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Copies should not be returned to the Aeronautical Systems Division unless return is required by security considerations, contractual obligations, or notice on a specific document.
This Final Technical Documentary Report covers all work performed under Contract AF 33(657)-7104 from 5 September 1961 to 31 December 1962. The manuscript was released by the author on 28 February 1963 for publication as an ASD Technical Documentary Report.

This contract with the W. R. Grace & Co., Research Division of Clarksville, Maryland, was initiated under ASD Project 7-838, "Reproducible Thermistor Refinement Program". It was administered under the direction of Lieutenant T. Bailey, Electronics Engineer, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Dr. M. C. Vanik of W. R. Grace's Washington Research Center, Inorganic Chemical Research Department, was the supervisor in charge. Others who cooperated in the research and in the preparation of the report were: Dr. M. G. Sanchez, Director of Inorganic Chemical Research Department; Dr. W. T. Barrett, Former Director of Inorganic Chemical Research; Mr. J. E. Herrera, Chemist, Inorganic Chemical Research Department; Mr. E. M. Glocker, Manager Research Statistics, Economic Evaluation Department; Mr. S. E. Ketner, Statistician, Economic Evaluation Department and Miss B. B. White, Assistant Librarian, Research Services Department.

This project has been accomplished as a part of the Air Force Manufacturing Methods Program. The primary objective of the Air Force Manufacturing Methods Program is to develop, on a timely basis, manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. The program encompasses the following technical areas:

- Rolled Sheet
- Forgings
- Extrusions
- Castings
- Fiber & Powder Metallurgy
- Component Fabrication
- Joining
- Forming
- Materials Removal
- Fuels
- Lubricants
- Ceramics
- Graphites
- Non-Metallic Structural Materials
- Solid State Devices
- Passive Devices
- Thermionic Devices

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.
ABSTRACT

Technical Documentary Report

ASD-TDR-63-387
February 1963

FINAL REPORT ON REPRODUCIBLE THERMISTOR REFINEMENT PROGRAM

Milton C. Vanik
et al
W. R. Grace & Co.
Research Division

Gold-doped monocrystalline silicon exhibits temperature-resistivity behavior suitable for making highly reproducible, predictable and sensitive thermistors. Two types of thermistors were developed with reproducibilities of ±2% and operable ranges including -85 to +200°C. Manufacturing methods were developed and demonstrated on an unbalanced pilot line.

A literature survey indicated that specially doped silicon or germanium offer the best possibilities of being used as single-crystal semiconductor thermistors. Silicon was chosen for development on the basis of energy gap, purity, resistivity, and availability.

Many elements were screened as possible silicon thermistor dopants. These include gold, copper, silver, nickel, iron, zinc, platinum, manganese, and thallium. Only gold in both P- and N-type silicon gave products suitable for use in the -85 to +200°C range.

Gold-doped N-type silicon produces a high resistivity thermistor material with nearly linear \( \rho \) vs \( 1/T \) response in the range -85°C to +200°C. This material has a temperature coefficient of resistivity of -7% per degree at 25°C. It is most suitable for high temperature, narrow range, high sensitivity applications.

Gold-doped P-type silicon is a lower resistivity thermistor material with linear \( \rho \) vs \( 1/T \) behavior over the range of -85 to about 50°C. The temperature coefficient of resistivity of this material is -4.5% per degree at 25°C.

A statistical study was completed comparing float-zone leveled with diffused gold-doped thermistor silicon. The float-zone leveling process produces the better product.
Forming of ohmic and mechanically strong contacts to the thermistor silicon is extremely important. Stability of the thermistors is very dependent upon these contacts.

Manufacturing processes encompassing 24 steps for gold-doped N-type thermistors and 28 steps for gold-doped P-type thermistors have been established. An unbalanced pilot line was set up and operated according to these processes. A demonstration run was completed for the Air Force contract office.

Six hundred samples were produced on this pilot line and sent to the Air Force for distribution. Many of these samples are within ±1% of a standard curve. All are within contract specifications of ±2%.

These thermistors should be useful in any application which calls for precise and/or closely matched thermistors.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

SACK R. MARSH
Assistant Chief
Manufacturing Technology Laboratory
Directorate of Materials & Processes
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I INTRODUCTION

Conventional thermistors are now made from transition metal oxides by ceramic techniques. These metal oxides are semiconductors. Their electrical resistance decreases as they are heated. Dozens of other semiconductor materials could conceivably be used in place of the present thermistors as temperature-sensing devices. However, a material that is to be selected for thermistor use should have adequate sensitivity to temperature, reproducibility, and reliability.

Probably the purest and most reproducible semiconductor materials available today are the large single crystals of silicon and germanium that are used in the manufacture of electronic devices such as transistors and diodes. Silicon crystals often contain electrically active impurities as low as one ppb or less.

The objective of this project is the development of manufacturing methods and the pilot plant production of thermally sensitive devices which take advantage of the high reliability and reproducibility offered by large single-crystal semiconductors.
II DISCUSSION

1. Literature Search

A literature search has been completed covering all available information on thermistor element properties, thermistor element fabrication, and those properties of semiconductors applicable to temperature-sensing devices. A copy of this survey is attached in the appendix. This survey indicates that specially doped germanium and silicon offer the best possibilities of being used as single-crystal semiconductor thermistors. For instance, it points out that germanium doped with arsenic is a suitable low-temperature (\(\sim 10^5\) K or less) thermistor\(^{(1)}\). When doped with iron, germanium produces a thermo-sensitive device operating from 0 to -200°C\(^{(2,3)}\). The temperature resistance behavior of manganese-doped germanium is described\(^{(4)}\). A straight line log resistivity versus 1/T relationship is obeyed in the temperature range of about 60°C to -140°C. Silicon doped with iron has been used in a thermally sensitive device operating in the -100° to +100°C range\(^{(5)}\). The resistivity of the iron-doped silicon is about 500,000 ohm cms at 25°C. A low-temperature thermometer (2° to 25°K) of copper-doped gallium arsenide is described\(^{(6)}\). Tantalum-doped aluminum antimonide also produces a thermistor material\(^{(7)}\).
2. Evaluation of Single-Crystal Semiconductors as Thermistor Materials

Several criteria are necessary for a semiconductor material which is to be fabricated into a single-crystal thermistor operating in a temperature range of -85°C to +200°C. The energy gap between the valence band and the conduction band should probably be greater than about 1 ev. Otherwise, appreciable intrinsic conduction might occur at temperatures below 200°C destroying any linearity in the conduction obtained by the introduction of resistivity-determining impurities. This would tend to eliminate germanium, indium arsenide, indium antimonide and gallium antimonide from the following list of single-crystal semiconductors:

| Table 1 |
|-----------------|--------|
| Eg(300°K)       | (eV)   |
| Si              | 1.09   |
| Ge              | 0.66   |
| InP             | 1.27   |
| InAs            | 0.33   |
| InSb            | 0.17   |
| GaP             | 2.25   |
| GaAs            | 1.43   |
| GaSb            | 0.70   |
| AlSb            | 1.52   |

In germanium, for instance, intrinsic conduction could become appreciable at 100° to 125°C and interfere with the conduction obtained by introducing impurities that create the so-called deep-lying energy levels between the valence band and the conduction band.

If one considers purity of the monocrystalline semiconductors, including both resistivity-determining impurities and electrically inactive impurities, only germanium and silicon can be obtained in the parts-per-billion range. Silicon is considerably better than germanium in that respect. If one considers which of the above materials are readily available commercially, only silicon and germanium fit the bill. For the reasons mentioned above, we felt that properly doped silicon offered the best possibilities for making a thermistor with the properties described in the contract. Germanium was not eliminated from our consideration, however, and some experimental work was done with it.
3. Screening of Dopants in Silicon

In this laboratory we doped silicon with various impurities that introduce energy levels between the valence and conduction bands of silicon. In order to obtain conduction resulting from energy gaps on the order of 0.2 ev or higher, it was necessary to dope with impurities that introduce the so-called deep-lying impurity levels. The normally used Group III and Group V elements which introduce shallow levels (less than about 0.1 ev) were not used except in conjunction with the deep-lying levels. We began a process of screening dopant materials which might introduce the desired deep-lying energy levels.

Most of the screening was done by float-zone leveling techniques. By this technique, impurities which have a low segregation coefficient (such as most of the dopants in which we were interested) can be added in a very uniform manner to a silicon rod during the float-zone process. A single crystalline rod of known resistivity is mounted in a zone refiner and butted against a seed of known resistivity. The dopant is weighed in at the seed rod junction, fused into the first zone, and then passed thru the rod with the zone. As the zone traverses the length of the rod, an essentially constant level of impurity is left behind. The distribution coefficient of an impurity in float-zone refining is the ratio of the impurity in the crystallizing solid to the amount in the molten zone. If the distribution coefficient is small, gold is about $3 \times 10^{-5}$, the amount that crystallizes from the zone is so small compared to the amount in the zone that the quantity in the zone essentially remains constant throughout a pass. Hence the amount of impurity left in the rod at any point is essentially constant.

Other techniques which are used to add impurities were crystal pulling and diffusion techniques. When pulling a crystal by the Czochralski technique and using a dopant with a low distribution coefficient, the impurity left in the crystal is inversely proportional to the fraction of the melt remaining. For instance, when 25% of the melt has been crystallized, the amount of impurity in the crystal at that point is two-thirds the amount of impurity in the crystal when 50% of the crystal has crystallized. This technique is therefore useful in determining the effect of varying amounts of dopant upon the temperature-resistance characteristics of a material. Diffusion was used mainly for doping silicon with gold. Diffusion was carried out in an argon ambient at 1200°C. If reasonable diffusion times are to be realized, the diffusion rate of the impurity in the substrate must be relatively fast. Such is the case of copper and gold in silicon.

Elements which were screened as dopants for thermistor silicon include gold, copper, silver, nickel, iron, zinc, platinum, manganese, and thallium. Only the highest-purity spectrographic- or electronic-grade dopants were used. No erratic behavior was noted from contamination of the dopants with impurities with segregation coefficients close to one. One would expect to see the effect of a contaminant such as boron (distribution coefficient $\approx 0.8$) and phosphorus.
(distribution coefficient 0.3) at the seed end of the crystal since only one pass is given in the float-zone leveling process. For most of the dopants which have distribution coefficients on the order of 3 x $10^{-5}$, 0.1 gram added to the initial zone of a 15/16" rod results in a residual rod concentration of about 20 ppb. This is based on the estimate of the zone size being equal to 20 grams of molten silicon.

Evaporation of zinc and thallium dope was so severe that RF arcing made float-zoning impossible. Thallium was tried by diffusion techniques. However, the results were not promising. Thallium has an acceptor level at 0.26 eV above the valence band, and we attempted to activate this level by diffusing thallium into 50-80 N-type silicon at 1200°C. As shown in Graph 1, a plot of $\log_{10} \rho$ versus $1/T$ is not linear over the temperature range -85° to 200°C. Furthermore, the thermistors cut from the same diffused silicon wafer were not reproducible as far as their temperature-resistance behavior was concerned. For these reasons, thallium was discarded as a possible silicon thermistor dope.

Iron, manganese, and platinum resulted in polycrystalline float-zone rods even when added at very low concentrations in the float-zone leveling process. Gold, silver, copper, and nickel were added until polycrystallinity occurred during float-zone leveling. With gold, this corresponds to about 0.15 grams per zone; silver, 0.15 grams; copper, 0.2 grams; and nickel, 0.06 grams. Silver and nickel were used as dopants in high-resistivity (greater than 100 ohm cm) N-type silicon, and medium resistivity (35 ohms cm) P-type. The $\log_{10} \rho$ versus $1/T$ plots of these elements as shown in Graphs 2 and 3 were not linear nor reproducible from one sample to another. Copper, which has a donor level 0.23 eV above the valence band, was also tried by diffusion techniques. Copper was diffused into a 3mm silicon wafer for 24 hours at 1200°C. The $\log_{10} \rho$ versus $1/T$ plot of float-zone leveled Cu is shown in Graph 4. There is an undesirable deviation from linearity. The diffusion results were even more erratic.

Gold-Doping of Silicon

Of the elements tried, gold in both P- and N-type silicon produced products which appeared most suitable for thermistor use in the -85° to +200°C range. Gold was first added to 40-60 ohm cm N-type silicon. The resulting silicon had a resistivity of about 150,000 ohm cm at 25°C. This resistivity was dependent on the phosphorus level. When the log of the resistivity of the gold-doped N-type silicon is plotted versus $1/T$ as in Graph 5, a straight line results over the range of -85° to 200°C with an activation energy measured from the slope of 0.54 eV. This slope corresponds to the gold acceptor level located 0.54 eV below the conduction band(9). Sufficient gold must be present to overcompensate the N-type impurities rendering them inactive electrically and to provide additional levels for the 0.54 eV conduction. In the particular case noted, 21 ppb of gold were added to a bar containing approximately 1.5 ppb of excess N-type carriers.

