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# FINAL REPORT

# SOLID STATE CRYOGENIC TEMPERATURE SENSOR

by

W. H. Closser and R. R. Speers

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Advanced Systems Division
F. Ralph Schuricht

PHYLATRON CORPORATION
1183 Chesapeake Avenue
Columbus, Ohio 43212

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# SUMMARY

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The objective of the program is stated. The various phases of the device development phase are discussed in detail. These include: the selection of the starting material and its pertinent electrical properties; wafer preparation; diffusion techniques and studies including preparation; diffusion techniques and a theoretical analysis which led to the selection of an elemental Zn diffusion source; contacting; mesa formation, and bonding.

Consideration is given to the packaging problem. The ceramic package initially intended for use with these devices is discussed along with the experimental difficulties encountered in sealing the package. The alternative TO-18 and TO-46 packages are discussed, and the experimental results leading to the selection of the TO-46 package are presented.

A description of the calibration system used for device testing is presented. The description includes the instruments used and the limits of accuracy on the experimental system.

The various calibrations and tests including shock and reproducibility tests, calibration and calibration at various current levels, and life tests are discussed. The results of the various tests are presented.

The junction evaluation studies which include I-V characteristics and junction capacity measurements are discussed. The measurement techniques, plotted data, data analysis and interpretation are given.

Conclusions based on data and analyses presented earlier in the report indicate that a GaAs temperature sensor possessing many of the properties of an ideal resistance thermometer has been developed. Several conclusions are made relating to the type of junction required for a good temperature sensor, the mode of operation, the stability, the sensitivity, and the usefulness of the sensor.

Author

# INTRODUCTION

The accurate measurement and control of temperature in the cryogenic range can be an important requirement in many current engineering projects. The measurement of temperature, however, is not as easy to accomplish as is the measurement of many of the other physcial properties of a substance. Unlike properties such as length or volume, temperature cannot be measured directly and must be measured in terms of another property. Some of the physical properites that have been utilized in temperature measurement include: (1) pressure of gas; (2) equilibrium pressure of a liquid with its vapor; (3) electric resistance; (4) thermoelectric emf; (5) magnetic susceptability; (6) volume of a liquid; (7) length of a solid; and, etc. The most suitable property which could be used for temperature measurement would be one which is directly proprotional to temperature over its complete range of measurement. However, such a property can be found only in an ideal gas, and even then practical limitations exist.

The importance of choosing the proper thermometer, or method of temperature measurement most applicable to a specific situation, is often not obvious. In some circumstances, only one particular type of thermometer can be used, but in most cases, a choice of thermometers or methods for the specific application does exist. In situations where small size, rigidity and remote monitoring are among the criteria used for selecting a temperature sensor, either an "electric resistance" or a thermoelectric type sensor must be used unless additional transducers are incorporated in the system to provide the electrical signals necessary for remote monitoring and control of the temperature.

Neither the thermoelectric, or the resistance type temperature sensor is an ideal thermometer because each has several disadvantages which limit their versatility. However, in spite of these disadvantages, these thermometers are the most widely used today. The inherent low sensitivity, and non-linearity of thermoelectric sensors, coupled with difficulty in manufacture of the materials required for these sensors, eliminated them from consideration in this program, leaving the "electrical resistance" type thermometer as one which could be used to satisfy the remote monitoring criteria.

The electrical resistivity of an element or a compound varies with temperature, and can be used as a simple and reliable temperature measuring device. Many elements and compounds, however, are not suitable for use in low temperature resistance thermometry since they lack one or more of the desirable properties of an ideal resistance thermometer. These properties include: (a) a resistivity that varies linearly with temperature; (b) high sensitivity; (c) high stability of resistance so that calibration is retained over long periods of time; and (d) capability of being mechanically worked.

Resistance thermometers presently available for low temperature work are divided into the following three general types: (1) pure metals; (2) alloys; and (3) semiconductors. Each of these types is generally weak in one or more of the desirable properties of an ideal resistance thermometer. The pure metal resistance thermometers generally have low sensitivity below 20°K; the temperature range of alloy type resistance thermometers is usually limited to the range of 2°K to 20°K; and the semiconductor resistance thermometers which have been used to date have resistivities which are very non-linear functions of temperature and lack a simple mathematical expression for the resistance-temperature relationship.

Recent work on GaAs p-n junction diodes[1] has indicated that these devices offer all the advantages of other semi-conductor temperature sensors, i.e., high sensitivity over the entire temperature range from 2°K to 300°K, ease of fabrication, uniformity of sensor characteristics, etc., and in addition have a linear temperature response over the temperature range from 75°K to 300°K and deviate only slightly from that linear response between 2.1°K and 75°K. A theoretical expression has been obtained which fits the experimentally determined temperature dependence of the devices quite well down to 50°K. These characteristics (the linear temperature response of the device and the availability of a mathematical expression for the temperature relationship of the device) overcome the major objections to the use of semiconductor temperature sensors and indicate that these devices could be useful for cryogenic work where a high degree of accuracy is required. These considerations led to Phylatron's proposal to develop a GaAs p-n junction device for use as a cryogenic temperature sensor.

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[1] B. G. Cohn, W. B. Snow and A. R. Tretola "GaAs p-n Junction Diodes for Wide Range Thermometry" RSI Vol 24, No. 10, pp 1091, October 1963.

## OBJECTIVE

The objective of this program was to develop a Gallium-Arsenide (GaAs) p-n junction diode as a high sensitivity temperature sensor which can be used for remote monitoring and control of cryogenic temperatures in the range of 4.2°K to 300°K. The sensors must be capable of precise temperature measurement at fixed points, have negligible long term drift, and must have good repeatability of measurement when subjected to repeated temperature cycling.

## DEVICE DEVELOPMENT

The primary objective of this program was to develop a GaAs temperature sensor using the techniques which had been reported in the literature [2]. However, when the first group of devices was calibrated over the temperature range of 4.2°K to 77°K the sensitivity (change in forward voltage per degree change in temperature or $\Delta V/\Delta T$) was found to be too low and a research effort was initiated immediately in order to determine why the devices had such low sensitivity and how the sensitivity could be improved.

Since considerable effort was expended on diffusion studies, contacting, packaging, calibration, and junction evaluation, each phase will be discussed separately.

### STARTING MATERIAL

The p-n junction temperature sensors were fabricated from tellurium doped n-type GaAs obtained from Monsanto Chemical Corporation. The pertinent electrical parameters of the starting material are shown in Table I.

TABLE I

| Parameter | Symbol | Value |
|---|---|---|
| Hall mobility | $\mu_H$ | 3000 $cm^2/V$ sec. |
| Carrier Concentration | $n$ | $1.6 \times 10^{17}/cm^3$ |
| Resistivity | $\rho$ | $1.3 \times 10^{-2}$ ohm cm. |

The values which are quoted were supplied by Monsanto; however, these values were checked by an independent concern at Phylatron Corporation's request. The measured values agreed quite well with those supplied by Monsanto.

The high value of mobility indicates a high quality crystal with little or no compensating chemical impurities. The etch pit count of the crystal, which is related to the dislocation density, was $650/cm^2$ and also indicates a good quality crystal.

Material from two different ingots of GaAs was used in prepairing devices for this project. Both ingots had nearly identical electrical characteristics and there was no significant difference in characteristics of the devices produced from the different ingots.

### WAFER PREPARATION

The GaAs ingots were wafered by a diamond cut-off wheel. Since the material is very soft, it was necessary to cut the wafers approximately 20 mils thick and then lap them to the appropriate thickness prior to diffusion. The wafers were lapped with 400 grit SiC. on both sides to a thickness of approximately 15 mils.

[2] Ibid.

One side of the wafer was then polished with 0.25μ alumina ($Al_2O_3$) powder. The alumina polish gave an optical finish to the wafer surface: this surface finish is necessary to insure good diffusions and planar junctions in GaAs.

After polishing, the wafers were cleaned in a 1:1 solution of 37.3% HCL and 48% HF rinsed in distilled water to quench the etch, rinsed in alcohol to remove oil surface films, and dried.

## DIFFUSIONS AND DIFFUSION STUDIES

The p-n junctions were formed by the diffusion of Zn, which is a p-type dopant, into the n-type tellurium doped GaAs wafers. The initial diffusions were made using ZnO as a source for the Zn and diffusing in an evacuated quartz ampule.

