indicated that the failures of Bi-Sb thermocouples with silver ink terminals in instrumented EED's were not caused by aging or humidity. It is possible that the thermocouple properties are affected by the glue or its solvents which are used to hold the instrumented EED's together. Testing was continued to determine if this is the main reason for failures.

D. Effects of Organic Solvents

Aging and humidity tests have indicated that past thermocouple failures of instrumented electro-explosive devices cannot be attributed to aging or humidity. In looking for a reason for failures, one must consider what part of the thermocouple's environment is likely to cause trouble. The aging and humidity tests at DRI used thermocouples which were not attached to squibs, whereas thermocouple failures mainly occur in instrumented devices. The tests did not include the procedure of fastening a thermocouple to a squib. Thermocouple base plugs are glued to the squib base in the proper orientation, and, because of the small geometry, liquid glue is in close proximity to the thermocouple. In addition the thermocouple and squib make a small, completely enclosed cavity in which vapors from the glue remain for long periods of time. Therefore, tests pertaining to the reactions of organic solvents which may be contained in glues and potting compounds were designed.

The test procedure was to measure thermocouple resistance and output initially. Then thermocouples were suspended one-fourth inch above the surfaces of the solvents at room temperature. Four hours later the thermocouples were removed and final resistances and outputs were measured. A control group, which was isolated from the solvents, was included to indicate the magnitude random variation.

The test was conducted with bismuth-antimony thermocouples on one-fourth mil Mylar substrates and a polyester plastic base plug. All thermocouples had silver ink terminals. There were three groups: (1) no coating, (2) silicon monoxide coating, and (3) lacquer coating. Figures V D-1 and V D -2 show the results of resistance and output change for group (1). Figures V D-3 and V D-4 show the results of resistance changes for groups (2) and (3).

Acetone and methyl ethyl ketone cause most damage to the thermocouples. This is not surprising, for acetone dissolves silver ink and it loosens evaporated films from substrates. In fact, it is used for cleaning the inside of vacuum systems. Methyl ethyl ketone dissolves the polyester base of the thermocouple. Figure V D-1 shows that all solvent vapors caused outputs to be reduced, and most caused a decrease greater than the random variation of the control group. Figures V D-2, V D-3 and V D-4 show that solvent vapors almost always cause an increase in resistance. Alcohol vapor has the least effect on thermocouple resistance. Coatings seem not to have significant protecting effect, most likely because of imperfect covering of vital parts. Lacquer coating is not so effective as SiO₂; however, minute holes in thin films of SiO₂ can also permit attack.

Results show that organic chemical solvent vapors can cause large changes in thermocouple characteristics and failure. It is quite probable that glues were the cause of thermocouple failures in the past. It is necessary to find a suitable glue which must have lasting strength and a short curing time as well as non-harmful solvents. The following glues have been used and found to be inadequate:

- | | | |
|----------------------|-----------|--------------------|
| (1) Elmer's Glue-All | - - - - - | Poor Strength |
| (2) Duco Cement | - - - - - | Harmful Vapor |
| (3) Pliobond | - - - - - | Rubbery, Non-rigid |
| (4) Polystyrene Dope | - - - - - | Slow Drying |
| (5) Shellac | - - - - - | Very Slow Drying |
| (6) Glyptal | - - - - - | Slow Drying |

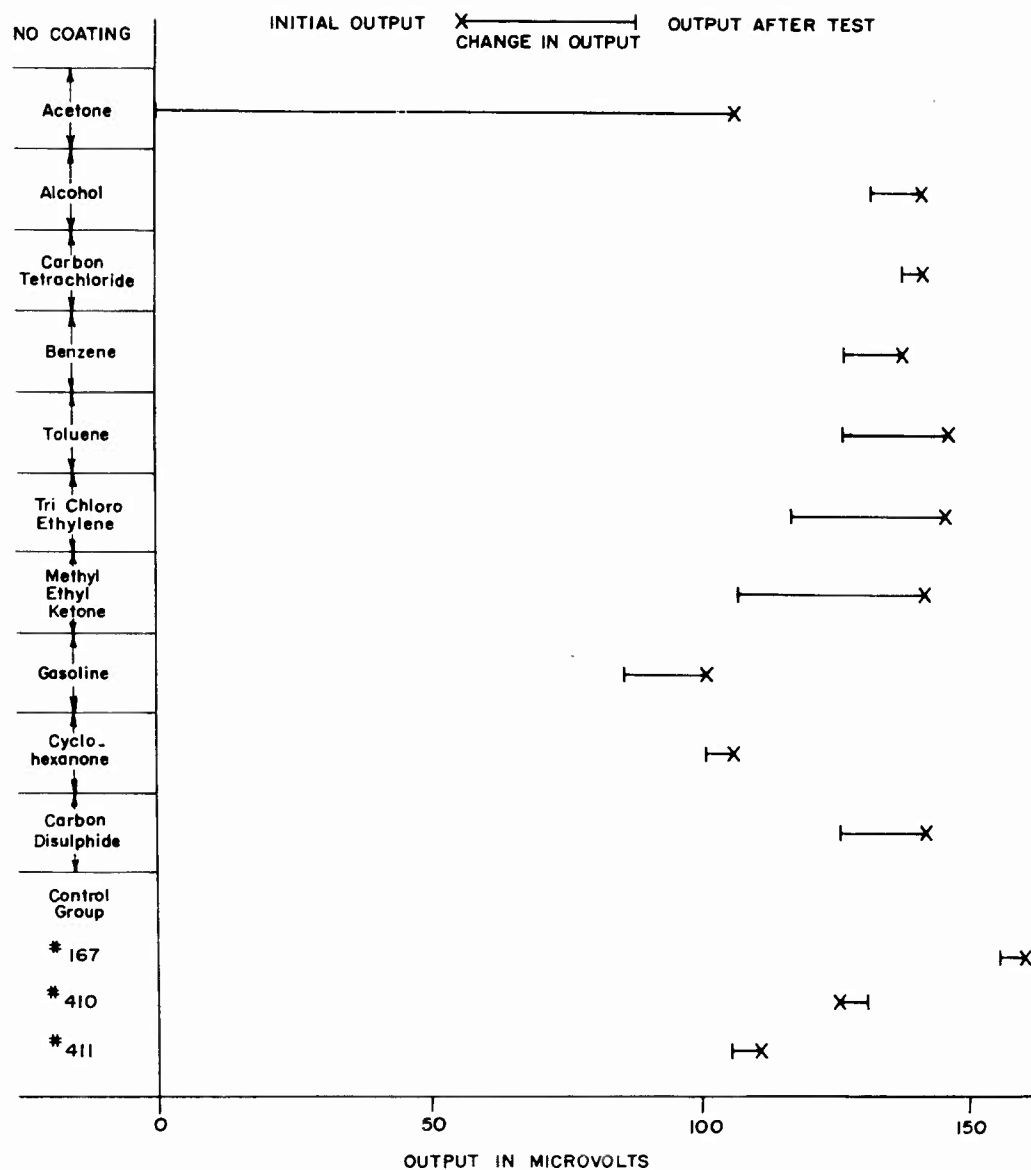


Figure V D-1 Output Change of Uncoated Thermocouples Caused by Exposure to Organic Chemical Vapors

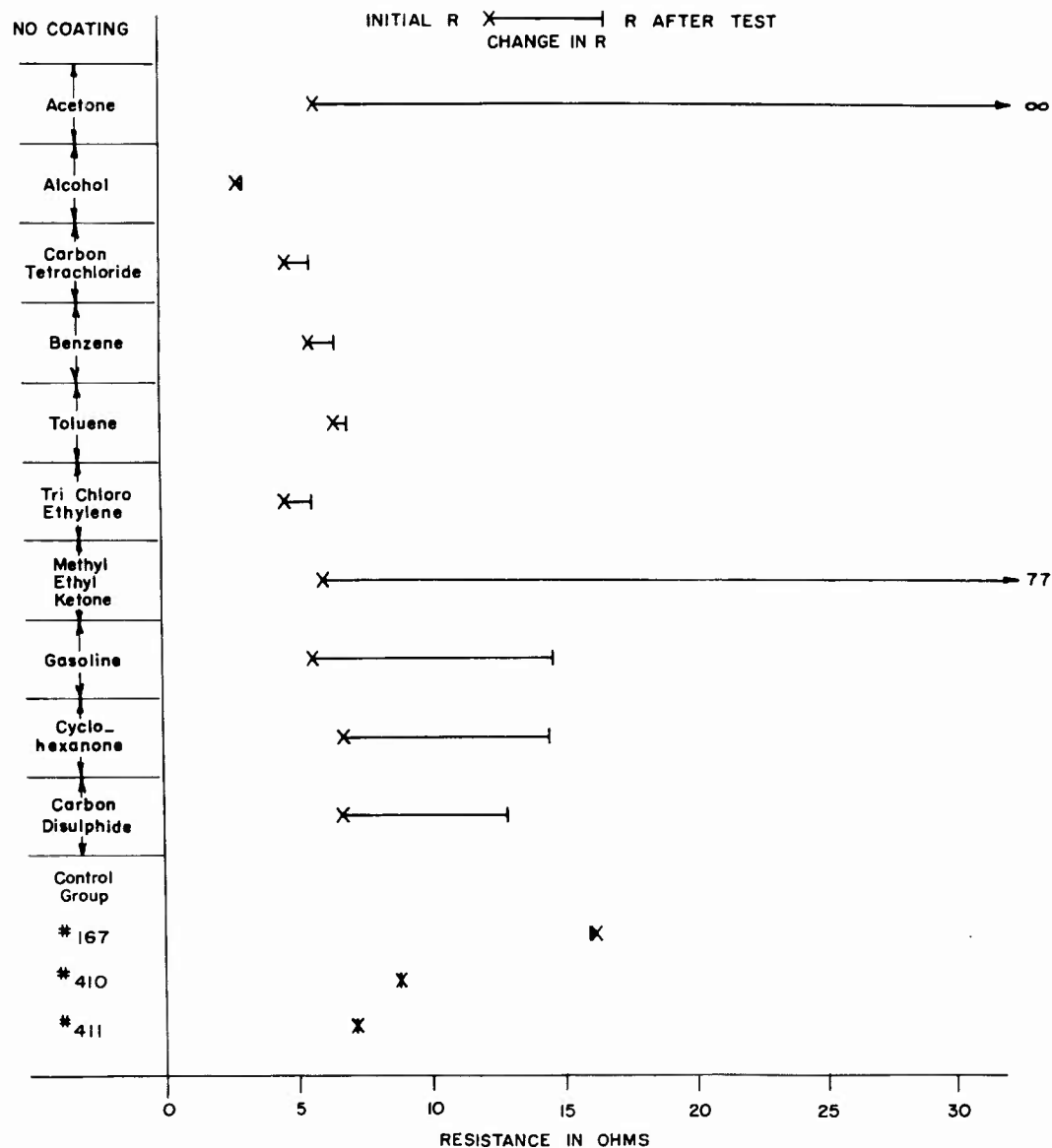


Figure V D-2 Resistance Change of Uncoated Thermocouples Caused by Exposure to Organic Chemical Vapors