The effect of the gold level relative to the excess N-type carriers

- 5 -
in the starting silicon is shown in Graph 6. The three samples plotted were cut from a pulled crystal in which the gold level was nearly critical. That is, the ppb of gold were approximately equal to the ppb of excess phosphorus at the head of the crystal. Since the gold (distribution coefficient $3 \times 10^{-5}$) concentration increases more rapidly than phosphorus (distribution coefficient 0.35) the relative gold concentration at 40% of the pulled crystal is larger than at 10%. Notice that the degree of linearity increases from 10 to 30% and at 40% there is linearity. This is the point at which the gold is in slight excess over the phosphorus.

Annealing experiments have further demonstrated the criticality of the gold level. Samples were annealed in argon at 300, 500, and 900°C for 48 hours. The samples had been cut from silicon which contained gold in excess over the N-type impurities. The silicon had been prepared by float-zone leveling. As shown in Graph 7, little change was noted in the temperature-resistance behavior of samples annealed at 300 and 500°C; the samples annealed at 700°C and 900°C began to lose linearity, however. At these temperatures, some of the gold is probably migrating to dislocations or other imperfections, reacting with them, becoming electrically inactive and resulting in a sub-critical gold level and non-linear temperature-resistance behavior.

In fabricating a precise thermistor from the gold-doped N-type silicon described, some difficulties were encountered. Most electronic devices, such as radiosondes, will not tolerate resistances above several megohms because of the parallel conduction of the chassis, insulators, and moisture. With the material described, if the resistance at the lowest temperature, -85°C, was about one megohm, the 25°C resistance of such a unit would be about 100 ohms. Since L/A for such a device would be about $10^{-3}$ cm$^{-1}$, even if the silicon were cut and lapped to the thinnest wafers possible (say 6-8 mils thick) the area of such a device would be completely unreasonable, being on the order of 20 cm$^2$. Therefore, material with such a high resistivity would not make a convenient low-temperature thermistor purely from geometry considerations. However, on the upper end of the temperature range, such material could make a very suitable thermistor and the log $\rho$ versus $1/T$ behavior is nearly linear all the way to 200°C.

Another factor to be considered with gold-doped N-type silicon is the temperature sensitivity. The gold-doped N-type silicon described is very sensitive, exhibiting a temperature coefficient of resistivity of about -7.1% per degree at 25°C. In a radiosonde, the electronic circuitry will tolerate a resistance change of about three decades over the -85° to +30°C range. For this purpose, the 0.54 ev thermistor is too sensitive, exhibiting about five decades of change in the same region. For some applications, it is therefore desirable to have a thermistor material which has a lower activation energy and a lower resistivity.
With this in mind, we tried to activate the 0.34 ev donor level of gold in silicon. The 0.34 ev level was activated by gold doping P-type silicon. This doping was accomplished by both float-zone leveling and diffusion techniques. When diffusing, single-crystal silicon wafers approximately 6mm thick are plated or dusted with gold and diffused in an argon ambient at 1200°C for 24 hours. The gold first melts into the surface of the silicon, causing an etched-like appearance and then diffuses throughout. About 1000 ppb of gold can be added by this technique according to the literature (10). By zone leveling techniques, about 30 ppb of gold is the maximum that can be added before polycrystallinity occurs. We feel that this difference is due to the fact that zone leveling results primarily in substitutional gold, whereas diffusion results in substitutional gold as well as gold which migrates to crystalline imperfections. Graph 8 is a typical plot of log $\rho$ versus $1/T$ for a P-type bar doped with gold by float-zone leveling techniques. Notice that the resistivity is about 400 ohm cm at 25°C. This is considerably lower than that obtained in the gold-doped N-type silicon case. Also, notice that there is some deviation from linearity at higher temperatures; there is essentially linearity between $+30^\circ$C and $-85^\circ$C, however. This is the practical working range of a radiosonde.

The resistivity of the gold-doped P-type silicon appears to be a function of the excess boron content, as indicated in Table 2 below:

<table>
<thead>
<tr>
<th>TABLE 2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>P-Type Resistivity Before Doping at 25°C (ohm cm)</strong></td>
</tr>
<tr>
<td>0.33 - 0.47</td>
</tr>
<tr>
<td>1.1 - 1.6</td>
</tr>
<tr>
<td>9.2</td>
</tr>
<tr>
<td>50 - 66</td>
</tr>
</tbody>
</table>

All samples were dusted or plated with gold and diffused in argon at 1200°C, for 24 hours. This relationship was significant because we had a technique for controlling resistivities so that a desirable L/A could be obtained for a device. This material undergoes about three decades of resistance change over the radiosonde temperature range.

In P-type silicon, the product obtained by diffusion is lower in resistivity than the product obtained by float-zone leveling. For example, a 50-66 ohm cm P-type silicon bar, when diffused with gold, has a resistivity of about 80.6 ohm cm at 25°C, while the float-zone leveled product has a resistivity of 400 ohm cm at 25°C. Since more gold is added by the diffusion technique, it is felt that the resistivity of gold-doped P-type silicon is also a function of the amount of gold present.
Another interesting phenomenon is the apparent decrease in activation energy or thermal sensitivity as the boron content of gold-diffused silicon is increased. Table 3 below gives activation energies of diffused silicon at various levels of excess boron:

<table>
<thead>
<tr>
<th>P-Type Resistivity Before Doping at 25°C (ohm cm)</th>
<th>Activation Energy AE of Linear Portion By Least Squares (electron volts, ev)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.33 - 0.47</td>
<td>0.22</td>
</tr>
<tr>
<td>1.1 - 1.6</td>
<td>0.24</td>
</tr>
<tr>
<td>9.2</td>
<td>0.26</td>
</tr>
<tr>
<td>50 - 66</td>
<td>0.33</td>
</tr>
</tbody>
</table>

Notice that the activation energy drops off to approximately 0.22 ev as the resistivity of the starting boron-doped silicon decreases to 0.4 ohm cm.

Statistical Study

At this point, we felt that the feasibility of using specially doped silicon as a thermistor material had been demonstrated. By varying the silicon type and the amount of gold dope, a series of thermistor materials with suitable activation energies and resistivities should be obtainable. As mentioned previously, doping can be accomplished either by diffusion or float-zone leveling techniques. A statistical study was begun to determine the variabilities to be expected in making devices by the diffusion or float-zone leveling techniques. The decision to make thermistors by diffusion or zone leveling was based on this study.

The float-zone leveled samples in this comparison were prepared by float-zone leveling a 7/8-1", 15 to 20 ohm cm P-type rod with 0.15 grams of high-purity gold. The rod was cut into 12 wafers approximately 5 millimeters thick. After hand lapping with 180 grit SiC paper, they were nickel plated on both sides by the electrodéless technique. Each wafer was cut into twelve 4mm x 4mm x 5mm thermistors. The wafers were numbered alphabetically and the thermistors within a wafer were numbered numerically. Copper leads were soldered to the thermistors with tin, and resistances were measured over the temperature range of -75° to 30°C. Eight rather evenly spaced measuring points were used over this temperature interval. Resistivities were calculated from the resistance readings at various temperatures and the physical size of the thermistors as measured by a vernier caliper.

The first step in preparing the diffusion samples was cutting a 15 to 20 ohm cm P-type rod into 12 wafers approximately 6 millimeters thick.
These wafers were plated with gold on both sides and diffused at 1200°C for 48 hours. The gold-etched surface of these wafers was lapped away so that the final thickness of the wafer was approximately 5 millimeters. The wafers were nickel plated by the electrodeless technique and each was then cut into 12 thermistors as described previously. Again the wafers were numbered alphabetically and the thermistors within a wafer were numbered numerically. Copper leads were soldered to the thermistors with tin, and the resistances were measured as in the float-zone leveled case.

The resistance vs. temperature data were then fed into a Bendix G-15 Computer which computed the least square lines through the points as well as the resistivity profiles along these computed lines. Since in the case of gold-doped P-type silicon there is some deviation from linearity of a log resistivity vs. 1/T plot at temperatures above ~30°C, only the points from +30 to -75°C were analyzed.

Standard deviations, \( \sigma \)'s, were calculated by variance analysis for \( \Delta E \) and resistivities at 25°C. A summary of the results of this analysis is given in the table.

**TABLE 4**

<table>
<thead>
<tr>
<th>Standard Deviations (( \sigma ))</th>
<th>Diffusion</th>
<th>Float-Zone Leveling</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \Delta E )</td>
<td>Resistivity at 25°C (% Average Reading)</td>
</tr>
<tr>
<td>Uniformity of devices within a wafer</td>
<td>0.011</td>
<td>11.5%</td>
</tr>
<tr>
<td>Uniformity of devices from wafer to wafer along a rod</td>
<td>0.019</td>
<td>12.0%</td>
</tr>
</tbody>
</table>

The standard deviations for resistivity of device to device and wafer to wafer along a rod are the same order of magnitude by both doping processes. However, the corresponding activation energy deviations are considerably better in the case of float-zone leveled material. This parameter is independent of physical size but the resistance is not. It was felt that some of the inability to control resistance was due to variation of physical size. The devices had been fabricated as cut on a precision diamond saw. A 2% error in dimensions could result in a 6% error in resistivity. Precision lapping could help reduce this error. The slope of a thermistor must be controlled very accurately to meet the requirements of the contract. The statistical data indicated that the float-zone leveling process produces the least variable product.
Improvements in Measuring Technique

Resistance measurements were improved by use of the comparison method or ratio technique. An unknown thermistor and a standard thermistor are mounted in opposing arms of a Wheatstone bridge:

\[
\frac{R_{\text{UNK}}}{R_{\text{STD}}} = \frac{R_A}{R_B}
\]

FIGURE 1

Both thermistors are immersed in the same measuring bath. The reading obtained is a ratio of the resistance of the unknown to the standard resistance. Standards are usually chosen so that the ratio will be approximately equal to one. The time constants of the thermistors are small compared to the time of temperature cycling of the bath. The effects due to temperature fluctuation in the measurements baths tend to be eliminated, since these do not appreciably affect the ratio at a given temperature. This technique improved our measurement precision considerably. A statistical study was completed comparing standard deviations of material measured by ratio technique and by direct Wheatstone bridge resistance measurement.
TABLE 5

Standard Deviations ($σ$)

<table>
<thead>
<tr>
<th></th>
<th>Conventional Wheatstone Bridge</th>
<th>Ratio Technique Using Wheatstone Bridge</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Resistivity at 25°C (ev) (% Average Reading)</td>
<td>Resistivity at 25°C (ev) (% Average Reading)</td>
</tr>
<tr>
<td>Uniformity of devices within a wafer</td>
<td>0.0041 1.2%</td>
<td>0.0017 1.6%</td>
</tr>
<tr>
<td>Uniformity of devices from wafer to wafer along a rod</td>
<td>0.0046 1.1%</td>
<td>0.0019 0.8%</td>
</tr>
<tr>
<td>Average value</td>
<td>0.3621 433</td>
<td>0.3648 436</td>
</tr>
</tbody>
</table>

The material measured by the ratio technique shows a significant improvement in activation energy deviations. Resistance deviations are about the same. The deviations represent a considerable improvement over those obtained by direct readings.

The ratio-measuring technique is very important as far as test measuring of the pilot line samples is concerned. There are many samples to be checked at various temperatures. Since all of the thermistors from a given run are to be uniform in resistance within ±2%, the production thermistors should be compared with a standard thermistor of the proper resistance at the various testing temperatures. Samples showing ratios below 0.98 or above 1.02 should be rejected. The variation in temperature of our baths at a given temperature level is on the order of ±0.01 to 0.001°C. The ratio technique tends to eliminate this variation and enables us to measure ratios without deviations that would be experienced if using a Wheatstone bridge and measuring resistance directly.

This technique was also useful in measuring samples during size adjustment which will be discussed later.
4. **Contact Problems**

During the course of the early work there were indications that the nickel-silicon contact being used in the experimental thermistors was causing some non-uniformity in the finished devices. Several factors led to this belief. The standard deviations for reproducibility of repeated measurements on a thermistor were often equal to, or greater than, the deviations for devices within a wafer or from wafer to wafer along a rod. As the length of a thermistor was decreased, the resistance was much higher than predicted by a low resistance contact. There seemed to be a dependence of the resistance ratio or resistance on the amount of current or voltage applied to a thermistor while being measured. We decided to make a systematic study of the contact problem and to consider other contact materials.

Two thermistors were measured by the ratio technique previously described. An ammeter was mounted in the measuring circuit as shown:

![Diagram of thermistor measurement circuit](image)

**Figure 2**

The amount of current flowing through the thermistor while not equal to the current registered on the ammeter is proportional to it. Graph 9 demonstrates the behavior of the resistivity ratio as the current in the circuit is increased. The two curves are a result of reversing the two thermistors in the ratio arms. Notice that ratio variations on the order of 8% can result by varying the current through the thermistors. These results are not believed to be due to the heating effect. The heat effect is the build-up of a temperature gradient in a heat-producing medium due to the inability of a system to dissipate heat rapidly. It is inversely proportional to the coefficient of thermal conductivity. This problem is discussed in the literature(11). In the present case the power produced in a thermistor under normal measuring conditions is on the order of a millimicro-watts.