The dopant source was prepared by mixing 1.4 gm. of crushed GaAs with 0.8 gm. of Zn and sintering the source at 800°C for one hour in an evacuated ($10^{-5}$ torr) quartz ampule.

The diffusions were made by placing both the p-type dopant source and the GaAs wafer in a quartz ampule, evacuating the ampule to $10^{-5}$ torr, sealing it and then placing it in a diffusion furnace which was operating at 800°C. After one hour the ampule was removed from the diffusion furnace and allowed to cool quickly to room temperature.

The diffusions were evaluated by visual inspection of the surface, measurement of the junction depth, and calculation of the surface concentration of Zn. Upon examination of the surface after diffusion, it was found to be smooth and have a dull appearance in contrast to the optical polish which existed prior to diffusion.

The junction depth was measured by an angle lap and stain technique. A 2.5° bevel was lapped on the p-type side of the diffused wafer and a stain etch was then applied to the beveled area. The stain etch was a solution of HCL:$H_2O_2$:HF:$H_2O$ in volume ratios of 5:5:3:30. Under conditions of high illumination (a focused 6.5V, 2.7A microscope lamp) this etch stains the n-type GaAs dark, while leaving the p-type material unstained. For this sealed ampule diffusion, the junction depth was found to be 0.8 mils below the p-type surface.

The surface concentration of Zn was calculated using the techniques of Mehta[3]. This method takes into account the concentration dependence of the diffusion coefficient of Zn.

The initial diffusions were carried out in a sealed quartz ampule. This system was adequate and could possibly have been used successfully for the entire program; however it was quite slow and the ampule had to be leak tested after sealing to insure that the diffusions were being made in vacuo. Also the sealed ampule system was not amenable to precise control or reproducibility. In view of these limitations an open box type diffusion system was constructed. A schematic diagram of the system is shown in Figure 1. This diffusion system operates in the following manner: The GaAs wafer to be diffused is placed on top of the quartz boat which contains the source material for the diffusion. The boat is then placed in the quartz capsule at the cool left end of the diffusion tube. The system is sealed and flushed for 30 minutes with Argon in order to insure a pure Argon atmosphere for diffusion. The quartz ampule is then pushed into the hot zone of the diffusion tube with the push rod shown. When the capsule is moved into the hot zone it is sealed on both ends by the end seals.

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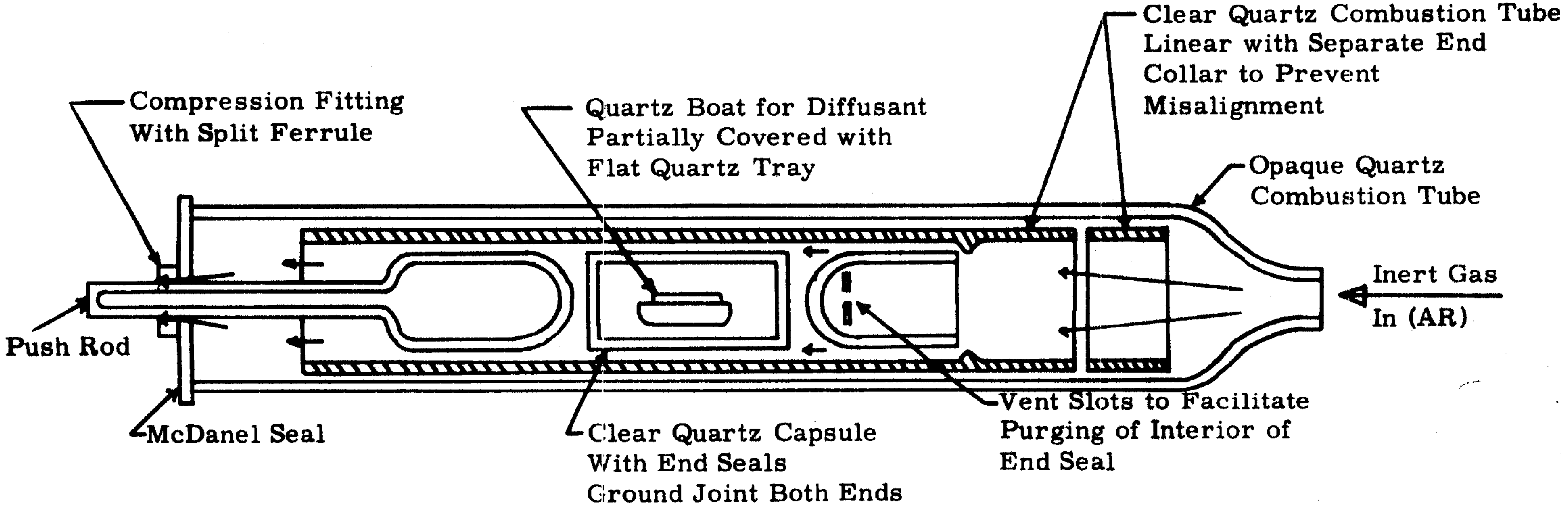
[3] R. Mehta "Analytical Study of Zn Diffusion in Gallium Arsenide and the Electrical Properties of the Resulting Diffused Layers" Technical Report No. 5103-1 Stanford Electronics Laboratories June 1964



Open Box Diffusion System

Figure 1

When the capsule seals it contains pure Argon and the diffusions are carried out in this atmosphere. If a seal should leak, the diffusion tube surrounding the system is filled with Argon, hence there is little chance of contaminating the sample during diffusion.

Devices fabricated from wafers which were diffused from a ZnO source in both the sealed quartz ampule and open box diffusion system were calibrated over the temperature range of 4.2°K to 300°K. The calibrations indicated that approximately 50 per cent of the devices fabricated from these wafers had adequate sensitivity from 300°K to approximately 60°K, but that the sensitivity fell off rapidly below that point to approximately -0.7 mv/°K at 20°K.

At this point a theoretical analysis was initiated to determine why the sensitivity of the devices decreased so rapidly at low temperature. It is known that the diffusion coefficient of Zn is dependent on the surface concentration of Zn, which in turn is dependent on the vapor pressure of the Zn source. The ZnO source gives extremely high surface concentrations, approximately $10^{20}$ atoms/cm$^3$. Diffusions from these high surface concentrations give extremely high Zn concentration throughout the entire p-type diffused region and yield very abrupt junctions. Analysis of the data taken on devices made from the wafers which were diffused from a ZnO source indicated that the sensitivity of the devices was directly related to the initial forward voltage drop at a given current at room temperature. That is, the higher the forward voltage drop, the higher the sensitivity. Devices with approximately 0.7 to 0.8 volts drop at 1.0 ma at room temperature had very low sensitivity over the entire temperature range, while devices with 0.9 volts drop or higher had adequate sensitivity to 60°K. The relationship suggested that a more graded junction which was capable of sustaining a higher voltage drop for a given current was necessary. The data also showed that the devices became very insensitive to temperature changes at approximately 60°K and nearly all the devices approached a maximum forward voltage drop of 1.4 volts at 1.0 ma at 4.2°K.

The fact that all the devices behaved identically at low temperature, coupled with the fact that the Zn concentration was extremely high in the "p" region, suggested that a metal-semiconductor junction was forming rather than a p-n junction. A calculation of the barrier height for such a metal-semiconductor junction from the difference in work functions of Zn and p-type GaAs yielded a barrier height of approximately 1.38 eV, which was in good agreement with the experimental results. Discussions with Dr. Cohen[4] confirmed the previous conclusions.

This analysis indicated that a lower surface concentration of Zn and a more graded junction (a metal-semiconductor junction is very abrupt) were needed in order to make high sensitivity temperature sensors. Elemental Zn which is known to yield low ($10^{16}$-$10^{17}$ atoms/cm$^3$) surface concentrations and which, under proper diffusion conditions, will give graded p-n junctions, was selected as a source.

The elemental Zn sources were made in the following manner: The Zn (99.999% purity) was etched in 48% HF to remove surface oxides and was then placed in the open box diffusion tube along with some powdered GaAs. 0.4 grams of Zn was mixed with 0.7 grams of powdered GaAs and the mixture was reacted at 800°C for 1.0 hour in an Argon atmosphere.