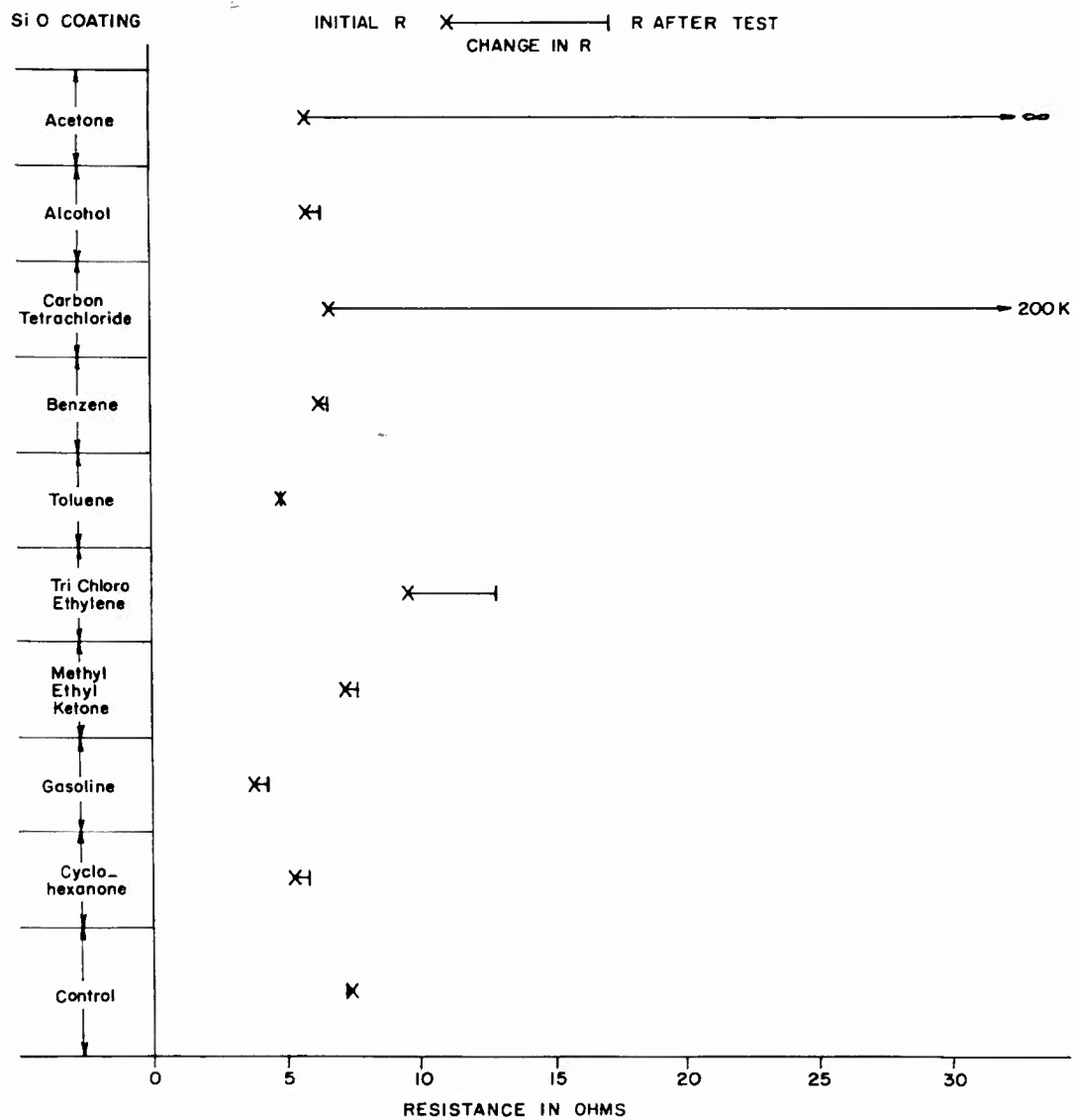


Figure V D-3 Resistance Change of SiO Coated Thermocouples
Caused by Exposure to Organic Chemical Vapors

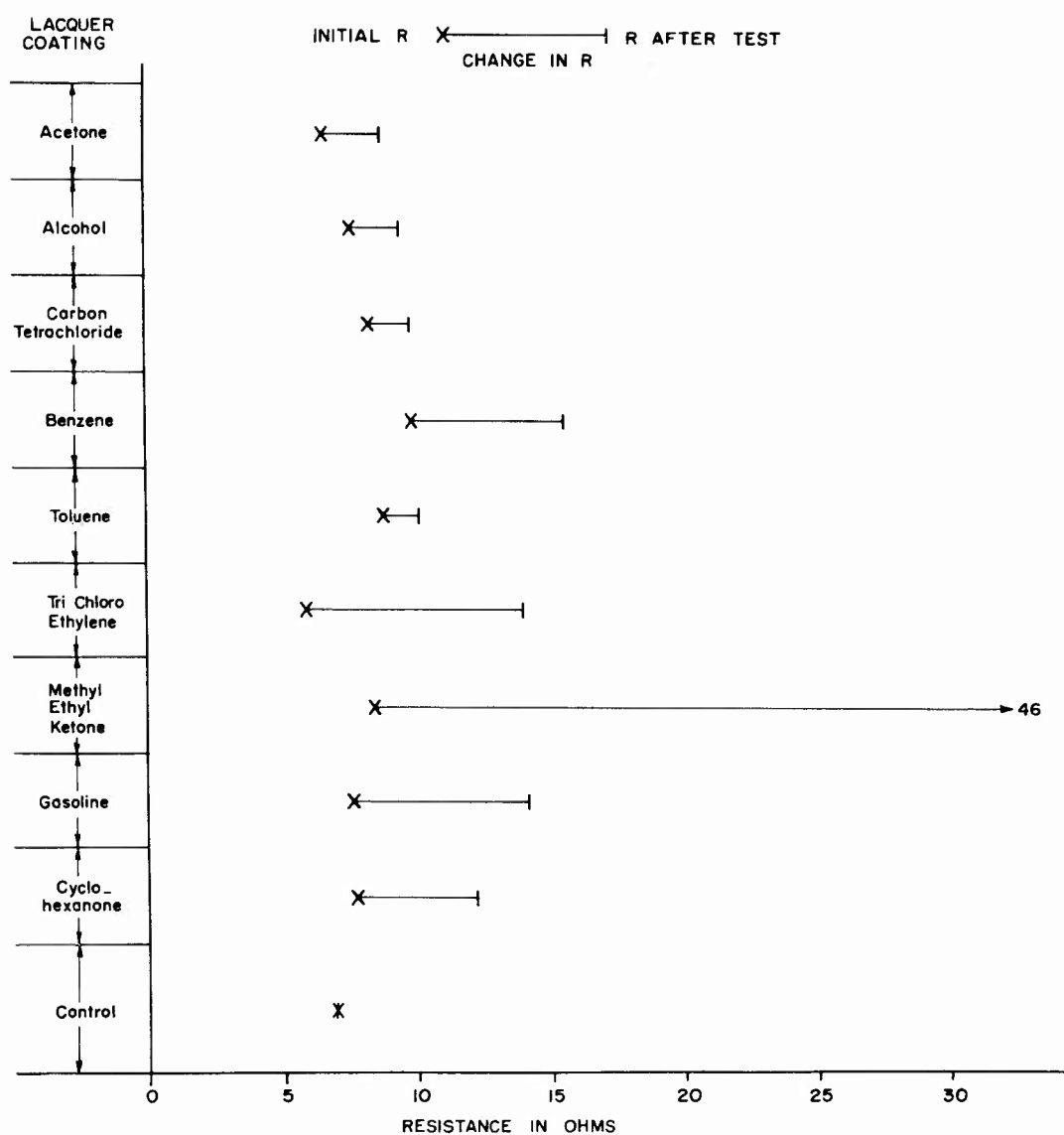


Figure V D-4 Resistance Change of Lacquer Coated Thermocouples Caused by Exposure to Organic Chemical Vapors

Slow drying glues take 6-8 hours before removal from the jig. Presently Eastman 910 Adhesive by Eastman Chemical Products, Inc., Kingsport, Tennessee is used. It has the quickest drying time, less than one minute, and forms a rigid and strong bond. Care is taken to avoid contact with any part of the thermocouple or silver ink contacts during application, and no failures seem to result.

E. Effects of Coating Materials and Thermocouple Constituents

The outputs of three sample groups of Mk 1 Squib Bi-Sb thermocouples were measured to determine the effects of SiO and lacquer coats. The results appear in Table V E-1. The SiO group had an average output which was approximately the same as the uncoated group but the average output of the lacquer coated ones was higher.

To check how much the lacquer improved the outputs two Bi-Te and two Alumel-Te thermocouple outputs were measured before and after they were coated. The average output of each pair was plotted as a function of the heat source monitoring thermocouple voltage in Figure V E-1. In each case the lacquer coat appears to increase the absorption of the radiated energy from the heat source. This effect is more noticeable at higher heat source temperatures where a larger part of the radiated energy is in the shorter visible wavelengths.