- 20 -
The temperature change to be expected from such heat production is on the order of 10-50°C, if the system were allowed to come to equilibrium. Such a temperature change would not cause a significant decrease in resistivity. It would take a change of 0.44°C at 25°C to result in a 2% error in resistivity. Besides, the standard thermistor would be varying in temperature along with the unknown thermistor, thus minimizing any decreases in resistance due to heating.

Further examples of the resistance behavior of nickel contacts to gold-doped silicon are demonstrated in Graphs 10 and 11. Both measurements were made on the same thermistor at 0°C and -35°C. The resistances were read directly on a standard Wheatstone bridge with the current through the bridge varying as shown on the ordinate of the graphs. Notice that as the current increases from 0, the resistance of the device increases, reaches a peak, and then steadily decreases. The dashed curves are the result of depressing the galvanometer key for an extended period of time and show the result of the heat effect especially at higher currents. It is interesting to note that the peak of the curve shifts toward the left as the temperature is decreased. In fact, at -75°C there is often no peak at all, but a steady decline of the resistivity as a function of the current.

It was obvious that the nature of the nickel to gold-doped silicon contact would have to be studied further. A 4 x 4 x 5mm gold-doped thermistor was set up for potentiometric measurement as shown in Figure 3.
TABLE 6

<table>
<thead>
<tr>
<th>REFERENCE POINTS</th>
<th>RESISTANCE READINGS (OMS)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-8</td>
<td>1072</td>
</tr>
<tr>
<td>1-5</td>
<td>524</td>
</tr>
<tr>
<td>1-3</td>
<td>257</td>
</tr>
<tr>
<td>1-2</td>
<td>207</td>
</tr>
<tr>
<td>1-7</td>
<td>911</td>
</tr>
<tr>
<td>5-8</td>
<td>560</td>
</tr>
<tr>
<td>4-6</td>
<td>532</td>
</tr>
</tbody>
</table>

Potentiometric measurements eliminate the effect of current since the current flow through the potentiometer probes is 0. Voltage readings were made between various points on the thermistor as indicated in the diagram.

Results of the measurement from points 1 to 8 indicated that the total resistance of the element is 1,072 ohms. The measurement between points 1 and 2 indicated the contact resistance is on the order of 200 ohms. By comparing measurements between points 1 and 2 and points 1 and 7, it was seen that the resistance of the gold-doped thermistor element was approximately 700 ohms. We had a case in which 400 ohm cms of a total element resistance of 1072 ohm cms is accounted for by contact resistance. This was much too great and could have had serious consequences, especially on thinner elements.

Another series of experiments had indicated that the nickel to gold-doped silicon contact varied with temperature in a manner similar to gold-doped P-type silicon. This usually occurred when the resistivity of the silicon to which the plating was attached was high. Graph 12 shows the effect of plating ordinary P-type silicon of various resistivities with nickel and subsequently measuring the total resistance of the element. In this case, the silicon slugs measured 4 x 4mm in size. In the P-type case when the resistance is high enough (excess impurity concentration is low) a linear log A vs. 1/T behavior similar to that of gold-doped silicon occurs from about -75 to 0°C.

This behavior might have two explanations. It may be due to the diffusion of nickel during the plating and soldering process into the silicon immediately adjacent to the contact. This nickel could introduce energy levels with conduction properties similar to those introduced by gold. In the high P-type case, the chances of the nickel controlling the conduction picture are greater and the phenomenon will manifest itself more easily.

Another possible explanation of this phenomenon may be the establishment of a surface barrier potential when the nickel is plated onto the gold-doped P-type silicon. This generally occurs because the Fermi level of the
semiconductor is higher than the Fermi level in the metal. When contact is made, the electrons flow from the semiconductor to the metal since they are at a higher potential energy. This leaves the semiconductor with a net positive charge which attracts the electrons in the metal to the contact. The positive electrostatic charges in semiconductor are attracted toward the contact so that a contact barrier potential occurs. This contact is usually rectifying and essentially non-ohmic. It is influenced by carrier injection and applied voltage. Technique of preparation of the contact can often have a great deal to do with the rectifying or ohmic behavior of a contact. (12) Frequently, contacts can be made more ohmic by merely roughing or damaging the surface before the application of the plating. Needless to say, the behavior of such contacts is unpredictable.

Contacts on Gold-Doped P-type Silicon

At this point, we decided to try producing contacts of aluminum on the gold-doped P-type silicon. Aluminum is a P-type dopant, hence if it is plated on the surface of P-type silicon and alloyed into that surface there should be a gradation from metallic conduction to strong P\(^+\) to the P level of the bulk of the semiconductor. The presence of the strong P\(^+\) -type area between the metal and the semiconductor proper prevents the injection of carriers when the junction is biased. There is not a barrier preventing the flow of charges from the semiconductor to the metal. Such a contact is essentially ohmic for all intensive purposes. Furthermore the resistance of such a contact should be small compared to the resistance of the gold-doped silicon. It should have little effect upon the total resistance of a thermistor unit.

Several contacts of aluminum were made to both ordinary P-type silicon and gold-doped P-type silicon. These contacts were studied to see if they were ohmic and suitable for use in thermistors. After many difficulties a research process was worked out for making aluminum to silicon contacts.

In preparing the silicon wafers for aluminum deposition, they are cut on a precision diamond saw and hand lapped with 180 grit silicon carbide paper. Roughness of the lapped surface is important, since fine grit and smooth lapping can result in poor adhesion between the aluminum and silicon surfaces. After lapping, the silicon is washed with D.I. water, wiped with acetone, washed with alcohol, and blown dry with air. It is very important that no dust gather on the silicon since any dust particle will result in an area of poor deposition on the final wafer.

A small quantity of reagent-grade aluminum turnings is placed in a crucible of densified graphite approximately 1 cm in diameter. This crucible is placed in the bottom of a 2 x 2 inch round bottom quartz crucible. A holder for the silicon wafer is mounted at the top of the crucible in notches cut for that purpose. (See Figure 4). A second crucible is inverted over the entire assembly and is placed on an RF coil of a Kinney vacuum float-zone refiner. When the vacuum in the Kinney reaches approximately 0.2 x 10\(^{-4}\) mm Hg the RF power is turned on and warming of the densified graphite crucible is begun. In our
case, power is maintained on the 10 kw, 2-5 megacycle RF generator at approximately 35% of full on the variac control. The aluminum melts and begins to evaporate inside the crucibles. Initially one can see the heat glow of the graphite crucible and the silicon wafer above it. When you can no longer see the crucible or silicon wafer the aluminum deposition is complete and the heat is turned off. The system is allowed to cool. Too much heat applied too rapidly can

ALUMINUM DEPOSITION APPARATUS

FIGURE 4

Approximately to Scale
result in a flash evaporation and a poorly deposited aluminum surface. It is desirable not to have a thick aluminum deposition.

After cooling, the wafer is taken from the vacuum chamber and handled very carefully. In this condition the aluminum plating is easily marred or damaged. We have found that a uniform white silver deposition is indicative of a good aluminum plating. A brownish-looking surface indicates that not enough aluminum has been evaporated, or that too much power was applied, so that aluminum has already alloyed into the silicone surface.

The next step is alloying the aluminum into the silicon surface creating the high P⁺ area. This is accomplished by heating the wafer under a helium ambient for 20 minutes at 580-630°C as read on a thermocouple mounted in a quartz tube near the wafer. After 20 minutes, the wafer is pulled to the end of the furnace and allowed to cool under the helium flow for a period of about 20 minutes. This is done to avoid oxidation of the aluminum surface at elevated temperatures. When the silicon wafer is taken from the furnace the alloyed surfaces should not be touched. A brownish-looking surface indicates that alloying has occurred. A white-silver appearance indicates that there has been no (or poor) alloying and the bond will probably be poor.

The next step is to nickel-plate the aluminum-alloyed surface by the electrodeless technique. It is essential to keep this plating thin. Plating aluminum-alloyed silicon by the electrodeless technique is somewhat slower than when plating silicon by the electrodeless technique. This plating can take 5 minutes at 96°C. Usually by the time you can observe nickel being plated on the aluminum surface, there is sufficient nickel-plating.

The wafers can then be cut into parallelepiped-type thermistors using the precision diamond saw, or cylindrical shapes using ultrasonic dicing techniques. Leads are soldered to the nickel-plated aluminum surfaces, using as little heat as possible.

The aluminum silicon contacts were tested for temperature-resistance behavior. First, aluminum contacts on ordinary P-type silicon were tested. Graph 13 shows the results of aluminum contacts to 19 ohm cm P-type silicon and 13 ohm cm P-type silicon. The resistances plotted are the total element resistances. In both cases, the resistances at 25°C can be accounted for solely by the bulk resistivity of the silicon. This means that the contact resistances are very small and negligible. The temperature-resistance curves are characteristic of ordinary P-type silicon.

Comparison was made between an aluminum contact and a similar nickel contact. Potentiometric measurements were made on thermistors made of similar gold-doped P-type silicon. In one case the contacts were aluminum, and in the other case the contacts were nickel. Measurements were made between one lead and a point approximately 1.5 mm from the contact.
Graph 13

Aluminum Contacts on P-Type Silicon

Resistance: Ohms x 10²

10,000,000

100,000

10,000

1,000

100

10

-85°C -75°C -60°C -35°C 0°C 25°C 65°C 125°C 200°C

\frac{1}{°C + 273}

- 30 -
Measurements were also made from lead to lead.

In the case of the aluminum contact, the potentiometric measurement between the lead and interior point (1-2) accounts for approximately 30% of the total resistivity of the element. In the case of the nickel contact, a similar measurement accounts for approximately 40% of the total resistivity of the element. These data indicate several things. First, the aluminum contact resistance is very small and when added to even a smaller section of gold-doped silicon, does not appreciably affect the total resistivity or temperature-resistance behavior. The nickel contact as mentioned previously can account for appreciable portion of the resistance of a thermistor. However, its temperature-resistance behavior is very similar to that of gold-doped silicon. In fact, adding this resistance to the resistance of a portion of gold-doped silicon does not affect the slope of the temperature-resistance curve to any appreciable extent. The nickel P-type silicon contact is undesirable from the standpoint of its behavior under applied voltage as discussed previously. There was little doubt that an alloyed aluminum contact to gold-doped P-type silicon would be very desirable for thermistor applications.

**Contacts on Gold-Doped N-Type Silicon**

Initially, electrodeless plated nickel was used as a contact for N-type gold-doped thermistors. While nickel is probably a donor and should produce an ohmic contact, the samples showed considerable drift when annealed at 210°C.
Fearing that the thermistors may have been contaminated while soldering the leads, a solution of nickelous chloride was used instead of the commercially available soldering flux. Annealing and remeasuring the samples gave the following results:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Flux</th>
<th>0 hrs.</th>
<th>672 hrs.</th>
<th>840 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1308</td>
<td>Ordinary</td>
<td>97,000</td>
<td>117,600</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Paste Flux</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1309</td>
<td>NiCl₂</td>
<td>101,400</td>
<td>114,600</td>
<td></td>
</tr>
<tr>
<td>1310</td>
<td>NiCl₂</td>
<td>313,900</td>
<td>303,100</td>
<td></td>
</tr>
<tr>
<td>1311</td>
<td>NiCl₂</td>
<td>45,940</td>
<td>45,490</td>
<td></td>
</tr>
<tr>
<td>1312</td>
<td>NiCl₂</td>
<td>247,100</td>
<td>251,500</td>
<td></td>
</tr>
<tr>
<td>1313</td>
<td>NiCl₂</td>
<td>110,300</td>
<td>114,600</td>
<td></td>
</tr>
</tbody>
</table>

Although there was some improvement in stability it was far from satisfactory.

Gold, gold-antimony alloy and antimony sub-layers were used as a "cushion" between the nickel and the silicon to improve the ohmicity of the contacts. The general technique of applying these materials is to evaporate under vacuum and deposit directly on the silicon wafers. The wafers are then heated, sintered, or alloyed at elevated temperatures. After etching, usually with concentrated HF, the wafers are plated with electrodeless nickel. The wafers are diced and leads are attached before the final check of resistance and annealing at 210°C.

Sub-layers of gold, when applied in the described manner, showed an increase in resistivity after annealing, probably because the gold contact was changing the concentration of gold in the gold-doped silicon.

Sub-layers of gold-antimony alloy were deposited using the Kinney unit. In fact that gold has a much higher melting point than the antimony made the deposition difficult to control. The final deposited alloy on the wafer had a gold concentration that was a function of evaporating time while the amount of antimony was practically constant. The following table gives the results of two typical samples:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>328 hrs.</th>
<th>852 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1314</td>
<td>55,920</td>
<td>55,750</td>
<td>Broken Contact</td>
</tr>
<tr>
<td>1315</td>
<td>54,520</td>
<td>54,590</td>
<td>55,540</td>
</tr>
</tbody>
</table>

- 32 -
Sub-layers of antimony were deposited on three wafers using the Kinney unit and then alloyed at 500°C, 800°C and 1200°C. After etching and nickel plating, leads were attached. Results after annealing for various lengths of time at 210°C are given below.