The remainder of the GaAs diffusions were made using these elemental Zn sources in the open box diffusion system. Several trial diffusion runs were made to optimize the diffusion process. The time-temperature cycle selected as optimum for these devices using the elemental Zn source was 825°C for 2 hours. This

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4. B. G. Cohen, Bell Telephone Labs Private communication.

diffusion cycle produced p-n junctions in the GaAs which were approximately 0.4 mils to 0.8 mils below the surface. The surface concentration of Zn was $10^{18}$-$10^{19}/cm^3$, and junction capacity measurements indicated the junctions were graded ($n = 2.4$, where $n=2$ indicates an abrupt junction and $n=3$ indicates a linearly graded junction).

Some difficulty was experienced in control of junction depth as the Zn sources depleted rapidly, i.e., the vapor pressure of Zn varied from diffusion to diffusion.

Devices fabricated from elemental Zn diffused wafers had higher sensitivity than devices fabricated in the same manner from ZnO diffused wafers. However, below approximately 40°K the sensitivity of the elemental Zn diffused devices also began to decrease. A comparison of the characteristics of a ZnO and elemental Zn diffused device is shown in Figure 2.

## CONTACTING

After diffusion the wafers were lapped to a final thickness of 6 mils. The wafers were then etched in 48% HF and rinsed in distilled water, and electrical contacts were applied to both the p and n sides of the wafer in the following manner: The clean wafers were nickel plated for five minutes in an electroless nickel plating bath. The bath was composed of 15 gm. of Nickel Chloride, 5 gm. Sodium Hypophosphite, 25 gm. Ammonium Chloride, 32.5 gm. Ammonium Citrate and 500 cc of water. The plating bath was kept at 95°C and maintained basic by the addition of Ammonium Hydroxide as needed.

The nickel plate was bonded to the GaAs by sintering. The sintering process was carried out in the open box diffusion chamber in an Argon atmosphere. The sintering temperature was 600°C and the time was 10 minutes.

Following the nickel sintering step, the wafer was etched in 48% HF for five minutes, rinsed in distilled water and gold plated. The gold plating solution was potassium gold cyanide. The temperature of the plating bath was maintained at 95°C. The thickness of the plated gold layer was approximately 1.2 $mg/cm^2$.

## MESA FORMATION

In order to make useful devices from the contacted wafers, it was necessary to etch mesa structures on the p-type side of the wafer. The main purposes of the mesa are to reduce surface leakage currents and confine the junction geometry so that suitable bulk current densities can be obtained.

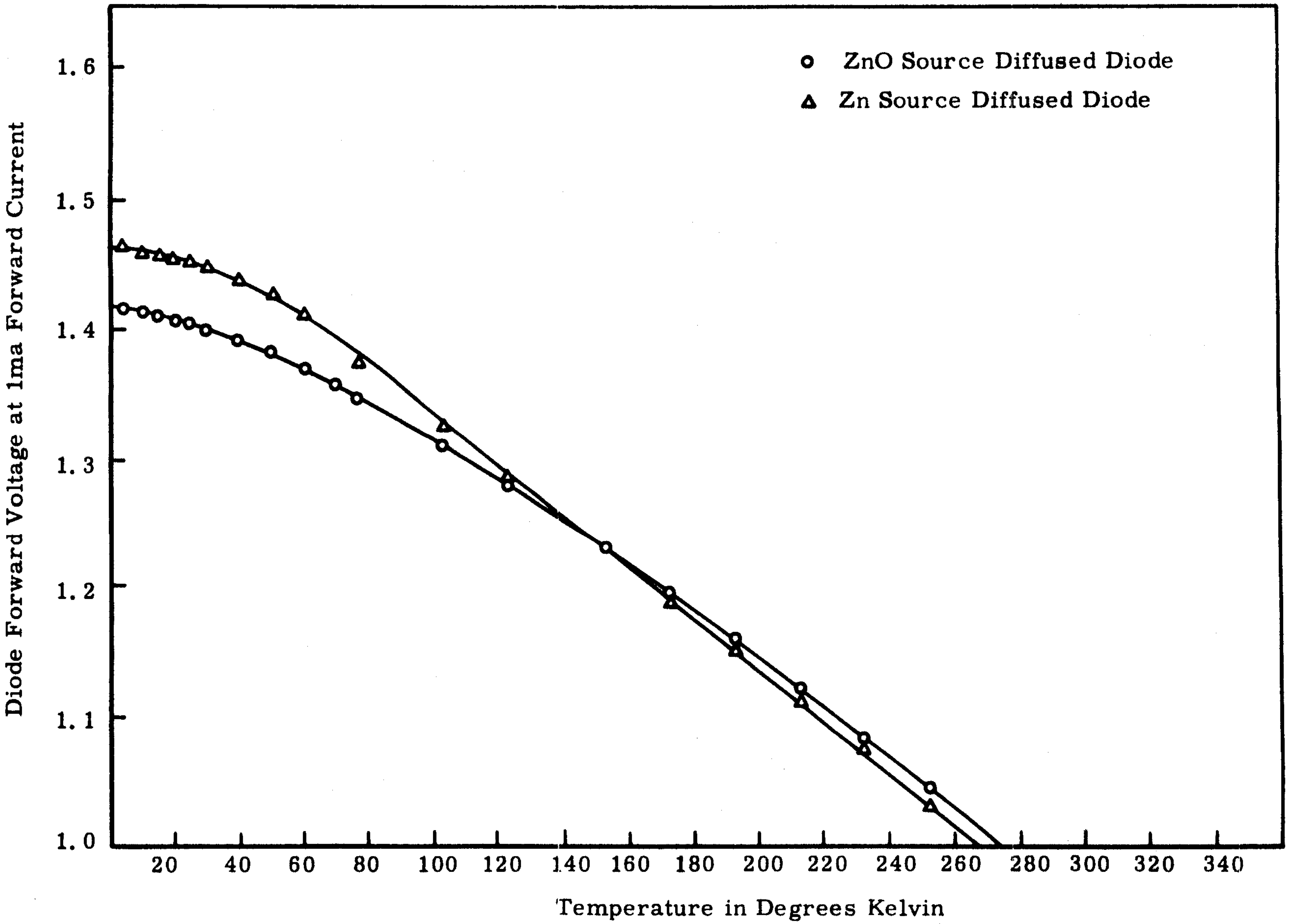
The mesas were formed by masking uniformly spaced small areas of the p-type side of the wafer with wax and then etching away the unprotected gold contact plating and the GaAs.

The wax used for masking was Ceresin wax which has a melting point of 65°C. The wax was applied with a heated, small bore ($\sim$4 mil I.D.) hypodermic needle. The dots applied with this needle were nearly circular and had approximately 4 mil to 6 mil diameters.

The etching process was carried out in two steps. First the unprotected gold contact plating was removed with a 1:1 solution of saturated $K_2I_2$ and water. Then approximately 1.5 mils of GaAs was removed by etching for 30 minutes in a 3:2:2 solution of $CH_3OH:H_2PO_3:H_2O$.

The Ceresin wax was then removed by washing the wafer in warm benzene and rinsing in alcohol.

The p-n junctions formed in these wafers were approximately 0.4 to 0.8 mils below the p-type surface. Since approximately 1.5 mils of GaAs is removed in the unmasked regions, the cross section of the p-n junction is well defined by the mesa.



Sensivity Comparison for Devices Diffused
from a ZnO Source and an Elemental Zn Source

Figure 2

Each device requires only one mesa containing a p-n junction so the wafers were cleaved into individual dice at this point in the process.

The wafers were cleaved under alcohol to prevent loss of many of the small fragments. The instrument used for cleaving was a sharp razor blade.

The cleavage planes of GaAs are the (110) crystallographic planes. The ingot from which the wafers were cut was grown in a (111) direction. The intersection of the 110 planes with the (111) crystal face form triangular patterns, hence the configuration of the dice is triangular. A schematic diagram of an individual die is shown in Figure 3.

## WAFER AND LEAD BONDING

Regardless of the type of package selected for device encapsulation, the next process step is bonding the individual device to the package header and bonding a lead to the contact on the mesa. A thermocompression bonding technique was used to form both the header and mesa bonds.