The time constants of the three types were also measured to determine if the coating materials changed the other thermal properties of the thermocouples. Table V E-2 is a comparison of the time constants.

The SiO coated and uncoated thermocouples have approximately the same time constants. However, the lacquer increased the time constants by a factor of two. Although the lacquer improves the output, it has a greater adverse effect upon the time constant.

The SiO coat had no measurable effect upon any thermocouple characteristic in the tests. The lacquer coat has the disadvantage of increasing the time constants. Neither of these two coating materials has improved the thermocouples. However, it may be possible to increase the thermocouple outputs by coating with a material that will increase the absorption of infrared energy and not increase the time constant.

TABLE V E-1

Output Voltages of Mk I Squib Thermocouples with
Different Coating Materials

Not Coated		SiO Coated		Lacquer Coated	
Microvolts		Microvolts		Microvolts	
	155		185		235
	175		175		240
	155		185		205
	170		215		250
	200		215		235
	165		235		285
	185		185		230
	215		225		310
	230		175		260
	200		175		280
	225				
Average	190	Average	195	Average	255

TABLE V E-2

Comparison of Thermocouple Time Constants

Not Coated		SiO Coated		Lacquer Coated	
	85 ms.		80 ms.		150 ms.
	100		90		200
	80		100		110
	75		80		400
Average	85 ms.	Average	90 ms.	Average	215 ms.

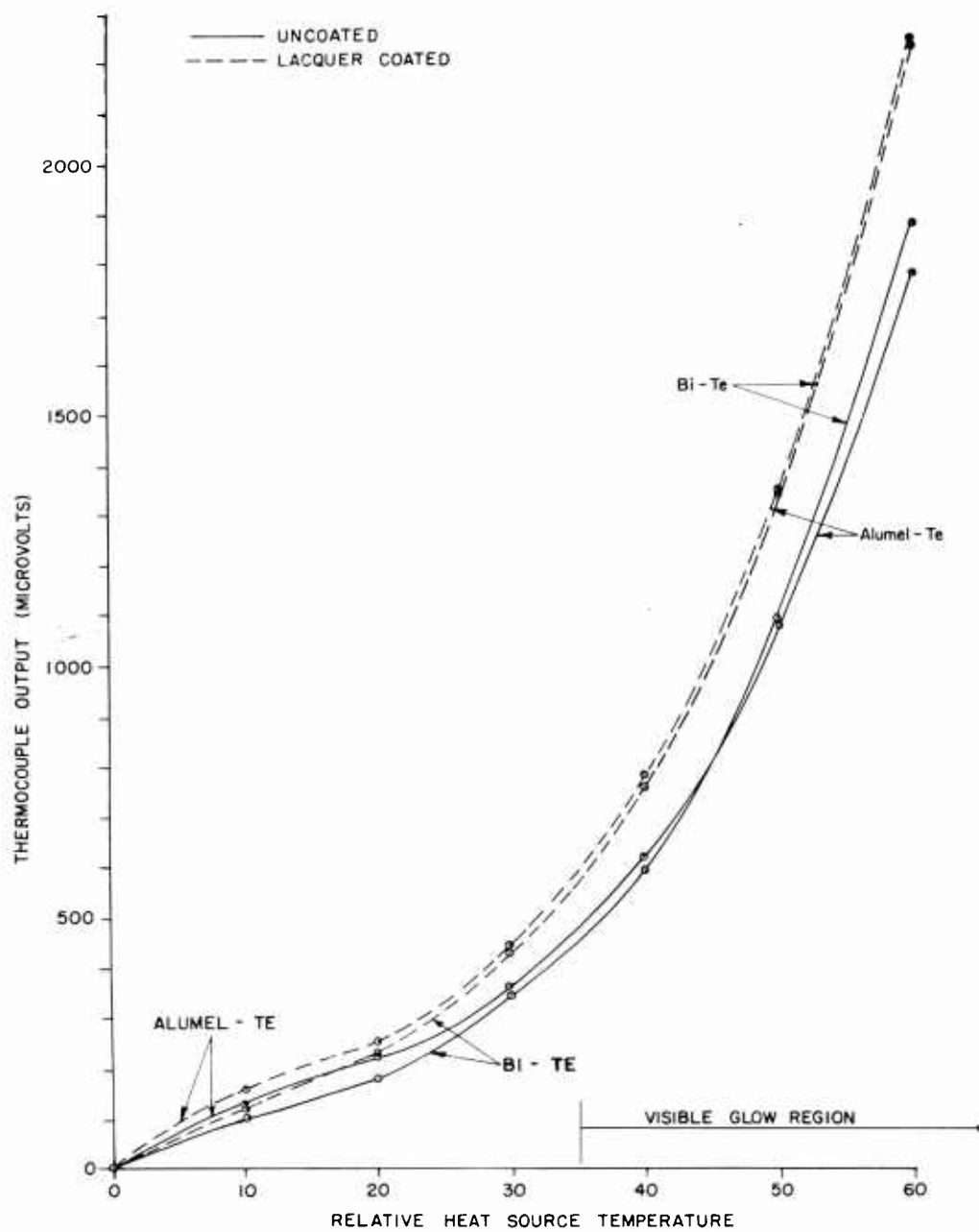


Figure V E-1 Effects of Lacquer Coating on Thermocouple Outputs

The sensitivity, resistance and time constant of three thermocouple material combinations were measured. The three types of Mk 1 squib thermocouples were:

- (1) Bi-Sb
- (2) Bi-Te
- (3) Alumel-Te

The measurements appear in Table V E-3. The initial resistance and the resistance after 20 days were measured. The change during the 20 day period was negligible for the group of Bi-Sb thermocouples but the Bi-Te group increased 50% in resistance and the Alumel-Te group increased 20%. The Alumel-Te group had the highest average output when tested in the heat source and less than half of the resistance of the Bi-Te group. A comparison of the outputs of the four thermocouples plotted in Figure V E-1 shows that the sensitivities are about equal when the heat source temperature approaches the visible glow region. The time constants of the three groups were approximately the same. This was expected since it is the Mylar and the thermocouple configuration that determine the time constants.

The average outputs and resistances in Table V E-3 indicate that Alumel-Te is the most sensitive thermocouple if the load resistance is greater than 150 ohms. If the load resistance is lower, the Bi-Sb thermocouple has the highest sensitivity.

F. Thermocouple Sensitivity Measurements Using an Instrumented Mk 1 Squib

A significant problem in developing a detector to indicate bridge wire temperature rise above ambient in an electro-explosive device is to determine sensitivity. This requires measurements which will lead to data relating detector output and bridge wire temperature rise above ambient. It is necessary to measure bridge wire temperature rise at very low excitation levels without disturbing the thermal properties and with sufficient sensitivity to avoid extrapolation. It was decided to use the bridge wire as a resistance thermometer and measure its volt-ampere characteristic accurately enough to determine the non-linearity due to resistance increase which is caused by Joule heating. This requires five significant figure accuracy in determining voltage and current. The temperature coefficient of resistance can be determined by measuring resistance at the freezing and boiling points of water. Finally, the temperature rise corresponding to a particular current passing

TABLE V E-3

Characteristics of Three Mk 1 Squib Thermocouple Combinations

Type	Resistance		Output Voltage	Time Constant
	Initial	20 Days Old		
	ohms	ohms	microvolts	milliseconds
Bi-Sb	4.7	4.3	155	80
"	4.5	4.3	175	100
"	4.1	3.6	155	90
"	4.7	4.1	170	100
"	3.0	5.1	185	90
"	6.5	7.5	175	100
"	3.2	3.3	185	80
Average	4.4	4.6	170	90
Bi-Te	1120	1390	860	70
"	1400	2260	910	90
"	1010	1380	890	90
"	920	1420	790	90
"	1200	1900	860	85
"	1040	1740	880	100
"	990	1370	830	85
Average	1100	1640	860	85
Alumel-Te	540	610	980	90
"	620	850	920	80
"	450	650	880	70
"	525	650	1100	85
"	430	570	1010	80
"	365	380	990	75
"	430	540	1020	75
"	540	550	1150	80
Average	490	600	1010	80

through the bridge wire can be determined. When the correlation between temperature and current is established, it is possible to determine what sensitivity the detectors have.

Such a set of measurements were made for the Mk 1 squib. A Mk 1 squib with copper jacket was connected in series with a Leeds and Northrup one ohm standard resistance. The circuit as used is shown in Figure V F-1. The current from the lead storage battery is adjusted by means of a rheostat. Provision is made during the tests to shield the squib from air currents. The ambient temperature varied so the temperature of the standard resistor was recorded. Because of the drift in the current it was necessary to use two potentiometers and obtain a simultaneous balance across the standard resistor and the squib. These measurements will result in an average value of resistance and temperature of the bridge wire.

The current I is determined from the potential E_r across the standard resistor R :

$$I = E_r/R$$

The resistance, R_s of the squib is determined from the potential E_s across the squib from the relation:

$$R_s = E_s/I \text{ or } R_s = (E_s/E_r)R$$

To obtain a value for the temperature coefficient of resistance α , the resistance at each of three temperatures was used, melting ice, 0°C , ambient temperature, and boiling water, which is about 94°C in Denver, Colorado.

$$\alpha = (\Delta R)/(R_a \Delta T)$$

R_a is the resistance of the bridge wire at ambient temperature. The resistance at each of these temperatures was determined by means of the potentiometers. A five milliamperic current was used in the circuit. It had been found previously that a five milliamperic current caused no measurable change in the resistance of the bridge wire. The value obtained for the temperature coefficient of resistance was $\alpha = 383 \times 10^{-6} \text{ (deg. C)}^{-1}$.