### TABLE 9

<table>
<thead>
<tr>
<th>Alloying Temperature</th>
<th>0 hrs.</th>
<th>17 hrs.</th>
<th>37 hrs.</th>
<th>87 hrs.</th>
<th>104 hrs.</th>
<th>222 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>500°C</td>
<td>1662</td>
<td>1675</td>
<td>1698</td>
<td>-</td>
<td>Contact</td>
<td>Broken</td>
</tr>
<tr>
<td>800°C</td>
<td>1735</td>
<td>1760</td>
<td>1798</td>
<td>-</td>
<td>1962</td>
<td>Contact</td>
</tr>
<tr>
<td>1200°C</td>
<td>1240</td>
<td>1258</td>
<td>-</td>
<td>1262</td>
<td>-</td>
<td>1264</td>
</tr>
</tbody>
</table>

The sample which was alloyed at 1200°C appears to be stabilizing after an initial 2% change in resistance. The continuous rise in resistance until the contact breaks was typical of weak mechanical contacts due probably to a layer of non-alloyed antimony between the nickel and the antimony-silicon alloy.

The results obtained were encouraging enough, however, to make some more experiments. Below are some of the results obtained by alloying antimony-deposited gold-doped N-type silicon wafers. 0.005g of antimony was deposited per seven wafer sides.

### TABLE 10

<table>
<thead>
<tr>
<th>Wafer No.</th>
<th>Alloying Temperature</th>
<th>0 hrs.</th>
<th>50 hrs.</th>
<th>104 hrs.</th>
<th>1050 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>1200°C</td>
<td>8,981</td>
<td>8,892</td>
<td>8,769</td>
<td>8,841</td>
</tr>
<tr>
<td>5</td>
<td>1000°C</td>
<td>10,376</td>
<td>8,889</td>
<td>8,584</td>
<td>8,535</td>
</tr>
<tr>
<td>6</td>
<td>1000°C</td>
<td>11,080</td>
<td>9,335</td>
<td>8,953</td>
<td>8,748</td>
</tr>
<tr>
<td>7</td>
<td>1000°C</td>
<td>10,934</td>
<td>9,238</td>
<td>8,827</td>
<td>8,653</td>
</tr>
<tr>
<td>8</td>
<td>1100°C</td>
<td>13,123</td>
<td>10,505</td>
<td>10,530</td>
<td>10,845</td>
</tr>
<tr>
<td>9</td>
<td>1100°C</td>
<td>12,630</td>
<td>12,117</td>
<td>12,214</td>
<td>12,235</td>
</tr>
<tr>
<td>10</td>
<td>1200°C</td>
<td>13,130</td>
<td>12,565</td>
<td>12,634</td>
<td>12,630</td>
</tr>
</tbody>
</table>

The lack of uniformity of these three sets of wafers made under the same conditions resulted in a closer examination of the technique of nickel plating N-type silicon. We assumed now that the lack of reproducibility was due to lack of uniformity, contamination or oxidation of the surface of the wafers, or some other fault of the nickel.
plating process.

A number of quick tests were performed on every step of the nickel plating process. The results were very interesting. It was found that a greasy fingerprint on the wafer before or after lapping could show as a spot difficult to nickel plate or as a well defined fingerprint after the wafer had been nickel plated. Two degreasing cycles were added to avoid this effect.

It was also noted that two wafers seldom nickel plated in the same way. We reached the conclusion that a strong etch just prior to the nickel plating operation was necessary. Etching the wafer with a mixture of HF and HNO₃ left a too-smooth surface for good nickel adhesion. Concentrated HF gave a fair bond, but not good enough. Sodium hydroxide gave a very good cleaning to the wafer. The small amount left on the wafer increased the chemical activity of the nickel plating solution yielding a nickel deposition so thick that it would peel off. Boiling ammonium hydroxide seemed to clean the surface of the wafer in a gentle and uniform way. Very small bubbles on the surface of the wafer indicate that the wafer is ready for the nickel plating solution.

A strength test for the adhesion of the nickel plated film to the silicon was made soldering leads of 28 or 30 gauge copper wire to 2.2 x 2.2mm thermistors and pulling the wires apart. The 2.2 x 2.2mm contacts were considered to be good only if the wire would break before the contacts did.

While in production two more sets of wafers were found to be defective. The failure was traced in one set to the Teflon-covered forceps used to handle the wafers. The Teflon had peeled off and the exposed metal was reacting with the solutions or the silicon. In the other set, the wafers had been exposed to the atmosphere for a short period prior to nickel plating and the surface was covered with a film of oxide.

For testing the new nickel plating technique we selected two gold-doped silicon crystals: 1177, initially 5 ohm cm N-type; and 3253A, initially 8 ohm cm N-type. Wafers 1.2mm thick were cut and measured:

<table>
<thead>
<tr>
<th>Wafer No.</th>
<th>Contacts</th>
<th>0 hrs.</th>
<th>41 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1177-W1</td>
<td>Ni-plating only</td>
<td>1502</td>
<td>1501</td>
</tr>
<tr>
<td>1177-W2</td>
<td>Ni-plating only</td>
<td>1531</td>
<td>1536</td>
</tr>
<tr>
<td>3253A-W1</td>
<td>Ni-plating only</td>
<td>4842</td>
<td>4885</td>
</tr>
<tr>
<td>3253A-W2</td>
<td>Ni-plating only</td>
<td>4870</td>
<td>4850</td>
</tr>
</tbody>
</table>

- 34 -
In most cases the changes upon heating are less than 1%. Wafers 3253 A-W1 and 3253A-W2 were cut into 2 x 2mm thermistors. Results of resistance measurements are given below:

**TABLE 12**

<table>
<thead>
<tr>
<th>Thermistor No.</th>
<th>17 hrs. (Ohms)</th>
<th>17 hrs. (Ohms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3253A-W1-1</td>
<td>460,000</td>
<td>460,000</td>
</tr>
<tr>
<td>W1-2</td>
<td>441,000</td>
<td>447,000</td>
</tr>
<tr>
<td>-3</td>
<td>448,000</td>
<td>443,000</td>
</tr>
<tr>
<td>-4</td>
<td>448,000</td>
<td>449,000</td>
</tr>
<tr>
<td>-5</td>
<td>450,000</td>
<td>450,000</td>
</tr>
<tr>
<td>-6</td>
<td>460,000</td>
<td>462,000</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Thermistor No.</th>
<th>17 hrs. (Ohms)</th>
<th>17 hrs. (Ohms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3253A-W2-2</td>
<td>457,000</td>
<td>461,000</td>
</tr>
<tr>
<td>W2-3</td>
<td>463,000</td>
<td>464,000</td>
</tr>
<tr>
<td>-4</td>
<td>452,000</td>
<td>454,000</td>
</tr>
<tr>
<td>-5</td>
<td>460,000</td>
<td>462,000</td>
</tr>
<tr>
<td>-6</td>
<td>478,000</td>
<td>475,000</td>
</tr>
</tbody>
</table>

The distribution of resistances is rather narrow. This is an indication of good stability. Changes upon heating are also relatively small.

Further measurements before and after the thermistors had been adjusted to 500K showed a continuous downward drift. The problem was traced to leakage on the edges of the thermistors. It seems that they were contaminated with some low conductivity material (i.e. finger oil).

A good washing of the thermistors with trichloroethylene followed by acetone and alcohol restored the resistance to the original value. For example: thermistor W2-39 had an original resistance of 475K. After 167 hours of annealing, the resistance fell to 450K. At 328 hours the edges were cleaned and the resistance was up to 473K - practically the same original value.

The case of the adjusted thermistors was similar.

**TABLE 13**

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Measurement Before Cleaning</th>
<th>Measurement After Cleaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>W-L-33</td>
<td>466K</td>
<td>495K</td>
</tr>
<tr>
<td>W-L-34</td>
<td>418K</td>
<td>499K</td>
</tr>
<tr>
<td>W-L-35</td>
<td>440K</td>
<td>492K</td>
</tr>
<tr>
<td>W-L-36</td>
<td>406K</td>
<td>505K</td>
</tr>
</tbody>
</table>

- 35 -
This chart shows clearly the effect of the leakage on the edges of the thermistors and the way to eliminate it. The following formula gives the value of the leakage as a function of the resistance of the thermistor.

\[ R_e = R_t (100-e) \]

where:

- \( R_e \) - is the leakage or resistance of the edge.
- \( R_t \) - is the resistance of the thermistor.
- \( e \) - is the value of the drift in percent.

For example:

A thermistor of 500K \((R_t)\) went down to 450K or 10% \((e)\), substituting, we have that \( R_e = 500K \times (100 - 10) = 500K \times (90) \)

\[ R_e = 40 \text{ Meg}. \]

After the nickel plating technique had been improved, we used the experience to prepare four more wafers with antimony sub-layers etching to take off the excess Sb. Ammonium hydroxide was used as an etch just prior to depositing the nickel film. Results of resistance measurements after a number of hours annealing at 200°C were as follows:

<table>
<thead>
<tr>
<th>Wafer No.</th>
<th>Contacts</th>
<th>0 hrs.</th>
<th>24 hrs.</th>
<th>386 hrs.</th>
<th>457 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>3253A-W3</td>
<td>Sb sublayer alloyed at 850°C for 5 min.</td>
<td>7305</td>
<td>7268</td>
<td>7304</td>
<td>7306</td>
</tr>
<tr>
<td>3253A-W4</td>
<td>Sb sublayer alloyed at 850°C for 5 min.</td>
<td>7385</td>
<td>7359</td>
<td>7345</td>
<td>7345</td>
</tr>
<tr>
<td>3253A-W5</td>
<td>Sb sublayer alloyed at 1000°C for 5 min.</td>
<td>5655</td>
<td>5649</td>
<td>5688</td>
<td>5690</td>
</tr>
<tr>
<td>3253A-W6</td>
<td>Sb sublayer alloyed at 1000°C for 5 min.</td>
<td>5586</td>
<td>5588</td>
<td>5641</td>
<td>5641</td>
</tr>
</tbody>
</table>

It appears that the technique of alloying Sb sub-layers to the silicon wafers can be used to obtain 200°C stability. Also, there is the possibility that the alloying temperature may be used to control the final resistivity of the gold-doped silicon.
At this point after several months of doubtful results, we felt that we had not one, but two solutions to the problem. For production purposes, just nickel plated wafers were used because the technique is simpler and there is no change in resistivity. More experiments would be necessary to find the exact relationship between alloying temperature and final resistivity of the wafer with Sb sub-layers. It was our opinion that either technique could satisfactorily meet the contract requirements, both mechanically and electrically. However, as we previously pointed out, the operators must handle the thermistors with extreme care or contamination with odd effects will follow.
5. **Slope and Resistivity Vs. Starting P-Type Resistivity**

A study was completed to determine the dependence of slope and resistivity upon P-type resistivity of the starting silicon. All rods used in this study were 7/8-1 in diameter and were float-zone leveled with 0.15 grams of high-purity gold. The results of this study are given in the table.

<table>
<thead>
<tr>
<th>Average Starting P-type Resistivity at 25°C (ohm cm)</th>
<th>Average Slope ΔE (ev)</th>
<th>Resistivity after doping at 25°C (ohm cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9.5</td>
<td>0.3273</td>
<td>246</td>
</tr>
<tr>
<td>19</td>
<td>0.3275</td>
<td>495</td>
</tr>
<tr>
<td>22</td>
<td>0.3263</td>
<td>522</td>
</tr>
<tr>
<td>25</td>
<td>0.3267</td>
<td>915</td>
</tr>
<tr>
<td>45</td>
<td>0.3264</td>
<td>1110</td>
</tr>
</tbody>
</table>

There is no appreciable change in slope, all values falling within .0012 ev. There is a definite increase in resistivity of the thermistor material as the resistivity of the starting silicon is increased. This relationship was useful for production purposes. For instance, the resistance of a unit not only was controllable by size but also by the resistivity of the thermistor material being used.
6. Annealing Experiments

We conducted annealing experiments at elevated temperatures to determine whether the thermistor elements could be stabilized by this technique. The temperature-resistance response of samples was measured after heating for various lengths of time at 210°C and higher temperatures. Data for a typical series are given below.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>93 hrs.</th>
<th>189 hrs.</th>
<th>351 hrs.</th>
<th>470 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1178</td>
<td>305</td>
<td>293</td>
<td>292</td>
<td>289</td>
<td>288</td>
</tr>
<tr>
<td>1180</td>
<td>305</td>
<td>284</td>
<td>284</td>
<td>282</td>
<td>280</td>
</tr>
<tr>
<td>1182</td>
<td>345</td>
<td>308</td>
<td>305</td>
<td>301</td>
<td>305</td>
</tr>
<tr>
<td>1184</td>
<td>287</td>
<td>283</td>
<td>282</td>
<td>282</td>
<td>282</td>
</tr>
</tbody>
</table>

**Average Slope After - Hrs. at 210°C (ev)**

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>93 hrs.</th>
<th>189 hrs.</th>
<th>351 hrs.</th>
<th>470 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1178</td>
<td>0.3269</td>
<td>0.3257</td>
<td>0.3266</td>
<td>0.3271</td>
<td>0.3266</td>
</tr>
<tr>
<td>1180</td>
<td>0.3268</td>
<td>0.3255</td>
<td>0.3258</td>
<td>0.3259</td>
<td>0.3259</td>
</tr>
<tr>
<td>1182</td>
<td>0.3283</td>
<td>0.3276</td>
<td>0.3274</td>
<td>0.3277</td>
<td>0.3272</td>
</tr>
<tr>
<td>1184</td>
<td>0.3269</td>
<td>0.3268</td>
<td>0.3270</td>
<td>0.3268</td>
<td>0.3264</td>
</tr>
</tbody>
</table>

There does not seem to be any appreciable change in slope. The resistance does tend to level off after several hundred hours at 210°C. Hundred-hour differences here frequently result in less than 1% change in resistance.