The header bonding process can be described as follows:

1. The header was placed in a machined slot in a graphite strip heater.
2. A small disc, 0.002" thick, approximately the same size as the device, of Au:Sn:Sb, (83%:16%:1%) solder was placed on the header.
3. The GaAs diode was placed on top of the solder, with the n-type base in contact with the solder.
4. Pressure was applied to the top of the GaAs diode by means of a sapphire point.
5. The header assembly and diode were enclosed in a forming gas atmosphere.
6. The heater strip was heated until the Au:Sn:Sb solder melted and wet the diode and header and then the assembly was allowed to cool rapidly.

The headers were Au plated as were the GaAs wafers. The solder wet both the header and the device and in effect formed a gold-gold bond at the interface.

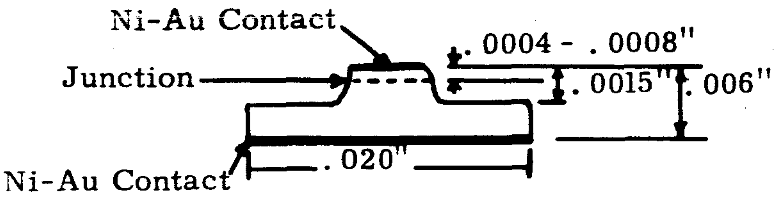
Contact was then made to the p-type mesa by thermocompression bonding a 1 mil annealed gold wire to the top of the mesa structure. The bonding was done in a forming gas atmosphere and a heated sapphire point was used to apply both heat and pressure to form the bond. Due to the nature of the tool, the bond would be considered as a wedge type bond.

During this bonding procedure, the surface of the mesa where the junction is exposed, is degraded. This degraded surface leads to surface leakage currents, which in turn degrade the junction characteristics and are detrimental to the temperature voltage characteristics of the diode. The effect of the surface degradation can be decreased by a post-bonding clean-up etch. The surface clean-up was accomplished by etching with concentrated $HNO_3$. In addition to cleaning the surface, the $HNO_3$ also passivates the surface to some degree, thereby protecting it from further atmospheric deterioration.

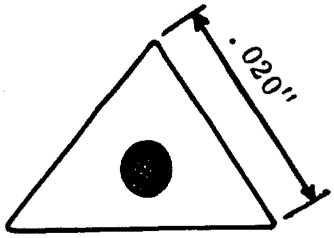
Unfortunately, during this clean-up etch, the top contact on the mesa is unprotected and will also etch. Considerable difficulty was encountered in controlling the etching time so that the p-n junction surface was "clean" while not etching away a significant portion of the top contact of the mesa. The clean-up etch was carried out in 4 second steps, with distilled water used to quench the etching action between steps. The progress of the clean-up etch was monitored by observing the reverse characteristics of the diodes on an oscilloscope.

## PACKAGING

It is necessary to package nearly all semiconductor devices. The main objective of packaging is to provide mechanical and chemical protection for the device. Mechanical protection implies that the package be strong enough to withstand all the stresses encountered in normal use. Chemical protection implies



Side View



Top View

Schematic Diagram of Completed Die

Figure 3

that the package provides a sealing or passivation to moisture and other environmental conditions which may cause changes in the surface properties of the device which will in turn lead to a change in the characteristics of the device.

The use of a semiconductor device as a cryogenic temperature sensor imposes several other stringent conditions on the device package. Among these conditions are the following: (a) the package must have a high thermal conductivity over the entire temperature range in order to provide good thermal contact between the device and its surroundings and allow the device to come to equilibrium with its surroundings rapidly; (b) the thermal expansion coefficients of the various parts of the package must be well matched to prevent failure due to thermal stresses in the package; (c) the thermal expansion coefficients of the package header and the semiconductor device must be well matched to prevent strains in the device or bonds which could lead to failure.

## CERAMIC PACKAGE

The package shown in Figure 4 (a), which was originally proposed for this cryogenic sensor was one which had suitable characteristics for use in this temperature range, and, in addition, was much smaller in diameter than standard transistor packages. Considerable difficulty was encountered in the use of this package. The major problem was connected with the final package seal, i.e., when the top pin was bonded to the ceramic. Several techniques for making this seal, including brazing, thermocompression bonding, and low temperature soldering were tried, but none proved successful. The thermocompression bond produced a very weak joint that pulled apart easily under a mechanical pull test. The low temperature solder bonds also had no mechanical strength. The brazing which was done in an $H_2$ atmosphere at 200°C - 400°C took the 1 mil gold contact wire into solution, i.e., the eutectic between gold and the brazing material was lower than the melting point of the brazing material and the wire alloyed with the other material and melted. Several brazing techniques were tried, but the results were the same in each case. The brazed joint was, however, very strong and apparently formed a good hermetic seal.

Due to the time scale of this program and the other problems encountered in fabrication of a sensative cryogenic sensor, the ceramic package was not perfected and standard transistor packages were used instead for the device encapsulation.

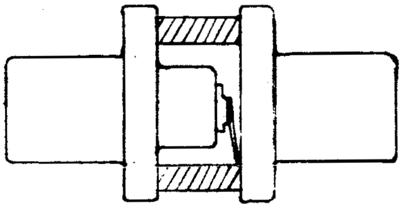
## STANDARD TRANSISTOR PACKAGES

Both TO-18 and TO-46 transistor packages were purchased and evaluated for temperature sensor application. Scale diagrams of the two packages are shown in Figures 4, b and c, respectively. The main difference between the two packages is the amount of metal in the header. The TO-18 header has a thin metal jacket over an epoxy center and glass to kovar seals for feed throughs. The TO-46 header is all metal with small glass to kovar seals for feed throughs.

Diodes were mounted on both types of headers by the techniques previously discussed, and the packages were sealed in the following manner: A 5 mil thick (Au 0.6% Sb) wire ring was placed on the header and a standard cap was placed on top of the wire while the package was in a helium atmosphere. The package was then resistance compression spot welded. The weld current was 55 amperes, and the weld time was 0.05 seconds. This weld produced a very strong package bond.

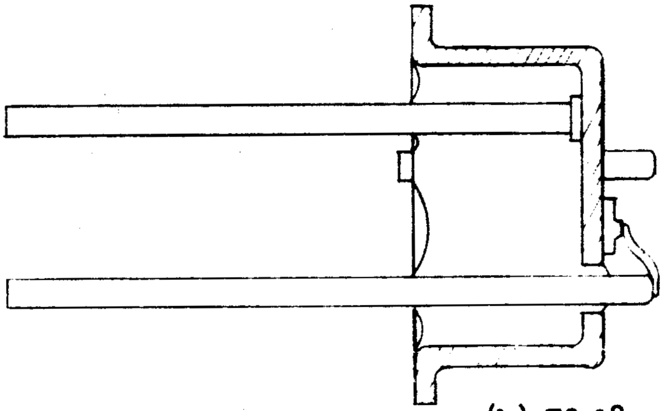
The packages were thermally stressed by rapidly dipping them in liquid nitrogen. None of the packages failed after repeated thermal stressing.

After it was determined that both the TO-18 and TO-46 packages could stand the thermal cycling, the packages were tested to determine which one had the highest thermal resistance, i.e., which package had the greater time constant for temperature stabilization. Thermocouples were mounted on a TO-18 header and a



Scale: 1" = 1/10"

(a) Ceramic Package



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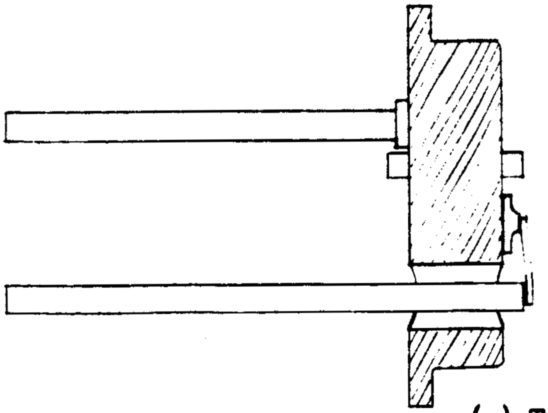
(b) TO-18



Scale: 1" = 1/10"

(c) TO-46

Figure 4.