Measurements of bridge wire resistance were made for values of bridge wire current ranging from 10 ma to 200 ma. The experiment was performed for a bare bridge wire and for a squib inert loaded with finely powdered plaster. Tables V F -1 and V F -2 show the values of

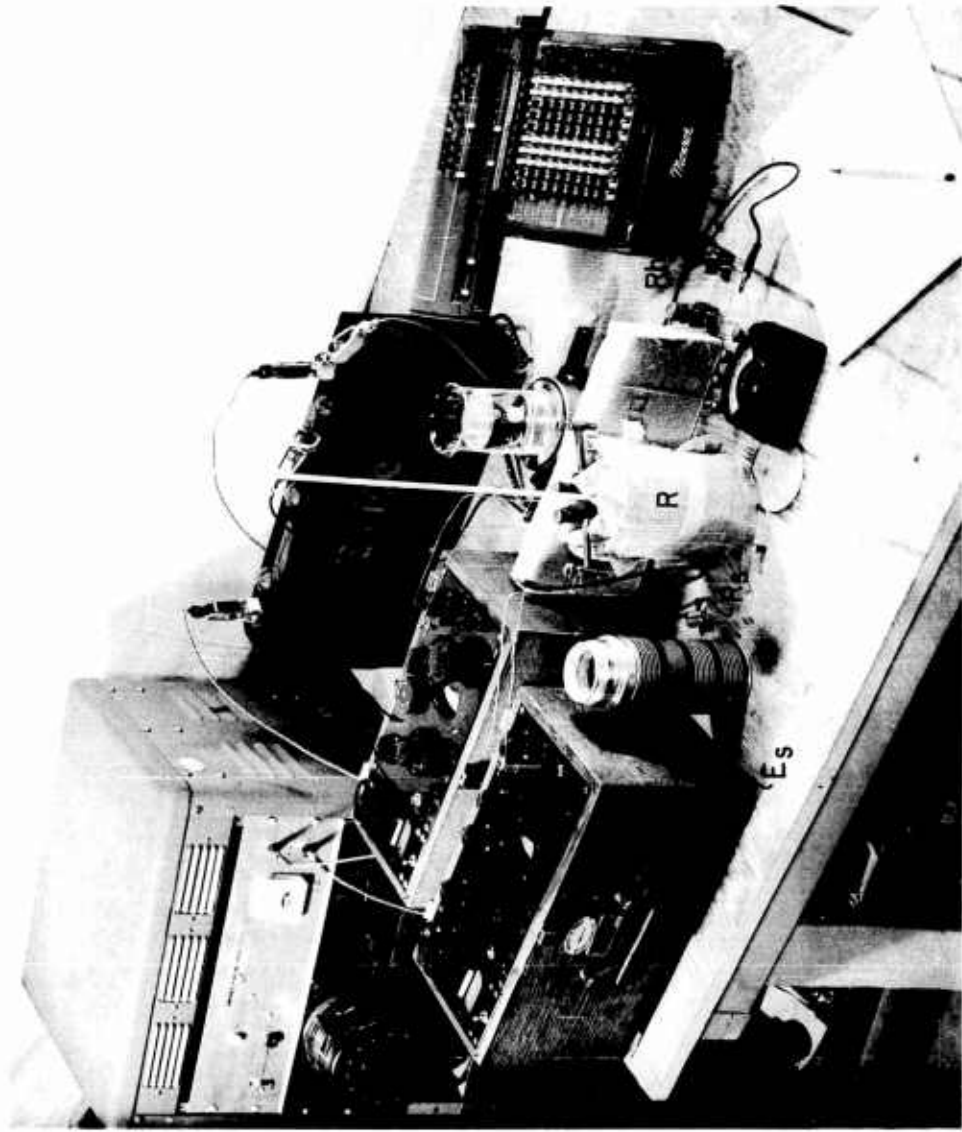


Figure V F-1 Experimental Circuit for Determining Resistance Variation of a Mk I Squib Bridge Wire with Bridge Wire Current

TABLE V F-1

Resistance and Temperature Variations of a Mk 1 Squib
Bare Bridge Wire with Current

Bridge Wire Current (ma)	Bridge Wire Resistance (ohms)	Bridge Wire Temperature Rise (°C above ambient)
5.000	1.0600	0
10.000	1.0601	0.11
15.000	1.0613	1.35
20.000	1.0620	2.25
25.000	1.0637	4.17
30.000	1.0653	5.97
40.000	1.0685	9.58
50.000	1.0732	14.88
60.000	1.0790	21.41
70.000	1.0860	29.30
79.030	1.0926	36.74
89.477	1.1012	46.43
99.432	1.1107	57.14
108.58	1.1202	67.85
119.61	1.1336	82.95
133.89	1.1514	103.01
146.42	1.1676	121.27
167.72	1.1932	150.12

TABLE V F-2

Resistance and Temperature Variations of an Inert Loaded
Mk 1 Squib Bridge Wire with Current

Bridge Wire Current (ma)	Bridge Wire Resistance (ohms)	Bridge Wire Temperature Rise (°C above ambient)
5.000	1.0540	0
10.000	1.0540	0
15.000	1.0540	0
20.000	1.0541	0.11
25.000	1.0546	0.68
30.000	1.0553	1.47
40.000	1.0565	2.83
50.000	1.0584	4.98
60.000	1.0608	7.70
70.000	1.0635	10.75
80.873	1.0665	14.15
90.441	1.0694	17.43
99.752	1.0731	21.62
109.04	1.0765	25.47
121.11	1.0833	33.17
132.85	1.0896	40.30
143.01	1.0953	46.75
152.71	1.0995	51.51
163.36	1.1055	58.41
172.75	1.1114	64.98
183.12	1.1194	74.03
196.68	1.1311	87.28

resistance obtained at each of the current settings. The values shown for the temperature rise above ambient were calculated by using the equation:

$$\Delta T = (\Delta R) / (\alpha R_a)$$

where R_a is the resistance value at ambient temperature and ΔR is the increase in resistance above the value at ambient temperature caused by Joule heating.

Figures V F-2 and V F-3 show the way in which the temperature of the bridge wire changes with bridge wire current. The spread in output of twenty Bi-Sb thermocouple detectors as a function of Mk 1 squib bridge wire temperature is displayed in Figure V F-4. Figure V F-5 shows the variation in thermistor response as a function of Mk 1 squib bridge wire temperature. This graph represents data from five G170 thermistors.

It is evident that an average thermocouple instrumented unloaded Mk 1 squib based on a sample of 20 and having an easily measurable output of 20 microvolts or a deflection of about 2mm on the type 102A40 galvanometers of the Midwest Recorder is capable of detecting a temperature rise of $2 \frac{1}{4}^{\circ}\text{C}$ caused by 20 ma flowing through the bridge wire. This is equivalent to less than 0.2°C bridge wire rise in an inert loaded Mk 1 squib. Since variations in ambient temperature are much larger than the measurable bridge wire temperature rises, it is evident that present thermocouple and thermistor sensitivities are adequate.

G. Time Constant Measurements

Thermocouple time constant measurements can be made quickly and accurately using the reference heat source described in Section V A. This system eliminates the EED's bridge wire time constant from the measurements and makes direct comparisons of time constants for different models of thermocouples possible. These comparisons could not be made when the bridge wire was used as the heat source and the thermocouple time constant was determined by measuring its rise time when a step function of current was applied to the bridge wire.

The thermal rise time of the thermocouple is measured by adjusting the final magnitude of the oscilloscope deflection to 3 cm and measuring the time required for a 2 cm deflection, since time constant is the time required for the thermocouple to reach 67% of its final value. Two thermocouple rise times are shown in Figure V G-1. The Mk 1

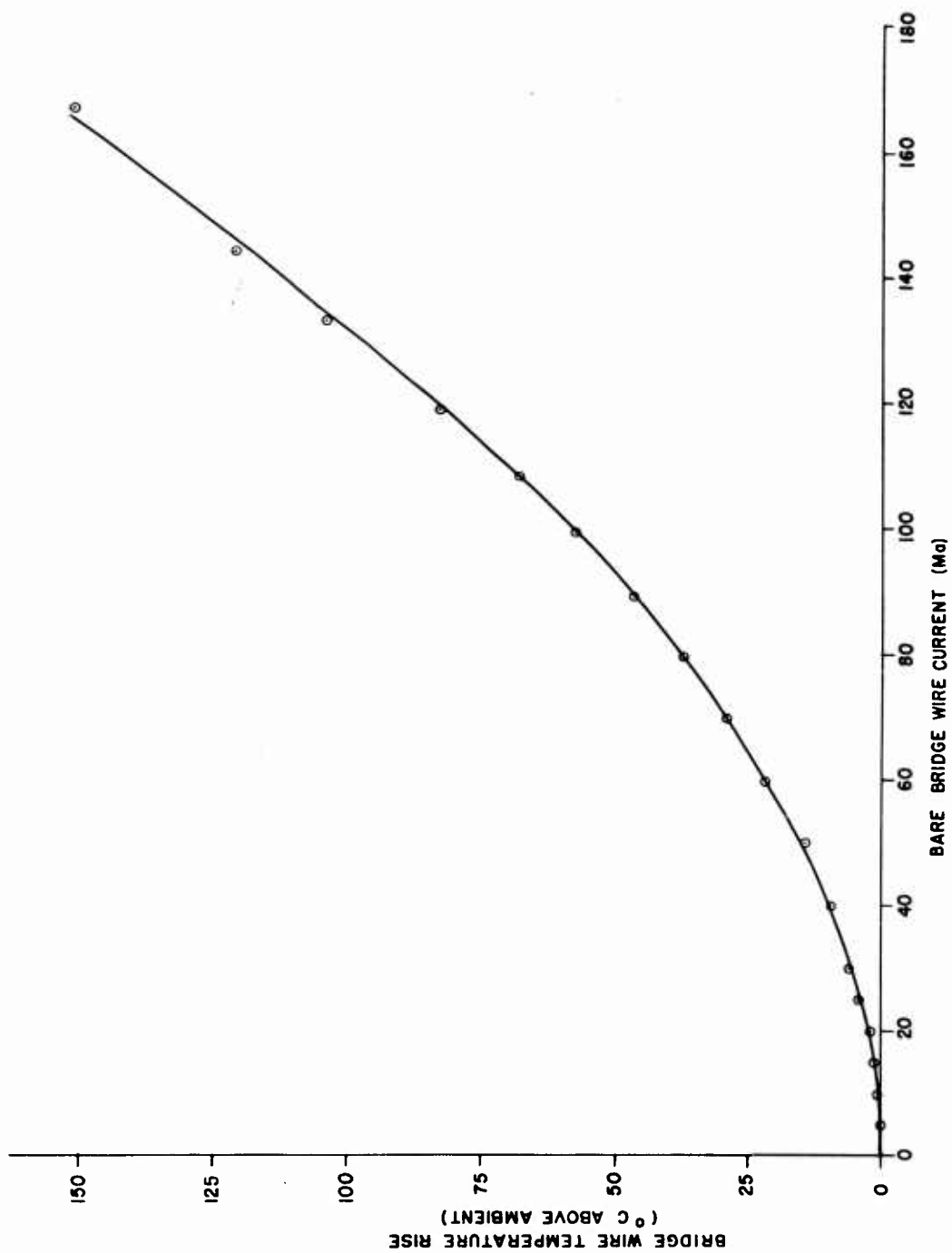


Figure V F-2 Bare Bridge Wire Temperature of a Mk 1 Squib as a Function of Current