A sample such as 1182 is to be viewed with suspicion. The decreasing resistance followed by an increase usually indicates a poor contact that is beginning to separate. Take for example, the following series:

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>162 hrs.</th>
<th>324 hrs.</th>
<th>464 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1238</td>
<td>599</td>
<td>412</td>
<td>414</td>
<td>446</td>
</tr>
<tr>
<td>1239</td>
<td>627</td>
<td>494</td>
<td>479</td>
<td>599</td>
</tr>
<tr>
<td>1240</td>
<td>400</td>
<td>342</td>
<td>321</td>
<td>321</td>
</tr>
<tr>
<td>1241</td>
<td>386</td>
<td>332</td>
<td>332</td>
<td>334</td>
</tr>
<tr>
<td>1242</td>
<td>612</td>
<td>489</td>
<td>534</td>
<td>C.B.</td>
</tr>
<tr>
<td>1243</td>
<td>601</td>
<td>600</td>
<td>621</td>
<td>C.B.</td>
</tr>
</tbody>
</table>

C.B. - Contact Broken
The contacts on these samples were generally poor. In almost every example, the resistance decreases and increases. Contacts actually broke in two cases after 464 hours and in the other cases were separating. The average slopes are not very uniform and frequently are less than the 0.3250 to 0.3280 normally found in most cases for gold-doped P-type silicon. This would be the case if a non-thermally sensitive contact resistance was being added to the resistance of a device.

Some samples show little change upon annealing as in the following series:

**TABLE 18**

Resistance After - Hrs. at 210°C (ohms)

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>95 hrs.</th>
<th>216 hrs.</th>
<th>428 hrs.</th>
<th>738 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1265</td>
<td>562</td>
<td>561</td>
<td>561</td>
<td>561</td>
<td>558</td>
</tr>
<tr>
<td>1266</td>
<td>556</td>
<td>556</td>
<td>557</td>
<td>554</td>
<td>554</td>
</tr>
<tr>
<td>1267</td>
<td>547</td>
<td>539</td>
<td>539</td>
<td>538</td>
<td>536</td>
</tr>
<tr>
<td>1268</td>
<td>518</td>
<td>514</td>
<td>516</td>
<td>514</td>
<td>516</td>
</tr>
</tbody>
</table>

Average Slope After - Hrs. at 210°C (ev)

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>95 hrs.</th>
<th>216 hrs.</th>
<th>428 hrs.</th>
<th>738 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1265</td>
<td>0.3262</td>
<td>0.3266</td>
<td>0.3270</td>
<td>0.3266</td>
<td>0.3265</td>
</tr>
<tr>
<td>1266</td>
<td>0.3263</td>
<td>0.3269</td>
<td>0.3260</td>
<td>0.3268</td>
<td>0.3268</td>
</tr>
<tr>
<td>1267</td>
<td>0.3263</td>
<td>0.3265</td>
<td>0.3258</td>
<td>0.3261</td>
<td>0.3256</td>
</tr>
<tr>
<td>1268</td>
<td>0.3268</td>
<td>0.3264</td>
<td>0.3249</td>
<td>0.3259</td>
<td>0.3257</td>
</tr>
</tbody>
</table>

After more than 95 hours at 210°C the changes in resistance are less than 1%. Changes in slope are not appreciable. Several other follow-up series also showed good results in stability and uniformity. The following series of thermistors was cut from the same wafer and shows good uniformity and initial thermal stability.
Only sample 1335 shows evidence of instability. This is indicated by an increase in resistance after 32 hours and a decrease in slope. The increase in resistance is indicative of a contact which is beginning to separate. In P-type silicon the value of the slope itself is indicative of a good ohmic contact. In samples with stable contacts the average slope will normally run between 0.3250 and 0.3280 ev. When the slope of a thermistor falls below these limits, as in the case of 1335, it is to be viewed with suspicion as far as stability is concerned.

The importance of a good contact cannot be overstressed. Frequently it is the samples which show the greatest decreases in resistance upon the initial heating that later increase and result in separated contacts.

Annealing experiments were also conducted at higher temperatures. Leadless samples were annealed at 300°C, 400°C, and 500°C in air and helium. At 400 and 500°C the contacts become discolored, brittle, and difficult to solder. They also tend to separate. The following series was annealed at 300°C and then at 210°C to see if changes would occur at the lower temperature.

### Table 19

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>0 hrs.</th>
<th>72 hrs.</th>
<th>0 hrs.</th>
<th>72 hrs.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1346</td>
<td>635</td>
<td>635</td>
<td>0.3263</td>
<td>0.3268</td>
</tr>
<tr>
<td>1387</td>
<td>604</td>
<td>603</td>
<td>0.3264</td>
<td>0.3270</td>
</tr>
<tr>
<td>1328</td>
<td>610</td>
<td>610</td>
<td>0.3259</td>
<td>0.3267</td>
</tr>
<tr>
<td>1329</td>
<td>611</td>
<td>612</td>
<td>0.3260</td>
<td>0.3266</td>
</tr>
<tr>
<td>1330</td>
<td>631</td>
<td>632</td>
<td>0.3249</td>
<td>0.3258</td>
</tr>
<tr>
<td>1331</td>
<td>614</td>
<td>613</td>
<td>0.3255</td>
<td>0.3272</td>
</tr>
<tr>
<td>1332</td>
<td>606</td>
<td>605</td>
<td>0.3264</td>
<td>0.3271</td>
</tr>
<tr>
<td>1333</td>
<td>610</td>
<td>609</td>
<td>0.3261</td>
<td>0.3270</td>
</tr>
<tr>
<td>1334</td>
<td>608</td>
<td>610</td>
<td>0.3261</td>
<td>0.3268</td>
</tr>
<tr>
<td>1335</td>
<td>680</td>
<td>704</td>
<td>0.3258</td>
<td>0.3246</td>
</tr>
<tr>
<td>1336</td>
<td>610</td>
<td>612</td>
<td>0.3260</td>
<td>0.3273</td>
</tr>
<tr>
<td>Sample No.</td>
<td>Resistance After Hrs at 210°C (ohms)</td>
<td>Slope After Hrs at 210°C (ev)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>------------</td>
<td>-------------------------------------</td>
<td>-----------------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>300°C for 78 hrs (ohms)</td>
<td>300°C for 78 hrs (ev)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>145 hrs.</td>
<td>290 hrs.</td>
<td>453 hrs.</td>
<td>145 hrs.</td>
</tr>
<tr>
<td>1258</td>
<td>1229</td>
<td>1232</td>
<td>1230</td>
<td>1228</td>
</tr>
<tr>
<td>1259</td>
<td>1330</td>
<td>1340</td>
<td>1347</td>
<td>1357</td>
</tr>
<tr>
<td>1260</td>
<td>1284</td>
<td>1340</td>
<td>1287</td>
<td>1370</td>
</tr>
<tr>
<td>1261</td>
<td>1365</td>
<td>1367</td>
<td>1366</td>
<td>1370</td>
</tr>
</tbody>
</table>

Although there is evidence of stabilization, there is also evidence of contacts separating. It appears desirable to anneal at lower temperatures with the leads soldered on. If this is true, it would restrict annealing to temperatures below the melting point of the tin solder, 232°C.
7. **Sizing of Thermistors**

We felt that final size adjustment might be accomplished by grinding, vapor-blasting, or chemical-etching techniques. The purpose of this adjustment is to obtain a given value of resistance at 25°C with an error of not over ±2%. The thermistor dice would be cut larger in size than actually necessary and material would be removed by one of the three techniques mentioned. The material removed would be continued until a resistance reading indicated that the thermistor was at the proper resistance level. This adjustment should not affect the temperature sensitivity or temperature-resistance ratios. So far, we have tried two of the techniques mentioned: vapor blasting and grinding.

The vapor-blasting unit tried was a S.S. White industrial air-abrasive unit. Cutting is effected by a high-speed stream of abrasive alumina particles, which are propelled by a gas such as CO₂ or nitrogen. This unit is effective for cutting the silicon; however, the propellant gas tends to cool the silicon during the cutting. This prevents any measurement of a resistance during the actual abrading operation, since the resistance is varying with the temperature of the thermistor. For these reasons, we have decided against vapor blasting as a technique for size adjustment of thermistors.

The other technique of size adjustment tried was that of grinding with a high-speed dentist-type drill and a diamond bit. The drill is driven by a flexible cable and has a top speed of about 12,000 rpm. A series of 4 x 4 x 1 mm thermistors was adjusted toward 525 ohms at 0°C. The samples were embedded in paraffin wax and ground under ice water until the resistance as read on a vacuum tube voltmeter reached approximately 525 ohms. Wheatstone bridge measurements on the thermistors were:

- 530 ohms 514 ohms 517 ohms
- 519 ohms 535 ohms 530 ohms
- 526 ohms 528 ohms 534 ohms

All, except possibly one, were within the ±2%. Another series was adjusted to 565 ohms at 0°C and read 564, 567, and 571 ohms. Since the grinding is done under ice water, measurements of resistance can be made during the grinding operation. If a slight elevation in temperature occurs during the grinding, the thermistors reach 0°C immediately upon removal of the grinding tool. Smaller sizes, of course, would be more difficult to adjust.

Size adjustment by high-speed diamond grinding was selected as the pilot line production technique. Pilot line experience proved that thermistors on the order of 2 x 2 x 1 mm could be easily adjusted to within 1% of a standard thermistor. In the pilot line set-up the thermistor being adjusted is compared to a standard by the ratio technique described earlier. The standard thermistor is mounted in the same silicone oil bath as the thermistor being adjusted. The P-type contract samples were adjusted.
at room temperature. The N-type contract samples were adjusted at about 100°C. Adjusting by the ratio technique seems to proceed better at resistances below 10,000 ohms.

Chemical etching has not been tried as a possible technique for material removal because we have not been able to select a suitable etching solution or technique.
8. Manufacturing Process for Gold-Doped P-Type Silicon

A 28-step manufacturing process was developed for producing gold-doped P-type silicon thermistors. The unbalanced pilot line was operated according to this process while producing the 400 contract samples of P-type thermistors. The 28 steps are outlined below:

1. Determine necessary starting silicon resistivity.
2. Purchase monocrystalline rod from a commercial supplier, or prepare the same.
3. Check resistivity profile with 4-point probe.
4. Prepare rod for zone leveling: Etch 1:3 HF/HNO₃, water, dry.
5. Zone level with high-purity gold.
6. Cut three wafers from seed end, chuck end, and center.
7. Prepare three wafer-size thermistors from these slices and measure resistance at 25°C.
8. Calculate desired thickness of production wafers based on resistivity of the three trial wafers and dicing tool sizes.
9. Cut remaining zone-leveled rod into wafers.
10. Wash with alcohol, acetone, alcohol.
11. Lap wafers with 180-C silicon carbide paper.
12. Wash with water, wipe with paper to remove fine particles.
13. Wash successively with acetone, alcohol, water, HF, water, alcohol.
15. Alloy aluminum to silicon at 580-630°C.
16. Etch wafers with concentrated HF-water.
17. Wipe with paper, wash with water, HF, water.
18. Immerse wafers briefly in boiling ammonium hydroxide.
19. Nickel plate wafers by electrodeless technique at 96°C.
20. Heat treat at 210°C overnight in argon or nitrogen.
21. Recheck resistivity of several wafers with pressure contacts.

22. Determine final dice size.

23. Dice wafers.

24. Solder leads to dice with tin at 240°C-250°C.

25. Wash with methyl alcohol.


27. Determine final temperature-resistance behavior at a minimum of three temperatures.

28. Paint and bake at 210°C.

Each step is discussed in more detail below:

Step 1 - Determine the necessary starting silicon resistivity.

The resistance of a thermistor will follow the equation,

\[ R = \frac{\rho L}{A} \]  

Equation No. 1

\( R \) = resistance of thermistor in ohms

\( \rho \) = resistivity of thermistor material in ohm cm

\( L \) = length of the thermistor between electrodes in cm

\( A \) = the cross sectional area of the thermistor in cm².