TO-46 header and the headers were mounted on a thermoelectric cooler. A thermocouple was also mounted directly to the cooler. The temperature of the thermoelectric cooler was decreased rapidly and the time lag between the thermocouples mounted on the headers and the thermocouple mounted on the cooler was measured. The results of these tests are shown in Figure 5. The data can be explained in the following manner: At t=0 the temperature of the headers and cooler was 25°C. At t greater than zero, the temperature of the cooler was decreasing and at t=12 seconds, the temperature of the cooler and TO-46 header was 20°C, whereas the TO-18 header did not cool to 20°C until 25 seconds had elapsed.

The results indicate that the TO-46 header has less thermal resistance than the TO-18 header, and is hence better suited for use as a cryogenic sensor package.

## CALIBRATION AND TEST

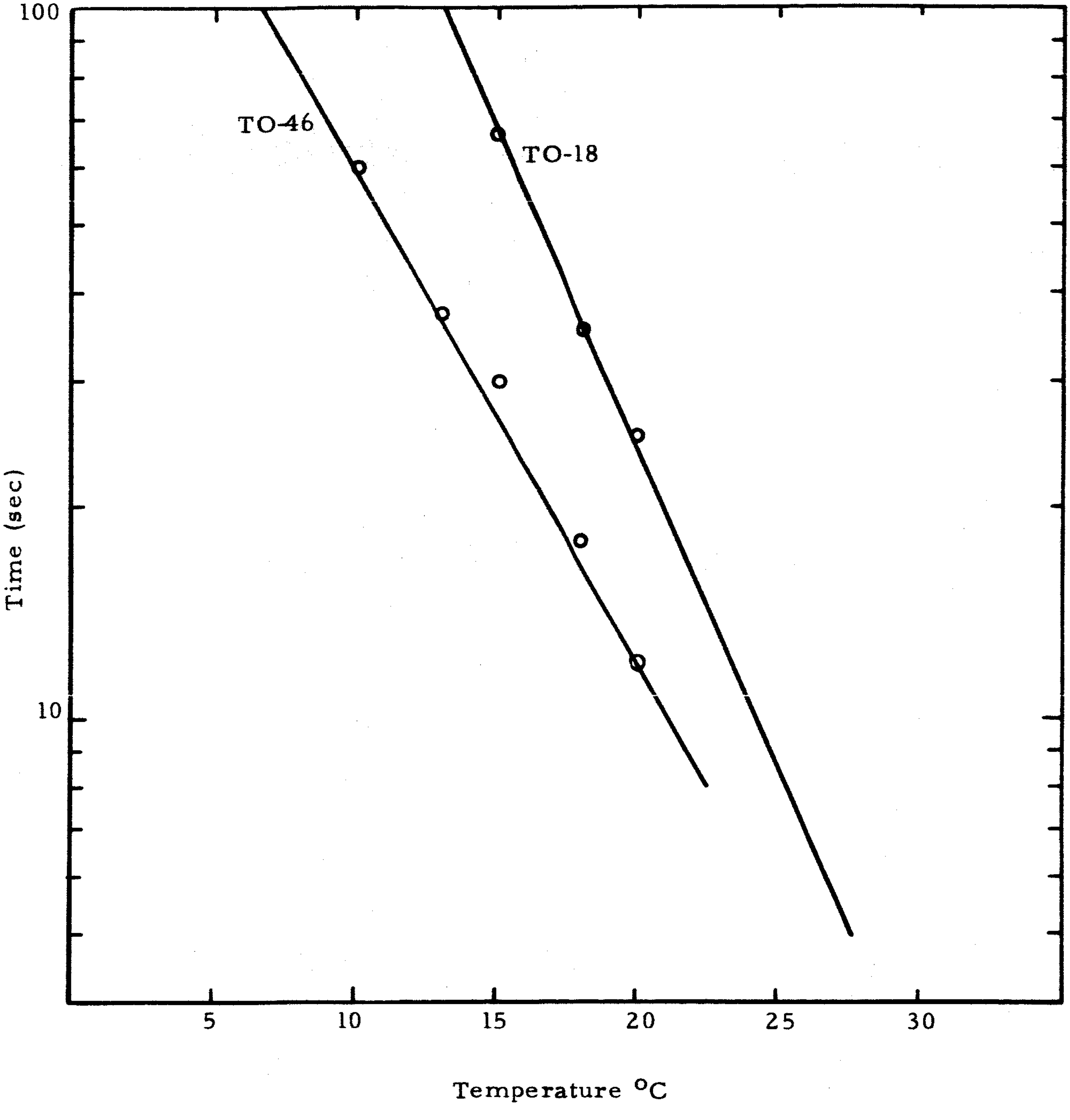
In order to determine the sensitivity, reproducibility, and long term aging characteristics of the GaAs temperature sensors, and in order to calibrate them against a known standard thermometer, it was necessary to design and build a precision calibration circuit. The circuit which was built and used on this work is shown in Figure 6. The constant current supplies are modified Power Designs Model 605, supplies with a current stability of 0.01%. The ammeters were standard laboratory meters which had recently been calibrated against a secondary standard at the Ohio State University. At 1.0 ma on the standard meter, the meters read 1.0 ma. The Leeds and Northrup K-3 potentiometer was used to measure the resistance of the platinum resistance thermometer. The K-3 has an accuracy of 0.015% of the measured value on the scales used. There is an additional error of 0.5 microvolt on the low (high sensitivity) range, and 2.0 microvolt on the medium sensitivity range. Neglecting the liquid helium point where there was no sensitivity data on the platinum resistance thermometer, the maximum error that should have been introduced on the temperature readings on the platinum thermometer was ±0.20°K at 10°K. The K-3 uses a null method of voltage measurement and therefore, there is no current flow in the voltage lead wires when a resistance reading is made. This eliminates any need for correction due to lead resistance. The digital voltmeter used to measure the voltage across the GaAs sensor was a Non-Linear Systems Model 481. This meter has an accuracy of 0.01% full scale ± one digit. In this calibration work the ± one digit was the limiting factor on the temperature reading. For the case of -2.0 mv/°K sensitivity, the one digit instability introduces a maximum error of ±0.5°K. It was possible to improve the accuracy of the GaAs temperature readings at the specified test points simply by switching to the K-3 for measurement of the forward voltage drop across the GaAs device. The digital voltmeter has an imput impedance greater than 10 meg ohms and therefore there is essentially no current flow in the voltage measuring leads and no lead resistance correction was necessary.

The overall accuracy of this test system was sufficient to maintain the accuracy required on the temperature measurements at the fixed temperature test points.