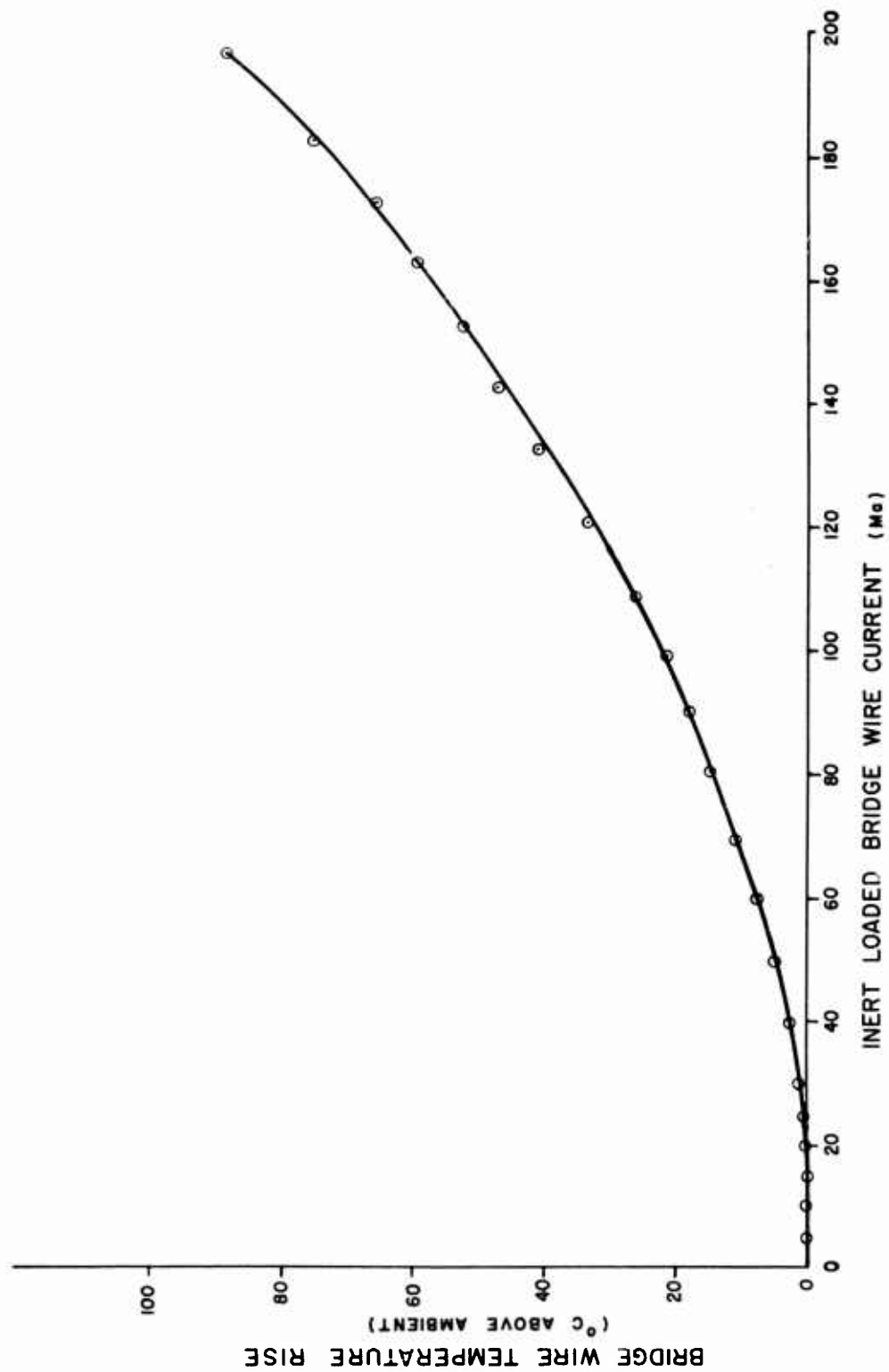


Figure V F-3 Inert Loaded Mk 1 Squib Bridge Wire Temperature as a Function of Current

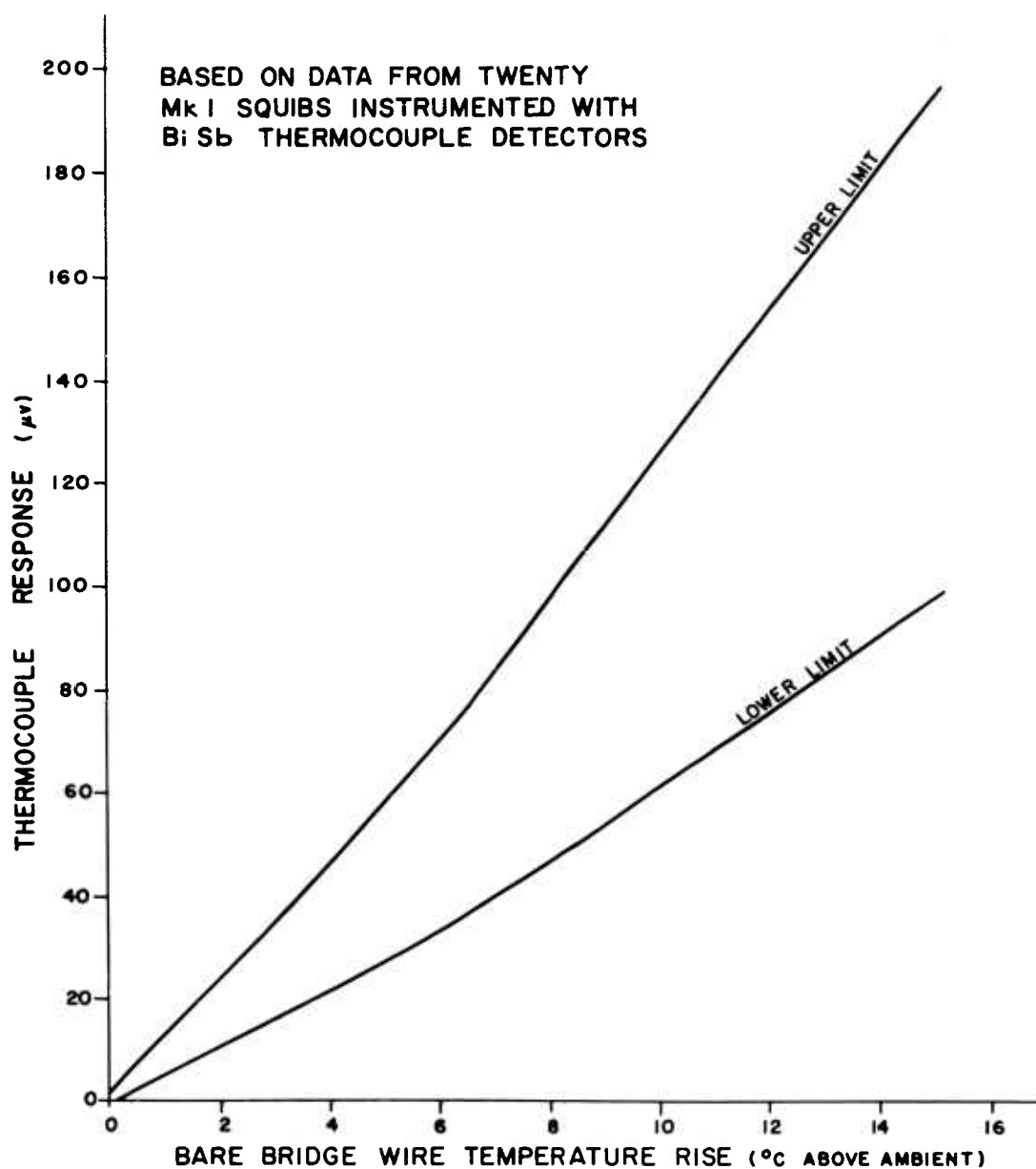


Figure V F-4 Thermocouple Detector Response as a Function of Mk I Squib Bridge Wire Temperature