If we know the resistance and approximate size of a desired thermistor, it is possible to calculate the resistivity of the gold-doped silicon to be used in the device by this equation. Then using the relationship shown in Graph 14, it is possible to determine the necessary starting silicon resistivity. These curves were determined experimentally by doping various resistivities of P-type silicon with 0.15 grams of high-purity gold in the zone-leveling process. The rod diameters were 7/8 to 1", the estimated weight of the molten zone was 20 grams. 0.15 grams of gold was found to be the maximum amount that could be added to such a zone without causing polycrystallinity problems.

Step 2 - Purchase monocrystalline rod from a commercial supplier, or prepare the same.

The silicon being used in this process can be purchased from any one of a number of commercial suppliers. Presently, we are purchasing single-crystal silicon from Merck & Co. in Rahway, New Jersey. We have been
Graph 1.4

Initial Resistivity - Ohm cm P-Type
purchasing material within ±25% of a given resistivity value. Material of ±10% resistivity variance can be obtained at a higher price. However, we have found that there are enough process-control variables to allow the use of the ±25% material. Of particular importance is the size adjustment which allows the up-grading of the resistance of any thermistor to a higher value.

Step 3 - Check resistivity profile with 4-point probe.

This is a standard 4-point probe measurement, commonly used for measuring silicon under 1000 ohm cm. This step could be eliminated once there is good agreement between supplier and purchaser on resistivity levels.

Step 4 - Prepare rod for zone leveling: Etch 1 to 3 HF/HNO₃, dry.

The rod is etched in a mixture of one volume of concentrated (49%) HF to 3 volumes of concentrated (70%) nitric acid until the surface is clean and shining. It is then rinsed thoroughly in distilled, deionized water of one megohm cm or greater resistivity. Droplets of water are removed from the rod by blowing with high-purity argon. The rod is further dried under an infrared lamp on a thoroughly clean quartz pedestal.

Step 5 - Zone level with high-purity gold.

This is a standard zone-refining procedure in which high-purity gold is used as a dope between the seed and the rod. The gold is melted into the first zone as the seed and the rod are fused together by means of radio-frequency heating. Proper matching of the seed and rod must be made in the case P-type silicon. The resistivity of the seed should be approximately the same as that of the rod.

The zone-leveling pass is made at the rate of about 8 inches per hour. If the rod does not go single on the first pass, it should be re-doped before the second pass in order to obtain the same amount of residual gold.

Step 6 - Cut three wafers from seed end, chuck end, and center.

After zone leveling, the resistance of the gold-doped silicon is normally so high that the resistivity of the material cannot be checked easily by any simple 4-point or 2-point probe technique. We check the resistance of a rod at the various points mentioned by cutting wafers and fabricating large area devices from them. Because the area is large, the resistance of the device will tend to be low even for large resistivities. The thickness of the wafer is determined on the basis of the anticipated resistivity and the resistance of the devices to be made. The crystal is cemented to a ceramic block with jewelers' wax. After being cut, it is left oriented on the high-speed diamond saw. Further cuts can be made parallel to those already made.
Step 7 - Prepare three wafer-size thermistors from these slices and measure the resistance at 25°C.

The wafers are aluminum deposited, alloyed, etched, and nickel-plated. Pressure contacts are made. The resistance of these wafers is then measured with a Wheatstone bridge in a 25 ± 0.01°C bath. From the resistance and the physical dimensions of the wafers, the resistivity of the silicon is calculated according to Equation No. 1.

Step 8 - Calculate the desired thickness of production wafers based on resistivity of three trial wafers and dicing tool sizes.

This calculation is made again using Equation No. 1. Normally, there will be only certain dicing tool sizes available. Presently, for instance, we are using dicing tools 2-1/2, 3, and 3-1/2mm in diameter. More sizes could be carried for dicing purposes. Normally, the thickness of the production wafers is calculated to be about 10% less than that defined in Equation No. 1. This means that the resistance of the devices will be less than the desired value. However, later in the size-adjustment step, the resistance of the devices can be up-graded to higher values. It is desirable to have the peak of the product-resistance distribution below the desired-resistance value so that only a small portion or none of the devices fall above the desired value.

Step 9 - Cut remaining zone-leveled rod into wafers.

The zone-leveled rod which was left oriented on the diamond saw is then cut into wafers of the thickness calculated in Step 8.

Step 10 - Wash with alcohol, acetone, alcohol.

This step is to remove any traces of jewelers wax, oil or other impurities which might be present as a result of diamond sawing.

Step 11 - Lap wafers with 180-C silicon carbide paper.

The wafers are lapped by hand under a metal weight making strokes in one direction only across the wafer.

Step 12 - Wash with water, wipe with paper to remove fine particles.

This treatment is used primarily to remove impurities, mainly particulate, which have collected on the silicon as a result of lapping. Wiping with paper seems to be necessary to remove all traces of fine matter. Some particles seem to remain even after the water washing.

Step 13 - Wash successively with acetone-methanol-water-conc. HF-water-methanol.

This step is used to prepare the wafer surfaces for aluminum deposition. It removes impurities such as grease, finger oil, oxide
film etc. which might interfere with a good aluminum deposition. Whereas good thermistors might be made by eliminating some of these steps, we feel that the most consistent results will be obtained by using all of them.

Beginning with this step and until the wafers are finally nickel plated, the circular surfaces should not be touched by the fingers or mechanical holders such as forceps. A mark, made by a forcep for instance, can result in poor adhesion of the aluminum or nickel coatings.

Step 14 - Vacuum deposit aluminum on wafers.

In this step a wafer or several wafers are mounted in a vacuum chamber above a densified graphite crucible containing high purity aluminum. The chamber is pumped down until the vacuum is below 8.5 x 10^-5mm Hg. The aluminum is flashed by means of a R-F heating coil around the graphite crucible. Other heating techniques could be used. The initial flash in our equipment lasts for 35 seconds. After a 5-minute cooling period another 35-second flash is made. After cooling the chamber is opened and the wafer or wafers are reversed so that the previously unexposed sides are now exposed. The process is then repeated so that both sides of the wafers are deposited. Normally we deposit 0.3 to 0.5 milligrams of aluminum per side of a wafer which is 7/8" to 1" in diameter. This amounts to 0.4 to 0.8 milligrams per square inch of surface.

Step 15 - Alloy aluminum to silicon at 580 to 630°C.

In this step the aluminum-deposited wafers are put into a homemade quartz-tube furnace under a helium ambient. Argon or nitrogen could probably be used also. The furnace is heated up and the alloying time is taken as the time the temperature remains above 580°C and below 630°C. Normally an alloying time of 20 minutes is used. Any furnace capable of these temperatures and an inert ambient could be used. The wafers are cooled under the helium ambient to prevent oxide formation.

Step 16 - Etch wafers with concentrated HF.

In this step, the aluminum-alloyed wafers, which have been allowed to cool, are etched in concentrated 49% HF. This etching not only removes oxide, which is formed on the aluminum-silicon surface, but also removes any excess aluminum which may be on the surface.

Step 17 - Wipe with paper, wash with water-conc. HF-water.

The wafer is wiped with paper to remove a blackish deposit formed in the previous HF treatment. The second HF treatment does not produce a blackish deposit. Water removes traces of HF and ionic impurities.

After the washings and HF treatments, the wafers are not exposed for any lengthy periods to the atmosphere. Exposure can result in an oxide film and subsequent poor adhesion of the metal platings. The wafers are kept under the solutions and transferred quickly from one to the other. Transfer to the ammonium hydroxide of Step 18 is also rapid.

- 50 -
Step 18 - Immerse wafers briefly in boiling ammonium hydroxide.

The wafers are immersed until the first signs of reaction are noted visually. This is usually a slight bubbling on the surface. They are then transferred rapidly to the nickel-plating solution with little atmospheric exposure.

Step 19 - Nickel plate wafers by electrodeless technique at 96°C.

The wafers are transferred very rapidly from the boiling ammonium hydroxide solution to the nickel-plating solution at about 96°C having the following formulation:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>gms/liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Chloride</td>
<td>30</td>
</tr>
<tr>
<td>Sodium Hypophosphite</td>
<td>10</td>
</tr>
<tr>
<td>Sodium Citrate</td>
<td>65</td>
</tr>
<tr>
<td>Ammonium Chloride</td>
<td>50</td>
</tr>
</tbody>
</table>

Add ammonium hydroxide until the solution turns from green to blue (7 to 8 pH)

The pH is maintained at about 7 during the plating procedure which usually lasts for less than five minutes. During this time, approximately 0.0095 gram of nickel is plated on each wafer.

Step 20 - Heat treat at 210°C overnight in argon or nitrogen.

This step can be done in any conventional oven which can be fitted with an argon or nitrogen supply. It is done primarily to stabilize the resistance of the unit and to bring about slight changes which sometimes occur at the beginning of the annealing period.

Step 21 - Recheck resistivity of several wafers with pressure contacts.

The number of wafers rechecked in this step usually depends upon the amount of resistivity variance that is expected from the measurements made in Step 7. If the measurements there indicated a great variance in resistivity, perhaps every other wafer should be checked. If the measurements indicated a very uniform resistivity, this step could be eliminated. Contacts are made with pressure, usually under a spring-type clamp. No soldered connection is made. We have found that resistivities obtained by this technique check well with those obtained in Step 7.

Step 22 - Determine final dice size.

Again, we rely on Equation No. 1. At this point the resistivity and thickness of the device are fixed and only the area is to be determined. If the dice are to be circular the diameter is to be chosen to coincide with a dicing tool which is available, and as mentioned previously, to yield devices which are on the low side of the desired resistance value.
Step 23 - Dice wafers.

For circular dice we send the plated wafer to Semiconductor Specialties, Corp., Lodi, New Jersey, for ultrasonic dicing. The equipment used for dicing is a Cavitron ultrasonic dicer. This is standard equipment in the semiconductor industry. The dicing could be done here if we purchased the equipment. For a 2-1/2 mm diameter dice size we obtain approximately 50 to 55 dice per wafer. For 3-1/2 mm diameter size we obtain approximately 20-25 dice per wafer.

For square, rectangular or other straight-edge dice, we use the high-speed diamond saw and crosscut the wafers in the proper manner. We have had no indication that shape affects the temperature response of these devices.

Step 24 - Solder leads to dice with tin at 240°C-250°C.

The thermistor dice are mounted in a Teflon jug and tinned copper leads are brought up to the nickel-plated surfaces of the thermistors. Other lead material could be used. Flux is applied to the plated surfaces and jug and thermistor are dipped into a tin pot at 240°C-250°C. The thermistor is held in the tin just long enough for the tin to wet the thermistor and lead. Too long a period of time in the solder can result in a separated contact.

Other solders might be used. We use tin because of an upper operating temperature of 200°C.

Step 25 - Wash with alcohol.

After removal from the solder pot, the thermistors are washed with methyl alcohol to thoroughly remove any remaining traces of flux and to clean the surface.

Step 26 - Adjust size of thermistors by high-speed diamond grinding comparing resistance-to-resistance of a standard thermistor.

Thermistors which are below the desired resistance value and need adjustment are mounted near a standard thermistor of the proper resistance. Both thermistors are submerged below the surface of a silicone oil bath. Since the adjustment is made by comparing the resistance of a thermistor being adjusted to the resistance of a standard thermistor, temperature control of the oil bath is not necessary. On P-type thermistors we usually adjust at room temperature. The standard thermistor and the thermistor being adjusted are mounted in opposite ratio arms of a Wheatstone bridge. Silicon is abraded away from the side of the thermistor until the resistance reaches the desired value. Abrasion is accomplished by a diamond bit in a 12000 rpm dentist-type drill, although other abrading techniques could work. The operator abrades until the galvanometer reaches 0, indicating no difference between the standard thermistor and the reference thermistor.
Step 27 - Final determination of temperature-resistance behavior at a minimum of three temperatures.

It is felt that measurement at three temperatures can be used to define the temperature-resistance behavior of these thermistors on a production basis, once a base curve has been carefully determined.

For gold-doped P-type silicon these temperatures might be $25^\circ C$, $0^\circ C$, and $-60^\circ C$. Thermistors which are on the low-resistance side according to this test can be sent for readjustment.

These measurements can be made by comparing the resistance of the production thermistor to the resistance of a well determined standard thermistor.

Step 28 - Paint and bake at $210^\circ C$.

The final painting is normally done by hand in order to get as much paint as possible on the edges of the thermistor. Spraying or dipping usually results in a deficiency of paint near the edges of the thermistor. Presently, we are using a white silicone enamel for painting. However, we believe that a room-temperature vulcanizing silicone rubber, such as Dow Corning RTV 731, if applied in a thin film, will withstand extremes of temperatures more readily. Thinness of film is needed in order to obtain a desirable time constant of response.

Normally we apply two coats of silicone enamel and bake 1 hour for the 1st coat and 2 hours for the second coat. Baking time or temperature in not critical as long as the coating cures sufficiently.

Experience with the described production process could lead to the elimination of certain steps such as 6, 7, 8, and 21. These are check points which might be dropped as knowledge is gained on the product output for a given silicon input. Although some short cuts might be made in the process, we feel the most consistent results will be obtained by following the described procedure.