## SHOCK AND REPRODUCIBILITY TESTS

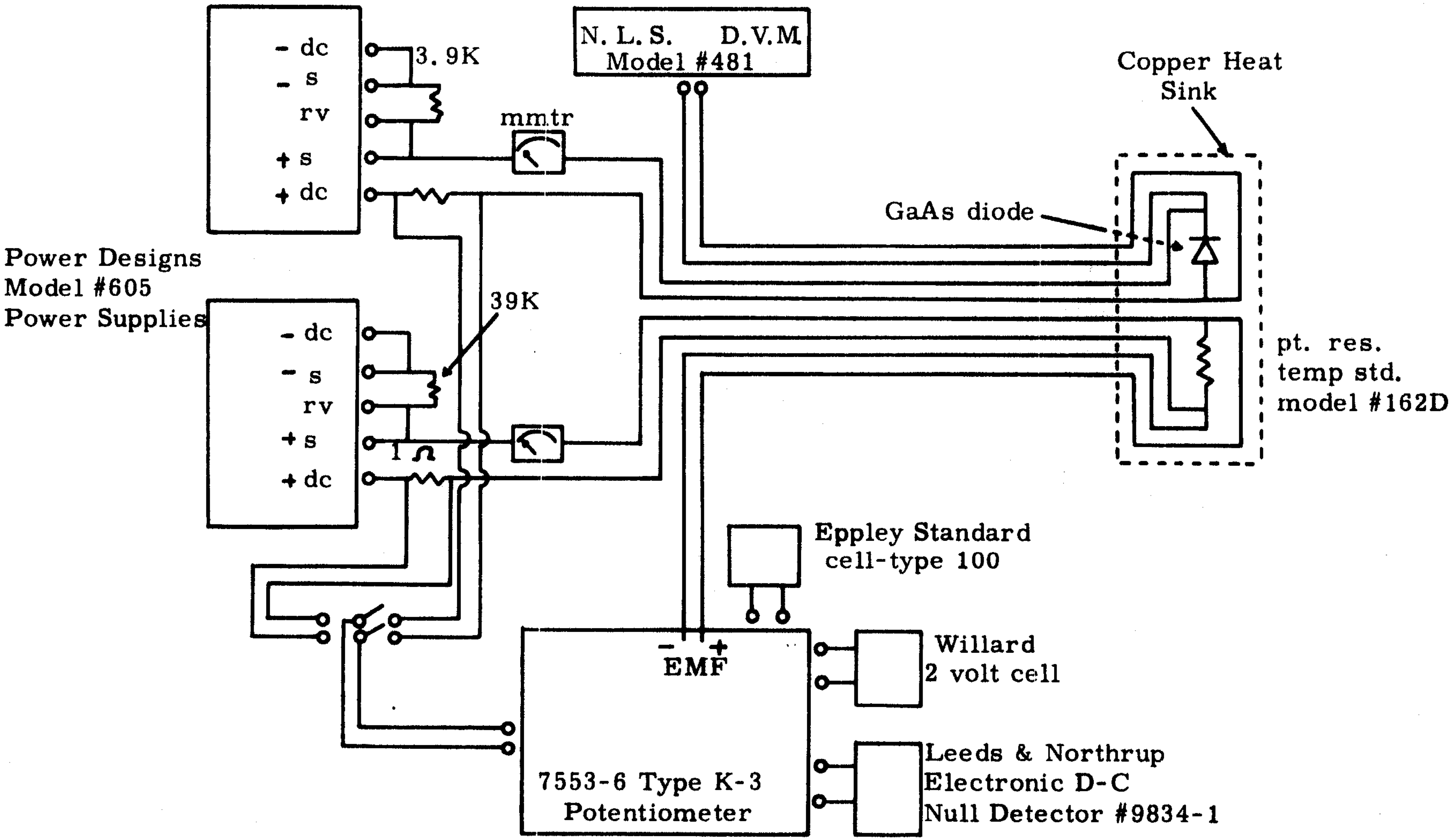
Shock tests were performed on both empty device packages and completely packaged sensors. The purpose of these tests was to determine whether the package and the encapsulated sensor could withstand the thermal shock caused by rapid immersion in liquid nitrogen.

Results of the shock tests on the ceramic package indicated that the package could withstand the thermal stress when both pins were brazed to the ceramic.



Thermal Lag for TO-18 and TO-46 Headers

Figure 5



Calibration Facility

Figure 6

However, the soft solder and the thermocompression bonds between the top pin and the ceramic failed frequently under repeated thermal shock.

Both the TO-18 and the TO-46 packages which were resistance spot welded, formed mechanically strong seals and were unaffected by thermal shocking.

Several of the initial attempts at the thermocompression bonding the GaAs diode to the header were made without the use of the Au:Sn:Sb solder. These bonds occasionally failed under shock test; however, when solder was used between the header and the device, the bond withstood the shock tests quite well.

It was possible to perform repeated thermal shock tests on the completed sensors and test their reproducibility at the same time. This was done by placing the sensor in a copper block with the platinum resistance thermometer, connecting the platinum thermometer and the GaAs sensor to the calibration system, immersing both the platinum thermometer and the GaAs sensor in liquid nitrogen, waiting for the system to reach equilibrium and then measuring the temperature on both the platinum and the GaAs devices. After the devices reached equilibrium at 77°K (liquid nitrogen) and the temperature readings were recorded, the copper block containing the platinum thermometer and the GaAs sensor was warmed quickly to 273°K, where the temperature was again recorded.. The devices were cycled between 77°K and 273°K a minimum of 10 times and the temperature was recorded at the end points. Approximately 80 per cent of the devices tested were able to withstand the repeated thermal shock and reproduce the readings at both 77°K and 273°K to within ±0.5°K.

## CALIBRATION TESTS

Devices which withstood the shock tests and could reproduce temperature readings at 77°K and 273°K within ±0.5°K were then calibrated using the calibration system described previously. The devices were initially calibrated between 77°K and 300°K. Devices that had a sensitivity of approximately -2.0 mv/°K at 273°K and approximately -1.5 mv/°K at 77°K were then calibrated between 4.2°K and 300°K. Approximately 50 percent of the devices tested had this sensitivity. In order to obtain the actual shape of the curve over the entire temperature range, measurements were taken at 5°K, 10°K, and 20°K intervals rather than just at the specified calibration points. Typical calibration curves taken at a forward current of 1.0 ma were shown previously in Figure 2. The sensitivity of the device at any temperature T is simply the slope of the calibration curve at that point. Many of the devices tested had nearly constant sensitivity between 77°K and 300°K.

## SENSITIVITY AS A FUNCTION OF CURRENT DENSITY

Several of the GaAs devices were calibrated at various values of constant forward current. The forward current range was 0.1 μa to 1.0 ma. The results of such a calibration test are shown in Figure 7. These tests indicated that the room temperature sensitivity of these GaAs devices showed little dependence on current density. This is in contrast to results reported by Cohen[5] and indicates a fundamental difference between the two groups of devices.

## LIFE TEST

A group of twenty devices was put on a thirty day life test. The purpose of the test was to determine the long term stability of the GaAs temperature sensors under continuous operation. The stability was determined by calibrating the devices over the temperature range of 4.2°K to 300°K, once every week for the thirty day period.

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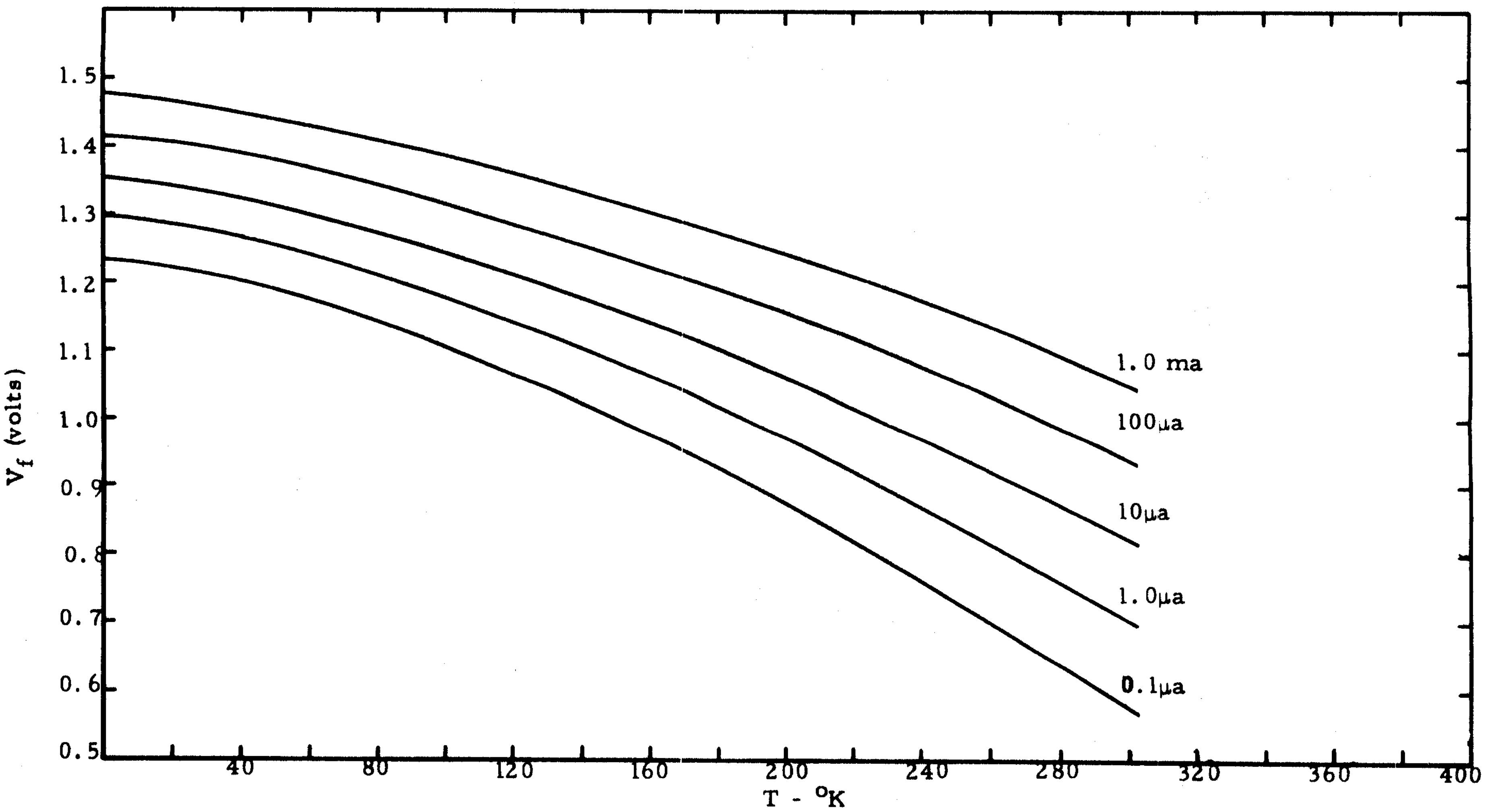
5. Cohen Op. cit. R.S.I.