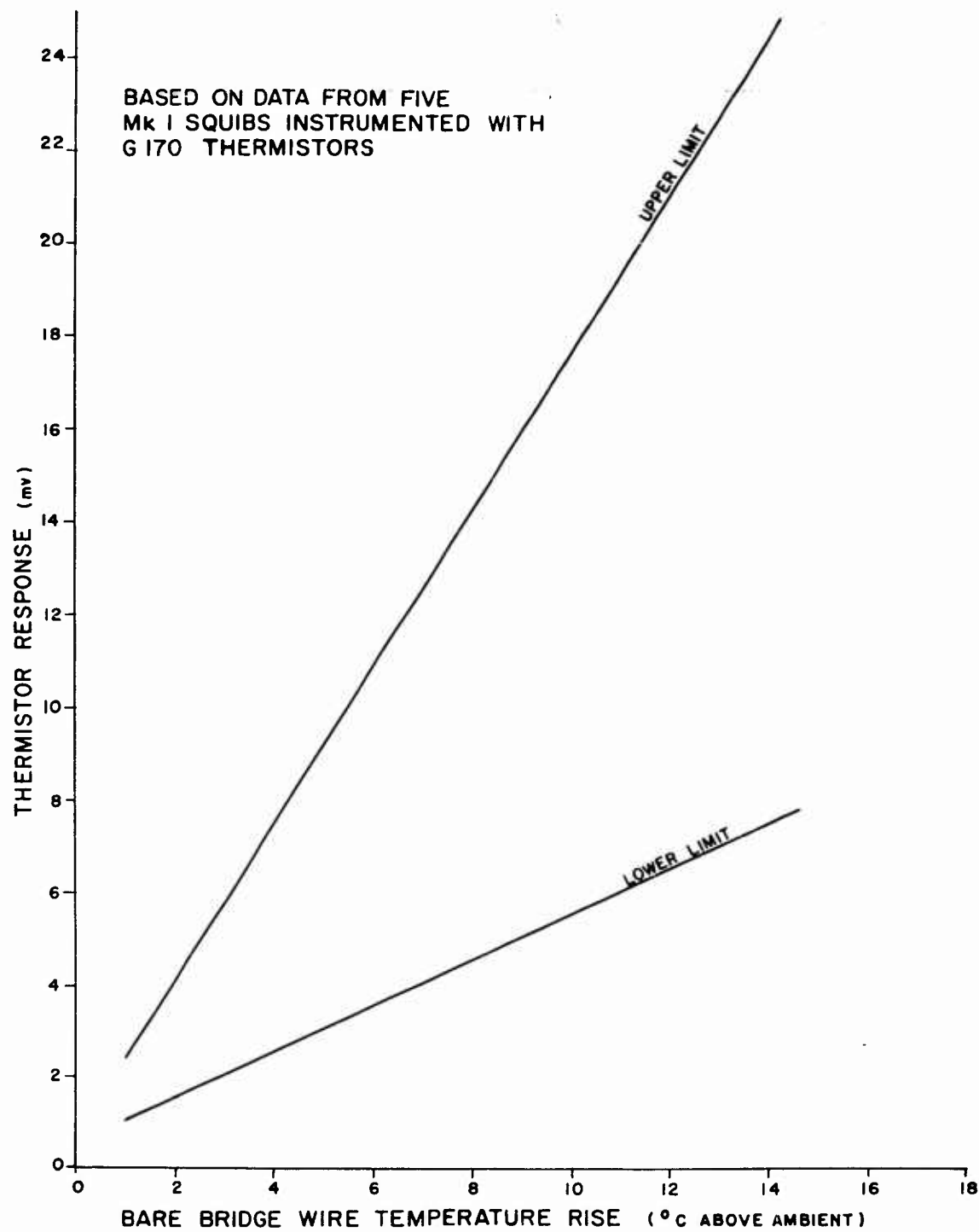
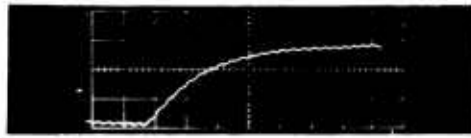
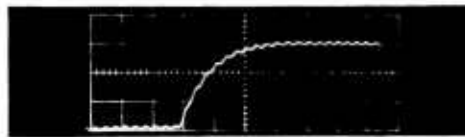


Figure V F-5 Thermistor Detector Response as a Function of Mk I Squib Bridge Wire Temperature



Mk 1 Thermocouple #357

Time 50 milliseconds per cm
Sensitivity 100 microvolts per cm



Mk 70 Thermocouple #207

Time 50 milliseconds per cm
Sensitivity 50 microvolts per cm

Figure V G-1 Thermocouple Response to a Step Function of Radiation

sqiub thermocouple has a time constant of 100 milliseconds and the Mk 70 thermocouple has a 50 millisecond time constant. These results can be reproduced within 10 percent.

The effects of different methods of reducing time constants can now be measured quickly. The reduction of the width of the Mylar substrate reduced the time constant of a thermocouple about 15 percent. An attempt will be made to reduce the thickness of the substrate in order to decrease the time constant and maintain the sensitivity. Other methods of decreasing time constants at the expense of sensitivity by increasing the thermal conductivity from the "hot junction" to the base will be considered. Time constant measurements have been made on thermocouples made of various materials and with coatings of SiO and lacquer. The results appear elsewhere in Section V.

A thin film thermocouple can be described by a first order differential equation, namely,

$$P_T = C_T \theta_T + \gamma_T \theta_T \quad (1)$$

where P_T = Power absorbed by thermocouple junction from infrared illumination (milliwatts)

C_T = Heat capacity of thermocouple junction (millijoules/°C)

γ_T = Heat loss factor (milliwatts/°C)

θ_T = Temperature rise of thermocouple junction above ambient (°C)

Equation (1) assumes that heat loss caused by reradiation and convection is much smaller than heat loss which is conducted away. This is valid for small θ . The time constant of the thermocouple is:

$$\tau_T = \frac{C_T}{\gamma_T} \quad (2)$$

This is the time required for θ to achieve $1 - \frac{1}{e}$ or 0.632 times the final value. For convenience this factor is usually rounded off to 2/3 when oscilloscope measurements are made. It has been found that τ_T had been about 100 ms for original Mk 1 type thermocouples, and it has been reduced to about 80 ms after minimizing the width of the Mylar substrate.

The time constant can be reduced by decreasing C_T or increasing γ_T . Decreasing C_T is by far the better method, for, as γ_T is increased, sensitivity is reduced proportionately. C_T depends on the combination of

the masses and specific heats of the junction materials and substrate. γ_T depends mostly on the combination of thermal conduction of junction materials and substrate. Because the junction materials are films normally having thicknesses of about 2000 Å with the Mylar substrate having a thickness of about 63,000 Å and because the substrate has a specific heat four to five times that of the junction materials, C_T depends mostly on substrate mass. The materials suitable for substrates have nearly equal values of specific heat. Therefore, effort exists to minimize substrate mass by using the thinnest possible films.

Choice of a thin film substrate depends largely on strength and ease of handling. The following materials have been tried: 0.17 mil polypropylene film, collodion which is a solution of pyroxylin in ether and alcohol, and Viton A which is a fluorocarbon elastomer made by DuPont. Characteristics of thermocouples made with these films are given in Table V G-1. Collodion makes the most successful thin film substrate. Polypropylene films cannot be made significantly thinner than Mylar and they are not so strong. There has been no success in making satisfactory films of Viton A, for holes spontaneously develop in the film after it is suspended.

At present vacuum deposited thermocouples on collodion substrates can be made consistently with time constant between 8 and 15 milliseconds. Figure V G-2 shows the rise time of a collodion substrate thermocouple. The time constant of the thermocouple is now nearly equal to the time constant of the Mk 1 squib bare bridge wire. An electronic scheme for making a collodion substrate, thermocouple instrumented Mk 1 squib having an effective time constant less than one millisecond is described in Volume III, Section V A. It now appears that thermocouple time response can be enhanced sufficiently to follow the most rapid changes in bridge wire temperature. Much work needs to be done in developing techniques to control substrate thickness and in selecting the best material for a thin film substrate. In addition testing is necessary before thermocouples can be used reliably in the field.

Because collodion films are thinner and not so strong as Mylar films, the low time constant thermocouple is expected to be more delicate than previous thermocouples; however, many low time constant thermocouples have exhibited surprising tenacity in normal handling in the laboratory. The thickness of the collodion film substrate is guessed to be about 7000 Å on the basis of time constant change.

TABLE V G-1

Comparison of Mk 1 Type Thermocouples
Having Different Substrates

Thermocouple Number	Junction Materials	Substrate	Output Micro- volts	Time Constant Milli- seconds
413	Bi-Sb	0.25 mil Mylar	195	80
414	Bi-Sb	0.25 mil Mylar	220	80
415	Bi-Sb	0.25 mil Mylar	215	75
416	Bi-Sb	0.25 mil Mylar	195	80
417	Bi-Sb	0.25 mil Mylar	130	50
418	Bi-Sb	0.25 mil Mylar	205	65
429	Bi-Sb	0.17 mil Polypro- pylene	175	50
430	Bi-Sb	Collodion	215	40
431	Bi-Sb	Collodion	240	160
432	Bi-Sb	Collodion	220	15
433	Bi-Sb	Collodion	225	10
462	Alumel-Te	Collodion	415	10
464	Alumel-Te	Collodion	460	10
500	Alumel-Te	Collodion	680	8