In the following section is a series of photographs demonstrating the most important steps in the process. These show equipment and techniques used in the specified steps.
Step 3

Equipment for measuring resistivity of silicon rods showing rod on 4-point probe table, power supply, galvanometer and potentiometer.

Closeup of silicon rod on 4-point probe.
Step 5

Zone leveling of gold in silicon rod. An argon ambient is being used in a Lindberg float zone refiner.
Mounting of wafer size thermistors in pressure clamps for resistivity measurement.
Step 11

Lapping of wafers in one direction on 180 C silicon carbide paper. Wafer is mounted under metal weight.
Step 14

Apparatus for vacuum deposition of aluminum on a single silicon wafer. Apparatus is mounted in chamber of a Kinney high vacuum float zone refiner. It is setting on a single turn secondary of a transformer type RF coil.

Umbrella shaped holder for seven wafers during vacuum deposition. This is also mounted in Kinney high vacuum chamber.
Homemade furnace for alloying aluminum to silicon at 580-630°C. Included is helium gas supply, Variac for power control and potentiometer for measuring.
Immersion of aluminum alloyed wafers in boiling ammonium hydroxide.

Nickel plating of wafers at 96°C.
Cutting of square dice on high speed diamond saw by crosscutting technique. Silicon wafer is embedded in jeweler's wax to prevent clipping of edges.
Square dice (2 x 2 mm) as cut on high speed diamond saw by crosscutting technique. Wafer is completely imbedded in jewelers wax.

Cylindrical dice as cut on an ultrasonic dicer. These measure 2.5 mm in diameter by 5 mm in length. Other sizes are easily obtainable.
Step 24

Closeup of Teflon jig with thermistor element wedged into central slot. Timed copper leads are butted against the nickel plated electrodes.

Dipping of Teflon jig with thermistor in molten tin to solder leads.
Step 26

Adjusting N-type thermistor. Thermistor is ground and then lowered into silicone oil bath at 100°C and compared to standard thermistor.

Complete size adjusting equipment showing electronic galvanometer, Wheatstone bridge, micro-amp meter, and oil bath.
Step 26

Adjusting of P-type thermistors in silicone oil bath at room temperature. Thermistor being adjusted and standard thermistor are in opposite arms of Wheatstone bridge.
Step 27

Attaching Wheatstone bridge leads to thermistors in 25°C constant temperature bath.
Hand painting of thermistors with white silicone enamel. Only hand painting resulted is good coverage of the thermistor edges.
Example of Application of P-type Manufacturing Process

A monocrystalline silicon rod was purchased from Merck & Co. The resistivity and diameter profiles are given below. The first measurements were made 6 mm from the seed end of the crystal. The remaining readings were made at equal intervals along the 3-11/16" crystal.

<table>
<thead>
<tr>
<th>Diameter in mm</th>
<th>P-type Resistivity in ohm cm</th>
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<tr>
<td>22.4</td>
<td>15.6</td>
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<td>22.7</td>
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<td>22.5</td>
<td>14.1</td>
</tr>
<tr>
<td>22.8</td>
<td>25.7</td>
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</table>

The rod and seed were cleaned and etched with a solution of 1 volume concentrated HF (49%) and 3 volumes concentrated HNO₃ (70%). They were blown dry with argon. The seed and rod were set up in an argon ambient zone refiner for float-zone leveling of gold. The seed used in this case was 22.2 ohm cm P-type. 0.1515g of high-purity gold was inserted as the dope between the seed and the rod. The seed, rod and dope were fused together in the first molten zone and the zone was passed through the rod at the rate of 8 inches per hour. At the end of the pass the rod was 100% monocrystalline.

Three wafers, 2.2 mm thick were cut from the seed end, center and chuck end of the zone-leveled rod. The wafers were deposited with aluminum and alloyed at 580°-630°C. Nickel plating was applied over the aluminum. The aluminum and nickel plating on the edges of the wafers were sanded away to prevent short circuiting. Pressure contacts were made. The wafers were immersed in a 25°C bath and measured. They read 19.5, 19.3 and 16.7 ohms respectively.

It was desired to make units of 500 ohms at 25°C from this material. Using the equation \( R = \frac{21}{\frac{1}{A}} \), it was determined to cut the remaining rod into wafers 2.2 mm thick. It was assumed that the final dice would be cut square with a diamond saw and measure 3 to 4 mm on a side.

The rod, which had been previously mounted on a ceramic block with jewelers wax and left oriented on the diamond saw, was cut into wafers 2.2 mm thick. Methyl alcohol was used to separate the jewelers wax from the wafers. The wafers were then washed with acetone and alcohol. The wafers were hand lapped in one direction with 180-C silicon carbide paper. They were washed with water and wiped with paper to remove fine particles. Successive washings with acetone, methanol, water, conc. HF, water and methanol then followed. The wafers were prepared for aluminum deposition.
Aluminum was deposited on both sides of the wafers as described under Step 14. Handling very carefully so as not to mar the surface the wafers were transferred to the alloying furnace. Here they were alloyed under helium at a temperature of 580-630°C for 20 minutes. The wafers were allowed to cool in the helium ambient.

After cooling the wafers were etched in concentrated (49%) HF to remove oxide and excess aluminum from the surface. They were then wiped with paper and washed successively with water, conc. HF, and water. Immersion in boiling ammonium hydroxide, NH₄OH, then followed. When the first signs of visible reaction occurred on the surface, the wafers were transferred rapidly to the nickel-plating solution. Nickel plating was carried out as described in Step 19.

The plated wafers were heated at 210°C overnight in a nitrogen atmosphere. Of the 20 wafers which had been cut from the bar, 7 from spaced intervals were measured at 25°C with pressure contacts. These measured 19.5, 20.0, 19.9, 21.7, 19.3, 18.3, 16.5, 18.6 ohms respectively. These readings checked with the readings on the first three trial wafers. Wafer 15 which measured 16.5 ohms was considered for dicing to 500 ohms at 25°C thermistors. Using the physical dimensions and the equation

\[ R = \frac{\rho L}{A} \]

the resistivity, \( \rho \), of the silicon in this wafer was calculated to be 282 ohm cm. Again using the equation and fixing R at 500 ohms, \( \rho \) at 282 ohm cm and 1 at 2.2 mm, the area, A, of a 500 ohm device would be 0.1240 cm² or a square of 3.5 mm on a side. Since it was desired to have the final devices fall below 500 ohms, square dice of 3.6 mm on a side were cut on the diamond saw. Leads were soldered to the dice with tin as described in Step 24. A washing with methyl alcohol followed.

The size of the thermistors was adjusted as described in Step 26 upgrading the resistance to 500 ohms at 25°C. Before adjusting, the thermistors from wafer 15 measured about 470 ohms. The resistance of the thermistors from this wafer were measured at 0, 25 and -58.5°C to determine the deviation from a standard curve. The results of these measurements are given below.
### TABLE 22

Resistance in ohms at various temperatures for different sample nos.

<table>
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<tr>
<th>Sample No.</th>
<th>25°C</th>
<th>0°C</th>
<th>-58.5°C</th>
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<tr>
<td>W-15-1</td>
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<td>1592</td>
<td>80,330</td>
</tr>
<tr>
<td>W-15-2</td>
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<td>80,648</td>
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<td>W-15-15</td>
<td>500</td>
<td>1592</td>
<td>80,250</td>
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</tbody>
</table>

Only sample W-15-14 was of questionable resistance at -58.5°C. The thermistors were then coated with a film of white silicone enamel, baked 1 hour at 210°C, recoated and baked 4 hours at 210°C. The thermistors are then finished and ready for use.

The standard curve for these thermistors is given below:
**TABLE 23**

Temperature vs. Resistance -5°C to +50°C.

<table>
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9. **Manufacturing Process for Gold-Doped N-type Silicon**

A twenty-four step manufacturing process was developed for producing gold-doped N-type thermistors. The unbalanced pilot line was operated according to this process while producing the 200 contract samples of N-type thermistors.

The twenty-four steps are outlined below:

1. Determine necessary starting silicon resistivity.
2. Purchase monocrystalline rod from a commercial supplier, or prepare the same.
3. Check resistivity profile with 4-point probe.
4. Prepare rod for zone leveling: Etch 1:3 HF/HNO₃, dry.
5. Zone level with high-purity gold.
6. Cut three wafers from seed end, chuck end, and center.
7. Prepare three wafer-size thermistors from these slices and measure resistance at 25°C.
8. Calculate desired thickness of production wafers based on resistivity of the three trial wafers and dicing tool sizes.
9. Cut remaining zone-leveled rod into wafers.
10. Wash with alcohol, acetone, alcohol.
11. Lap wafers with 180-C silicon carbide paper.
12. Wash with water, wipe with paper to remove fine particles.
13. Wash successively with acetone, alcohol, water, conc. HF, water.
15. Nickel plate wafers by electrodeless technique at 96°C.
16. Heat treat at 210°C overnight in argon or nitrogen.
17. Recheck resistivity of several wafers with pressure contacts.
18. Determine final dice size.
19. Dice wafers.
20. Solder leads to dice with tin at 240°C.
21. Wash with methyl alcohol.

22. Adjust size of thermistors by high-speed diamond grinding comparing resistance to resistance of a standard thermistor.

23. Determine final temperature-resistance behavior at a minimum of three temperatures.

24. Paint and bake at 210°C.

Many of these steps are the same as in the case of gold-doped P-type silicon. Only the steps which are different will be discussed in more detail below:

**Step 1 -** Determine the necessary starting silicon resistivity.

The resistance of a thermistor will follow the equation,

\[ R = \rho \frac{L}{A} \]  

Equation No. 2

- \( R \) = resistance of thermistor in ohms
- \( \rho \) = resistivity of thermistor material in ohm cm.
- \( L \) = length of the thermistor between electrodes in cm.
- \( A \) = the cross sectional area of the thermistor in cm².

If we know the resistance and approximate size of a desired thermistor, it is possible to calculate the resistivity of the gold-doped silicon to be used in the device by this equation. Then using the relationship shown in Graph 15, it is possible to determine the necessary starting silicon resistivity. These curves were determined experimentally by doping various resistivities of N-type silicon with 0.15 grams of high-purity gold in the zone-leveling process. The rod diameters were 7/8 to 1", the estimated weight of the molten zone was 20 grams. 0.15 grams of gold was found to be the maximum amount that could be added to such a zone without causing polycrystallinity problems.

**Step 2 -** Purchase monocrystalline rod from a commercial supplier, or prepare the same.

**Step 3 -** Check resistivity profile with 4-point probe.

**Step 4 -** Prepare rod for zone leveling: Etch 1 to 3 HF/HNO₃, dry.

**Step 5 -** Zone level with high-purity gold.

This is a standard zone-refining procedure in which high-purity gold is used as a dope between the seed and the rod. The gold is melted into the first zone as the seed and the rod are fused together by means of radio-frequency heating.
Graph 15

INITIAL RESISTIVITY - OHM CM. N-TYPE
The resistivity of the seed should be smaller than the resistivity of the rod by an amount depending upon the amount of the seed that is fused into the first zone. This level has to be determined by experience with the float-zone leveling process. Normally, the amount of N-type impurity in the first zone should be equal to approximately 3 times that of N-type impurity that the zone will see as it passes up the rod.

The zone-leveling pass is made at the rate of about 8 inches per hour. If the rod does not go single on the first pass, it should be re-doped before the second pass in order to obtain the same amount of residual gold.

**Step 6** - Cut three wafers from seed end, chuck end, and center.

**Step 7** - Prepare three wafer-size thermistors from these slices and measure the resistance at 25°C.

**Step 8** - Calculate the desired thickness of production wafers based on resistivity of three trial wafers and dicing tool sizes.

**Step 9** - Cut remaining zone-leveled rod into wafers.

**Step 10** - Wash with alcohol, then acetone, alcohol.

**Step 11** - Lap wafers with 180-C silicon carbide paper.

**Step 12** - Wash with water, wipe with paper to remove fine particles.

**Step 13** - Wash successively with acetone-methanol-water-conc. HF-water.

This step is used to prepare the wafer surfaces for nickel plating. It removes impurities such as grease, finger oil, oxide film, etc., which might interfere with a good aluminum deposition. Whereas, good thermistors might be made by eliminating some of these steps, we feel that the most consistent results will be obtained by using all of them.

Beginning with this step and until the wafers are finally nickel plated, the circular surfaces should not be touched by the fingers or mechanical holders such as forceps. A mark, made by a forcep, for instance, can result in poor adhesion of the plating.

**Step 14** - Immerse wafers briefly in boiling ammonium hydroxide.

The wafers are immersed until the first signs of reaction are noted visually. This is usually a slight bubbling on the surface. They are then transferred rapidly to the nickel-plating solution with little atmospheric exposure.

After the washings and HF treatments the wafers are not exposed for any lengthy periods to the atmosphere. Exposure can result in an oxide film and subsequent poor adhesion of the metal plating. The wafers are kept under the solutions and transferred quickly from one to the other. Transfer to the ammonium hydroxide of Step 18 is also rapid.
Step 15 - Nickel plate wafers by electrodeless technique at 96°C.

Step 16 - Heat treat 210°C overnight in argon or nitrogen.