Sensitivity Vs. Forward Current for a GaAs Temperature Sensor

Figure 7

Four of the devices which were life tested showed early signs of instability, i.e., gave erratic voltage readings at fixed temperatures, and finally opened up electrically. Apparently, either the Au wire contacting the p-type mesa broke, or the device came loose from the header. The remaining sixteen devices performed well over the entire period. Weekly calibrations showed less than a $\pm 0.5^{\circ}$K temperature variation at any of the fixed test points, and there was no indication that the device characteristics were changing with time.

The devices which were life tested were shipped to NASA along with the calibration curves and calibration data.

## JUNCTION EVALUATION

The low sensitivity of the GaAs devices in the low temperature range, below $50^{\circ}$K, required an investigation of the fundamental properties of the devices and the device processing techniques in order that the devices could be improved as temperature sensors. Several of the investigations, both theoretical and experimental were discussed previously in relation to the device development phase of the program. Since the GaAs sensor characteristics are primarily related to the p-n junction formed in the GaAs, the major portion of the experimental investigation was directed toward determining the nature of the junction, its characteristics and how these characteristics changed with temperature. The techniques used for investigation involved measurement of the terminal electrical characteristics of the devices. The measurements and their interpretation will be discussed separately.

## CURRENT-VOLTAGE CHARACTERISTICS

The forward I-V characteristics of the devices were used to determine the mode of operation of the device. The measurements were made primarily by the use of a Tektronix curve tracer. Analysis of these measurements indicated that devices with higher forward voltage drops for a given current at room temperature were more sensitive to temperature change than devices with lower forward voltage drops.

A p-n junction diode has a forward I-V characteristic which can be expressed as:

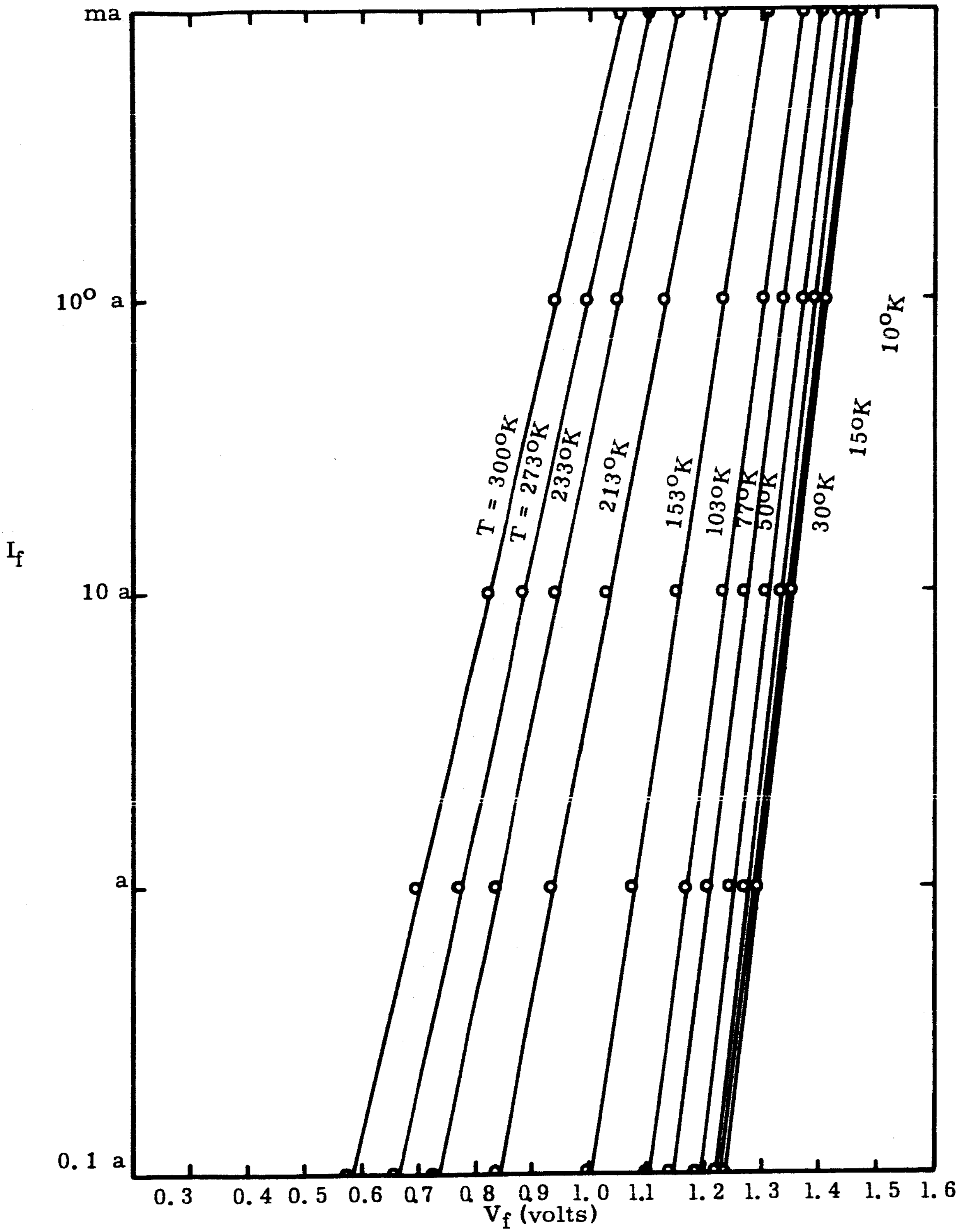
$$I=I_s e^{qV/nkT}$$

where I is the forward current, $I_s$ is the saturation current, V is the junction voltage, n is a constant which is determined by the dominant current mode, and q, k and T have their usual significance, i.e., electron charge, Boltzman factor, and temperature.

Semi-logartithmic plots of the I-V characteristics were made to determine whether the junction characteristics followed this relationship, and to determine the value of n in the current expression. Figure 8, which also shows the I-V characteristics as a function of temperature, is typical of the Zn diffused junction devices. As can be seen from the figure, the exponential relationship between current and voltage exists over four orders of magnitude in current.

The slope of the curves in Figure 9 is related to the factor n. Since q, k, and T are constants for any given temperature, n can be calculated from the slope. Typical room temperature values of n for the Zn diffused diodes were between 2.0 and 2.5. According to Sah, Noyce and Shockley[6], a value of n=2 is indicative of a diode which conducts primarily by a generation-recombination currents.

[6] C. T. Sah, R. N. Noyce, and W. Shockley Proc. I.R.E. 45 1228 1957



Forward Current Versus Forward Voltage as a Function of Temperature for Typical GaAs Temperature Sensor Diode #48

Figure 8

The I-V characteristics at both 300°K and 273°K fit the logarithmic relationship between current and voltage quite well over the current range from 0.1 μ amp to 1.0 ma. Since the characteristics do fit the theoretical relationship and show no change in n over that current range, there is no reason to expect a change in sensitivity ($\Delta V/\Delta T$) over the given current range at room temperature. If the diode characteristics were to become dominated by diffusion currents at higher current levels, i.e., I=1.0 ma or higher, then n would decrease from 2 to 1 and the sensitivity would be expected to change. This, however, is not the case with these Zn diffused diodes. The forward current is predominantly a generation-recombination current over the entire current range and the sensitivity remains nearly constant.