Figure V G-2

Thin Film Substrate Thermocouple Response
to a Step Function of Radiation

Mk 1 Thermocouple Alumel-Tellurium

Time 10 milliseconds per cm

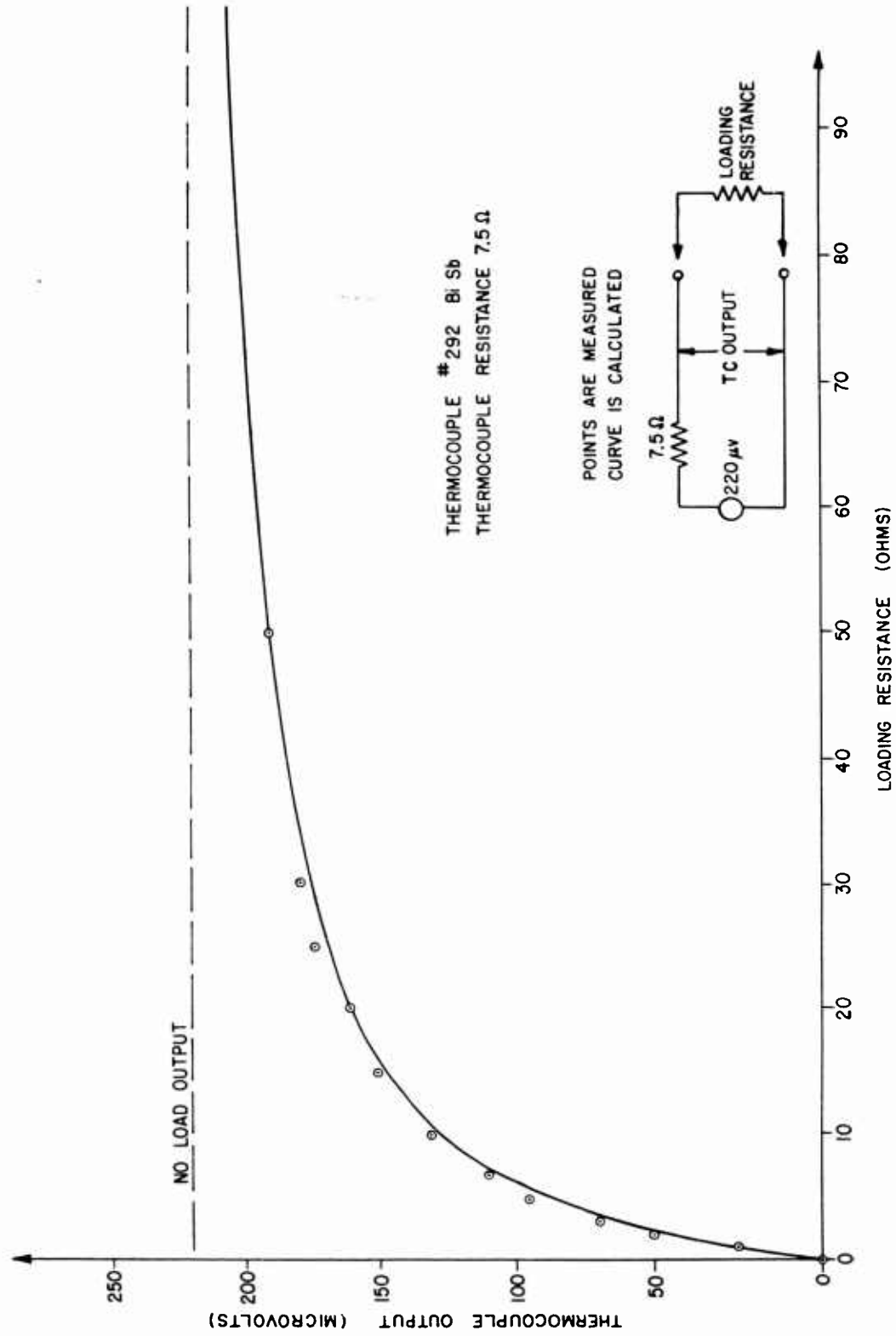
Sensitivity 200 microvolts per cm

Several Mk 1 type vacuum deposited thermocouples were made with thin Formvar film substrates. These films were prepared by a technique commonly used in electron microscopy to cast thin film replicas. The thermocouples have nearly the same electrical and thermal characteristics as do thermocouples made with thin collodion film substrates. No tests of mechanical strength or resistance to chemicals have yet been made. Collodion films are easier to prepare; however, Formvar films are expected to be stronger.

H. Output Loading

Bismuth-antimony thermocouples typically have a resistance of about six ohms as measured by an ohmmeter or bridge; and it is not uncommon for this resistance to increase by one or two ohms as the thermocouple ages. Since metering is frequently done with 30 ohm galvanometers, calibrations change according to thermocouple resistance. In order to determine whether the measured thermocouple resistance equals Thevenin's equivalent generator resistance, thermocouple output was measured for various load resistances. See Figure V H-1. Then, using the measured resistance and the no-load thermocouple output, a curve was calculated and compared with the experimental points. The results show that the measured resistance can be used as the generator resistance.

Figure V H-2 is a calculated curve showing how thermocouple output decreases as thermocouple resistance increases for a 30 ohm load typical of galvanometers and a 5000 ohm load typical of transistor amplifier input. For a typical thermocouple resistance of 6 ohms a 30 ohm galvanometer will measure 83% of the no-load output while a transistor amplifier will measure essentially 100%. If the thermocouple resistance changes from 6 ohms to 8 ohms, the 30 ohm galvanometer measures 79% of the no-load output while the transistor amplifier still measures 100%. Therefore, a thermocouple resistance change of 6 to 8 ohms causes a 4.8% decrease in calibration with a 30 ohm galvanometer, and a change from 6 to 10 ohms causes a 9.6% decrease in calibration. When the load is 5000 ohms, thermocouple resistance can change from 0 to 50 ohms without causing more than a 1% change in calibration. Figure V H-2 can also be used to compare thermocouple calibrations made with a vacuum tube voltmeter with those made with a 30 ohm galvanometer.



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Figure V H-1 Bi-Sb Thermocouple Loading Characteristic

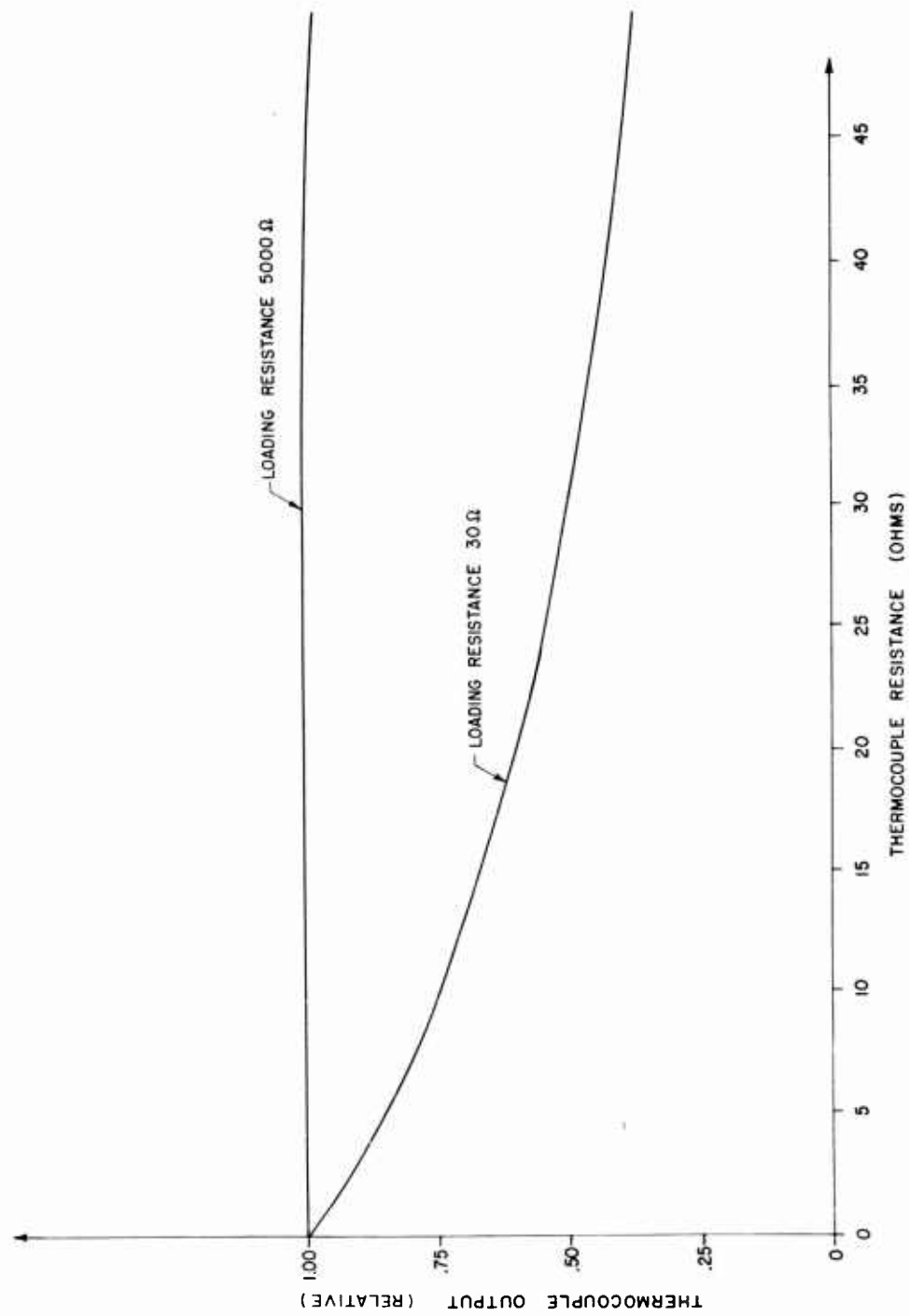


Figure V H-2 Dependence of Thermocouple Output on Thermocouple Resistance

Although generator resistance linearity has been verified for bismuth-antimony thermocouples, no check has yet been made for other types such as bismuth-tellurium. Tellurium, being a semiconductor, may cause thermocouples to have non-linear resistances which are due to contact and junction properties.

VI. SUMMARY

It has been demonstrated that rockets and missiles can be initiated inadvertently while being loaded onto their launchers when adjacent radio or radar transmitters are operating. Initiation is due to the heating of bridge wires of electro-explosive devices by induced rf currents. A necessary phase of investigation of the problem, which is called HERO (Hazards of Electromagnetic Radiation to Ordnance), is to provide instrumentation capable of indicating the degree of hazard under actual conditions. In particular, the instrumentation must be capable of measuring the temperature rise of a bridge wire which may be energized by induced currents at any frequency without perturbing the geometry of the rf excitation or the thermal characteristics of the bridge wire. In addition the sensor and associated instrumentation must be insensitive to rf. The sensor must be capable of indicating temperature rises which are smaller than normal variations in ambient and its output must be capable of driving a data recorder. The time constant of the sensor should be less than the time constant of the bridge wire. These specifications are complicated by the miniature size of the electro-explosive devices. Denver Research Institute of the University of Denver has investigated and developed several types of sensors which include radiation thermocouples, thermistors, photoconductors, etc. In addition some investigations concerning electro-explosive device characteristics and mechanism of rf power transfer into bridge wires was necessary to supplement sensor development.

Table VI-1 summarizes the present (1960) status of sensors considered by Denver Research Institute. Sensors are compared characteristic by characteristic in a general way based on accumulated data from laboratory testing, experience from field tests and information from technical literature. It is not yet possible to make a table having precise figures of merit based on actual electro-explosive device instrumentation.

It should be stated that sensors can be used for two distinct purposes, namely:

- (1) to indicate the joule heating in bridge wires of electro-explosive devices in a particular missile or rocket on a particular airplane or ship so that the actual degree of hazard to ordnance is determined. (field testing)

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TABLE VI-1
Summary of Sensor Characteristics (November, 1960)

Sensor	Quantity Detected	Sensitivity	Time Response	Perturbation of Bridge Wire Thermal Characteristics	Perturbation of Bridge Wire rf Characteristics	Frequency Selective
Radiation Thermocouple Vacuum Deposited	Temperature	Good	Medium	Negligible	Negligible	No
Radiation Thermocouple Wire	Temperature	Fair	Slow	Negligible	Negligible	No
Wire Thermocouple Attached to Bridge Wire	Temperature	Excellent	Follows Bridge Wire	Very Large	Large	No
Radiation Thermistor	Temperature	Good	Slow	Negligible	Negligible	No
Thermistor Attached to Bridge Wire	Temperature	Excellent	Follows Bridge Wire	Very Large	Large	No
Photoconductor (Uncooled)	Infrared Electromagnetic Radiation	Poor	Very Fast	Small to Medium*	Small	No
Photoconductor (Cooled)	Infrared Electromagnetic Radiation	Excellent	Fast	Small to Medium*	Small	No
Toroidal Coil	Electric Current	Excellent	Essentially Instantaneous	None	Small	Yes
Hall Effect Power Flow Probe	Electric Power Flow (Poyntings Vector)	-	Essentially Instantaneous	None	-	No
Electron Emission Detector	Temperature	-	-	Small	None	No
Photo electromagnetic Detector (PEM)	Infrared Electromagnetic Radiation	Very Poor	Very Fast	-	Small	No

* Caused by need for radiation chopper

TABLE VI-1 (Cont.)

Summary of Sensor Characteristics (November, 1960)

Sensor	Reliability	Ease of Fabrication	Relative Cost to Make Large Quantities of Sensors	Susceptibility of Instrumentation to rf	Adaptability to Automatic Recording	Other Complications
Radiation Thermocouple Vacuum Deposited	Good	Good	Low	None from dc to 1 kMc	Excellent	Require Low Level dc Amplifiers
Radiation Thermocouple Wire	Excellent	Good	Low	None from dc to 1 kMc	Excellent	
Wire Thermocouple Attached to Bridge Wire	Good	Poor	--	None from dc to 500 Mc	Excellent	
Radiation Thermistor	Excellent	Fair	Medium	None from dc to above 1 kMc	Fair	
Thermistor Attached to Bridge Wire	Excellent	Fair	Medium	None from dc to 500 Mc	Fair	Require Bridge Balancing
Photoconductor (Uncooled)	-	Poor	Very High	-	Fair	Requires Radiation Chopper
Photoconductor (Cooled)	-	Poor	Excessive	-	Fair	Requires Radiation Chopper Requires Cooling
Toroidal Coil	Excellent	Poor	Medium	Unuseable above 100 Mc	Poor	Requires Tuning
Hall Effect Power Flow Probes	-	-	-	None	-	Requires Low Level dc Amplifiers
Electron Emission Detector	-	-	-	None	Excellent	Requires Magnetic Field; Requires Constant Current Source
Photo electro-magnetic detector	-	Poor	Very High	-	Fair	

- (2) to indicate joule heating in bridge wires of electro-explosive devices in missiles or rockets in the laboratory so that methods of rf power transfer can be studied and minimized for future use. (laboratory testing)

Although optimum characteristics for sensors for both purposes are similar, significant differences exist. For example, sensors for field testing must be sensitive enough to detect bridge wire temperature rises which are small compared to ambient variation, whereas sensors for laboratory testing ideally should be capable of detecting infinitesimal amounts of power dissipated in the bridge wire in order to determine rf coupling. Sensors for field testing must be compatible with miniature portable equipment, whereas sensors for laboratory testing could involve large and complex equipment. Sensors for field testing are expendable and are required in large quantities, thereby implying a need for ease of fabrication and low cost, whereas sensors for laboratory testing are not expendable nor are large numbers required, thereby relaxing the need for ease of fabrication and low cost.

Effort at Denver Research Institute has been to develop sensors to be used for field testing and Table VI-1 indicates that, although vacuum deposited thermocouples are ranked lower than other sensors for certain characteristics, vacuum deposited thermocouples are the most practical sensors overall. Thermistors also make practical sensors with disadvantages of the need for bridge balancing and the apparent need for fabrication of instrumented EED's by hand. (See Volume II of this report.) Photoconductors are impractical because of their need for a high speed, micro-miniature radiation chopper which must be placed in close proximity to the bridge wire. (See Volume III of this report.) Detectors such as the Hall Effect power flow probe and the electron emission detector have not been developed sufficiently to make firm judgments. Other sensors such as the toroidal coil, PEM, wire thermocouple, and thermocouples and thermistors in thermal contact with the bridge wire are impractical for field testing. (See Volume III of this report.)

Evaporated thermocouple development can be divided into three phases, namely, fundamental studies, evaporation and fabrication, and testing evaluation. Effort in fundamental studies was essential to recent thermocouple improvement. Measurements of resistance versus thickness and thermoelectric power versus thickness of evaporated films of bismuth, antimony and tellurium have indicated that electrical resistance, not thermoelectric power sets the lower limit in film thickness

for thermocouples. Approximately 2000 Å for antimony, 3000 Å for bismuth, 8500 Å for tellurium and 700 Å for alumel are the points below which the resistance increases very rapidly. Thermoelectric powers do not change appreciably with thickness. Knowledge of the thermoelectric powers of evaporated films of metals and semiconductors has provided information leading to a thermal coupling coefficient for instrumented Mk 1 squibs. Measurement of thermoelectric powers of evaporated films of bismuth, antimony and tellurium as a function of age have shown slight decreases, which are not considered to be serious. Investigation of the effect of temperature on evaporated film resistances indicate that films on glass substrates are not damaged by exposure to air at 300°F. Experimentation with evaporated films of "alumel" has resulted in a thermocouple with an output higher than the thermoelectric powers would indicate. More investigation is necessary. A multiple beam interferometer which utilizes Fizeau fringes has been successfully constructed. This optical system can measure film thickness to ± 20 Å, thereby replacing the coarser method of weighing the gain in mass after evaporation.

Progress in evaporation and fabrication has resulted in an efficient deposition mask which enables simultaneous production of eight thermocouples of the same or different sizes without breaking vacuum. Also, the combination of various materials including alloys has brought about thermocouples having high outputs but increased resistance. A significant advance in casting thermocouple base plugs has reduced the necessary curing time from four hours to ten minutes. Use of collodion films in place of Mylar films as substrates for evaporated Mk 1 thermocouples has resulted in the lowering of the time constant from 90 ms to 10 ms. This approaches the time constant of the Mk 1 squib bare bridge wire. By electronic means, the low time constant thermocouples can be made to have an effective time constant of 500 microseconds, which equals that of a PbS photoconductor. The system has been demonstrated; however, considerable effort will be necessary before the system can be used in the field.

A reference heat source has been developed for the following reasons: one, to separate thermocouple characteristics from bridge wire source characteristics so that significant comparisons of thermocouples can be made; two, to provide a fast and convenient means of making a large quantity of thermocouple output measurements; and three, to provide a method of direct display of thermocouple time response so that time constants can be measured conveniently. In addition the heat source has been designed to be useful for photoconductor

measurements. Because infrared sensors behave quite differently at different wavelengths of radiation, it is necessary that the heat source radiation spectrum be in the range of interest. The heat source, which has demonstrated satisfactory performance, was used for most of the testing described in this report. The results of aging and humidity tests on silver ink connected Bi-Sb thermocouples definitely indicate that failure cannot be traced to 100 day aging or high humidity, in fact, only small variations in thermocouple resistance and output are caused by humidity, and, after 100 days, 90% of the thermocouples had changed less than two ohms. It has been shown that silver ink connections are more reliable than deposited copper connections, and the effect of protective coatings of SiO and lacquer was not noticeable in keeping resistance and output constant during aging and humidity tests. Tests of the effects of organic solvent vapors on evaporated thermocouples indicate that large changes in thermocouple characteristics and failure can occur. Solvents which must be avoided are listed and a glue is recommended for attaching the thermocouple to the EED. Variation in the calibrations of instrumented electro-explosive devices is aggravated by loading the thermocouple with 30 ohm galvanometers, since thermocouple resistance can change slightly over a period of time. This could be alleviated by using transistor amplifiers which drive a data recorder.

The sensitivity of evaporated thermocouples and thermistors used in instrumenting the Mk 1 squib has been measured. The average detector of a sample of 20 thermocouple and 5 thermistor instrumented Mk 1 squibs can readily measure a $2 \frac{1}{4}^{\circ}\text{C}$ rise of the bridge wire temperature of an inert loaded Mk 1 squib. Since changes in ambient temperature can be much larger than the minimum measureable bridge wire temperature rises, present thermocouple and thermistor sensitivities are adequate.

VII. BIBLIOGRAPHY

1. DRI Quarterly Report No. 39, "Test Equipment for and Evaluation of Electromagnetic Radiation," 1 January through 31 March 1960, Denver Research Institute, University of Denver, Section II.
2. Tolansky, S., Multiple Beam Interferometer, (Oxford, Clarendon Press), 1948.
3. *ibid.*, p. 147.
4. DRI Quarterly Report No. 33, "Test Equipment for and Evaluation of Electromagnetic Radiation," 1 October through 31 January 1960, Denver Research Institute, University of Denver, p. 53.

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