The value of n begins to increase at approximately 100°K and continues to increase at lower temperatures. The reason for this behavior is not clear at this time. Perhaps the apparent increase in n is due in part to an increase in surface leakage currents at lower temperatures. Further investigation would be necessary to clarify this point.

### CAPACITANCE-VOLTAGE CHARACTERISTICS

It can be shown that the capacitance of a p-n junction is related to the applied voltage in the following manner:

$$C = K \frac{1}{(V_D-V)^{1/n}}$$

where K is a proportionality constant, $V_D$ is the diffusion potential, and V is the applied voltage. The constant n is determined by the type of junction and can vary between 2 and 3. If the p-n junction is abrupt, then n takes on the value of 2. If the junction is linearly graded, n takes on the value of 3. In general, most devices have values of n somewhere between 2 and 3 depending on the abruptness of the junction.

A measurement of the junction capacity vs the applied voltage is thus a method of determining the type of p-n diffusion obtained from a particular set of diffusion conditions. Figure 9 is a typical plot of the junction capacitance of a diode diffused from an elemental Zinc source. The slope of the curve gives an n=2.4 which indicates that the junction is graded.

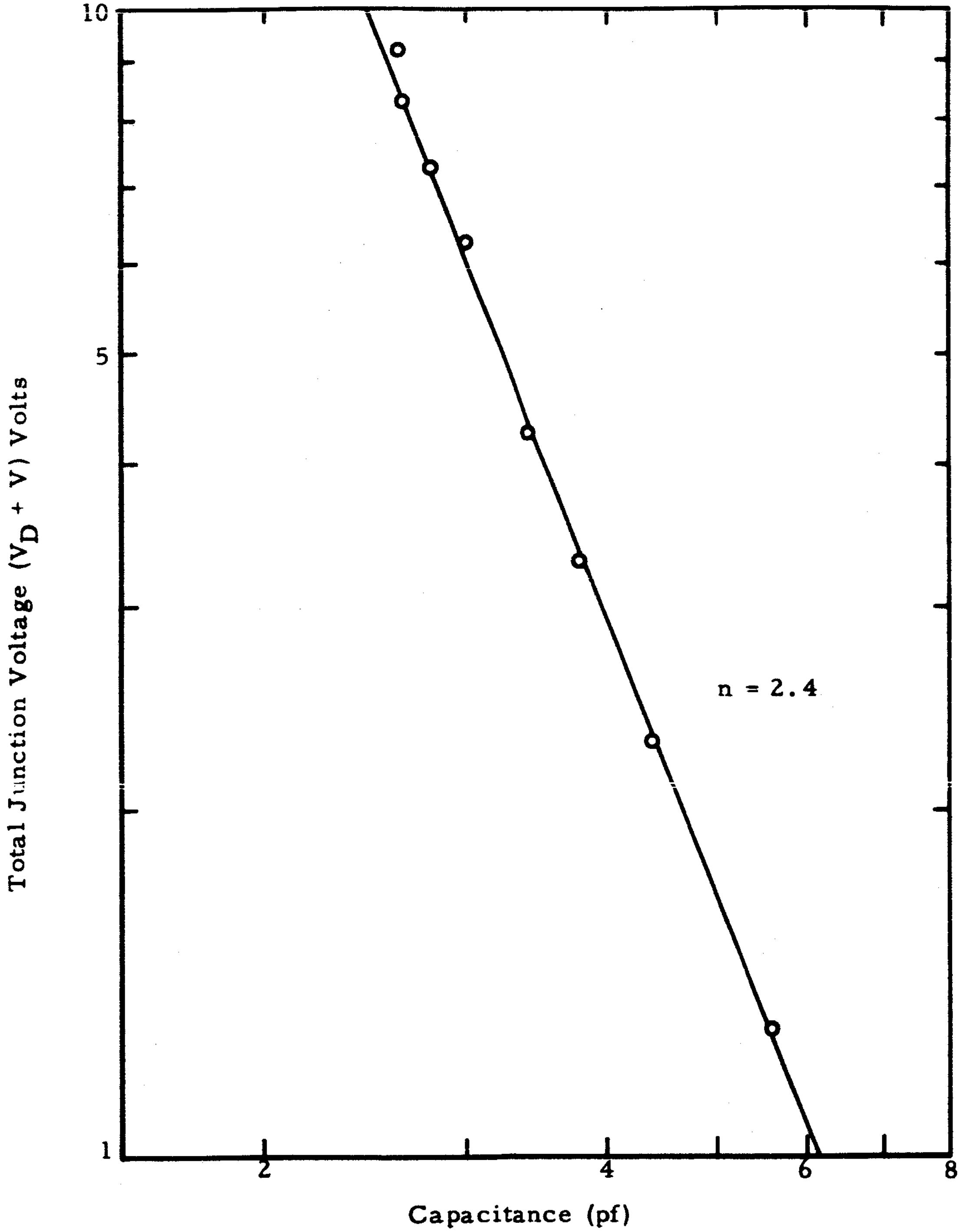
The junction capacity was measured with a Boonton RX meter. The diodes were mounted directly to the meter terminals and the internal DC voltage supply was used as a bias voltage V. The measurement is made by superimposing a small AC signal (less than 10 mv peak to peak) with a frequency of 1 KC on top of the DC bias and measuring the capacity by an AC technique.

### SELF HEATING

An important consideration for any cryogenic sensor is the amount of self heating produced by the device. Using the data obtained from the Hughes EPIC reports on the thermal conductivity of GaAs, the self heating of the device was calculated at 4.2°K, 77°K, and 300°K for a forward current of 1.0 ma. The results indicated that the self heating of the devices is approximately $1 \times 10^{-2}$°K at 4.2°K, $3 \times 10^{-3}$°K at 77°K and $1 \times 10^{-2}$°K at 300°K. These low values indicate that the devices are adequate for very accurate low temperature thermometry.

## CONCLUSIONS

Through the experimental efforts of this program the technology has been developed to produce a GaAs p-n junction device which is suitable for use as an accurate thermometer in the temperature range of 4.2°K to 300°K. The sensors are very small and when packaged properly, such as in a TO-46 transistor package, have very little thermal lag. The sensitivity of the devices is nearly a factor of 100 better than the sensitivity of a standard platinum resistance thermometer



Junction Capacitance vs Junction Voltage

Diode #16

Figure 9

over the entire temperature range and therefore, can be used in any system to replace the platinum thermometer and improve the accuracy.

The self heating of the devices is low and therefore the devices can be used in applications where power dissapation is a limiting factor. Also, since the self heating is low, the accuracy of the temperature measurement can be improved.

Unlike most GaAs devices which are high frequency devices and require abrupt junctions, the temperature sensors require graded junctions. The diffusion process which produced the best junctions and hence the best temperature sensors was diffusion from an elemental Zn source at 825°C for two hours in an open box system. The diffusions were carried out in an inert Argon atmosphere.

The forward current of the elemental Zn diffused devices was dominated by generation-recombination current over the entire current range from 0.1 μ amp to 1.0 ma at 300°K and hence there was little change in device sensitivity at that temperature.

The low temperature, below 60°K, sensitivity of the sensors is only adequate and could possibly be improved with further investigation. The reason for the low sensitivity of the ZnO diffused devices at low temperature was that a metal-semiconductor junction was formed instead of a p-n junction, and as the junction voltage approached the difference in work function between Zn and p-type GaAs, the current increased rapidly for small increases in the forward voltage. The reason for the low sensitivity of the elemental Zn diffused devices is not known, however, one possible explanation may be surface leakage currents. Reasons for suggesting this include the rapid increase in n below 100°K and the experimentally observed changes in the I-V characteristics after the surface clean-up etch. If the surface leakage currents are causing the sensitivity to decrease at low temperature, then new bonding techniques which would reduce surface deterioration should be investigated.

Devices which are properly bonded and packaged can readily withstand repeated thermal shock and are capable of reproducing temperature measurements at fixed points to within ±0.5°K. The long term stability of the devices also appears to be adequate. The devices were capable of reproducing any temperature measurement within ±0.5°K when continuously tested over a thirty day period. Thus, if any drift did occur, it was less than 0.5°K.