Step 17 - Recheck resistivity of several wafers with pressure contacts.

Step 18 - Determine final dice size.

Step 19 - Dice wafers.

Step 20 - Solder leads to dice with tin at 240°C.

Step 21 - Wash with alcohol.

Step 22 - Adjust size of thermistors by high-speed diamond grinding comparing resistance-to-resistance of a standard thermistor.

Step 23 - Final determination of temperature-resistance behavior at a minimum of three temperatures.

It is felt that measurement at three temperatures can be used to define the temperature-resistance behavior of these thermistors on a production basis, once a base curve has been carefully determined.

For gold-doped N-type silicon these temperatures might be 25°C, 100°C and 200°C. Thermistors which are on the low-resistance side according to this test can be sent for readjustment.

These measurements can be made by comparing the resistance of the production thermistor to the resistance of a well determined standard thermistor.

Step 24 - Paint and bake at 210°C.

Experience with the described production process could lead to the elimination of certain steps such as 6, 7, 8, and 17. These are check points which might be dropped as knowledge is gained on the product output for a given silicon input. Although some short cuts might be made in the process, we feel the most consistent results will be obtained by following the described procedure.

Example of Application of N-type Manufacturing Process

An N-type monocrystalline rod, 5-1/2" long and 7/8" in diameter was prepared for float-zone leveling. The resistivity profile was measured. The first reading was made 6mm from the seed end. The remaining readings were made at equally spaced intervals along the rod.
TABLE 24

N-Type Resistivity
in ohm cm

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<th>Value</th>
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<td>8.2</td>
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The rod and a seed, 640 ohm cm N-type, were etched in a solution of 1 volume of 49% HF to 3 volumes 70% HNO₃ to a bright-finish brown with argon and dried under infrared heat. The seed and rod were set up in an argon ambient zone refiner for float-zone leveling of gold. 0.1579 gms of high-purity gold was inserted as a dope between the seed and the rod. Also inserted was 0.0247 gms of a silicon phosphorous dope containing 14,000 ppb phosphorous. The seed, rod and dope were fused together in the first molten zone and the zone was passed through the rod at the rate of 8 inches per hour. At the end of the pass the rod was 100% monocrystalline.

Three wafers, 1.2mm thick were cut from the seed end, center and chuck end of the zone-leveled rod. They were cleansed, lapped and plated with nickel by the electrodeless technique. The nickel plating on the edges of the wafers was sanded away to prevent short circuiting. Pressure contacts were made. The wafers were immersed in a 25°C bath and measured. They read 4864, 4848 and 4882 ohms respectively.

It was desired to make units of 500,000 ohms at 25°C from this material. Using the equation $R = \frac{\rho L}{A}$ it was determined to cut the remaining rod into wafers 1.2mm thick. It was assumed that the final dice would be cut square with a diamond saw and measured about 2mm on a side.

The rod, which had been previously mounted on a ceramic block with jewelers' wax and left oriented on the diamond saw was cut into wafers 1.2mm thick. Methyl alcohol was used to separate the jewelers' wax from the wafers. The wafers were then washed with acetone and alcohol. The wafers were hand lapped in one direction with 180-C silicon carbide paper. They were washed with water and wiped with paper to remove fine particles. Successive washings with acetone, methanol, water, conc. HF, and water then followed. The wafers were prepared for nickel plating.

Immersion in boiling ammonium hydroxide, NH₄OH, then followed. When the first signs of visible reaction occurred on the surface,
the wafers were transferred rapidly to the nickel-plating solution. Nickel plating was carried out as described in Step No. 15.

The plate wafers were heated at 210°C overnight in a nitrogen atmosphere. Of the wafers which had been cut from the bar, 6 from spaced intervals were measured at 25°C with pressure contacts. These measured 4855, 4889, 4916, 4920, 4994, 4873 ohms respectively. These readings checked with the readings on the first three trial wafers. Wafers 1 and 2 which measured 4864 and 4848 ohms were considered for dicing to 400,000 ohms at 25°C thermistors. Using the physical dimensions and the equation \[ R = \frac{\rho L}{A} \] the resistivity, \( \rho \), of the silicon in this wafer was calculated to be 150,000 ohm cm. Again using the equation and fixing \( R \) at 500,000 ohms, \( \rho \) at 150,000 ohm cm and \( L \) at 1.2mm, the area, \( A \), of a 500,000 ohm device would be 0.040 cm² or a square of 2.0mm on a side. Since it was desired to have the final devices fall below 500,000 ohms, square dice of 2.1mm on a side were cut on the diamond saw. Leads were soldered to the dice with tin as described in Step 20. A washing with methyl alcohol followed.

The size of the thermistors was adjusted as described in Step 22 up-grading the resistance to 500,000 ohms at 25°C. Before adjusting, the thermistors measured about 430,000 ohms.

The resistance of the thermistors from this wafer was measured at 25, 60, 100 and 200°C to determine the fit to a standard curve. The results of these measurements are given below.

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The thermistors were then coated with a film of white silicone enamel, baked 1 hour at 210°C, recoated and baked 4 hours at 210°C. The thermistors were finished and ready for use.
The temperature at which these thermistors show the most variation is at 200°C. Most of the variation in N-type thermistors occurs near 200°C. A master curve for these thermistors is given below.
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10. **Contract Sample Thermistors**

Figure 20 is a photograph of typical thermistors prepared under the contract. Gold-doped P-type samples were prepared with resistances at 25°C of 4000, 5000 ohms and 500 ohms. The four and five thousand ohm samples were cylindrical in shape, being approximately 2.5mm in diameter by 2mm in length. The 500 ohm P-type samples measured approximately 3.8 x 3.8 x 2.3mm, the electrode area being square. These were the largest thermistors produced and had the most undesirable time constants of response. However, they were the easiest to adjust to a given resistance.

The gold-doped N-type samples made under the contract were 500K ohms at 25°C and measured 2x2x1mm. Again the electrodes were square and the dice were made by cross-cutting techniques on the high-speed diamond saw.

We are including in Tables 27 and 28 temperature-resistance curves for the 4000 and 5000 ohm P-type samples. The 500 ohm P-type and 500K N-type curves were given under the sections on process examples.
Finished thermistors, actual size. Comparison is made with a Bendix ML419/AMT-4 thermistor.
### TABLE 27

Temperature vs. Resistance -85°C to +30°C.

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# TABLE 28

Temperature vs. Resistance -85°C. to +30°C.

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11. **Thermistor Variance Before Adjustment**

Thermistor resistance data at 25°C were examined to estimate variances between wafers and within wafers prior to adjustment. The purpose of the calculations was to estimate the per cent acceptable thermistors if adjustment is by-passed and wafers are cut to get the specified average resistance.

Wafers are cut into thermistors that are about 15% larger than the size desired. The thermistors are then individually adjusted to give the specified resistance by grinding. This process insures no losses.

Data on the average thermistor resistance from 22 wafers from seven rods were examined (Table 29) to see how closely the target value could be hit. The standard deviation was ±7.7% of the target value, with some evidence of bias in the square, N-type devices. Next the variation of the thermistors within a wafer was examined. Three wafers, producing 47 thermistors each, gave a standard deviation of ±4.5% around the wafer average. Therefore, the standard deviation for the thermistors around the target value was ±9.0%.

If the process were changed to set the target value at the final desired resistance, some of the thermistors would be too small and would have to be discarded. With a standard deviation of ±9%, 17.5% of the thermistors would fall within ±2% of the target value. 41.3% would be too small and 41.3% would be too large. If a ±1.0% limit is set, only 8.8% would be acceptable and 45.6% would be out of limits on each side.

If the target value were lowered slightly from the objective to cut losses, the percent acceptable would drop also, but at a slower rate. An economic optimum exists and can be calculated if costs can be assigned to an unadjusted thermistor and to the adjustment process.
TABLE 29

WAFFER TARGET RESISTANCE AND AVERAGE OBTAINED AT 25°C.

Circular

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<th>Target</th>
<th>Average Obtained</th>
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<th>%Deviation from target</th>
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Rod C-189-15B P-Type

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Square

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MINIMUM COST CONDITIONS

The acceptable thermistor unit cost is:

Cost of thermistors + cost to adjust

Final total of acceptable thermistors

OR:

\[ C_U = \frac{100 \ C_T + N_L \ C_A}{N_L + N_A} \]

WHERE:

- \( C_T \) = Cost of unadjusted thermistor, $/thermistor
- \( C_A \) = Cost of adjusting, $/thermistor
- \( C_U \) = Product unit cost
- \( N_L \) = % of thermistors too large and requiring adjustment
- \( N_A \) = % of acceptable thermistors (not requiring adjustment)
- \( N_S \) = % of thermistors too small
- \( 100 = N_L + N_A + N_S \)

The unit cost will be a minimum when:

\[ \frac{dN_L}{dN_S} = \frac{(100 \ C_T + N_L)}{(N_L + N_A)} \]

This means for a given thermistor to adjustment cost \((C_T/C_A)\) ratio, the optimum target resistance below specifications can be calculated. The results are shown on graph 16 for a \( \pm 2.0\% \) specification.

In the case of the thermistors produced under the contract it is very unlikely that the cost of adjustment \( C_A \) (estimated at 15$/device) is greater than the cost of a thermistor. Hence \( C_T/C_A \) would be greater than 1. Under these circumstances it would be desirable to cut the thermistors such that the target resistance is 14 to 15% below specs. The target resistance could be even farther below specs, but generally it is desirable to remove as little material as necessary in adjusting.
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<td>13.4</td>
</tr>
<tr>
<td>9</td>
<td>11.1</td>
</tr>
<tr>
<td>10</td>
<td>9.1</td>
</tr>
<tr>
<td>12</td>
<td>5.3</td>
</tr>
<tr>
<td>14</td>
<td>3.4</td>
</tr>
<tr>
<td>16</td>
<td>2.3</td>
</tr>
<tr>
<td>18</td>
<td>1.3</td>
</tr>
</tbody>
</table>
12. Yields

Yields to be expected by using the manufacturing methods described are summarized in the block diagram (Figure 21). Notice that the yields when producing samples similar to those of the contract were approximately 85% based on the thermistor dice. We feel this figure can be considerably improved by operating a continuous balanced pilot line.

Included are estimated data for devices of half the diameter and thickness of the contract samples. Notice that at only 70% yield the number of thermistors obtained per unit of silicon is 3 to 4 times that obtained with the contract sample sizes. The problem of course is that size adjusting to within ± 1% is very difficult on the smaller samples. Perhaps improvements could be made to the size adjustment step so that these small sizes could be adjusted efficiently. In any case this seems to be the desirable direction in which to move in terms of quickness of response and yields.
Silicon Rod 8" Long 9/8" Dia.
Wt. 257 gms.

9" of Gold Doped
Silicon Monocrystal

160 Wafers .9 mm Thickness

20,000 Thermistor Dice
1.25 mm Dia. x .8 mm

60,560 Finished Thermistors in 92% Spec. Estimated 70% Yield on Thermistor Dice.
~ 10% Lost Soldering
~ 10% Lost Adjusting Size
~ 10% Rejected in Final Tests

110 Wafers 1.0 mm Thickness

4,950 Thermistor Dice
2.0 mm x 2.0 mm x 1.0 mm
(Similar to Contract Samples: N-Type 500,000 Ohms)

4,207 Finished Thermistors in 92% Spec. Approximate 85% Yield on Thermistor Dice.
~ 5% Lost Soldering
~ 5% Lost Adjusting Size
~ 5% Rejected in Final Tests
Yield based on actual pilot line operation

68 Wafers 2.0 mm Thickness

3,740 Thermistor Dice
1.8 mm Dia. x 2.0 mm
(Similar to Contract Samples: 4,000 - 5,000 Ohms P-Type)

3,179 Finished Thermistors in 92% Spec. Approximate 66% Yield on Thermistor Dice.
~ 5% Lost Soldering
~ 5% Lost Adjusting Size
~ 5% Rejected in Final Tests
Yield based on actual pilot line operation
13. Fitting of Experimental Points by Empirical Equation

As mentioned previously, the curves for both P- and N-type gold-doped silicon can deviate from a straight line on a logarithm of resistivity versus reciprocal of the absolute temperature plot. This deviation is greater in the case of P-type silicon, becoming significant at 25 or 30°C. At higher temperatures the curve flattens out such that at 200°C there is little sensitivity to temperature. However, there is still some sensitivity to temperature at say 100°C. We felt that if the experimental points could be fitted by a carefully selected empirical equation, it would extend the predictable portion of the curve. Hence the thermistors might be useable to 100°C, admittedly with some loss of sensitivity.

Equations for the resistivity as a function of temperature have been derived for gold-doped silicon. These have the general form:

$$R = B_1T^F + B_2e^{\Delta E/KT}$$

where:

- $R$ = resistance in ohms
- $B_1$ = a constant
- $B_2$ = a constant
- $F$ = a constant
- $\Delta E$ = a constant
- $T$ = absolute temperature in °K
- $K$ = Boltzman constant

The experimental data were processed through a computer to find the values of the constants which gave the best fit or smallest standard deviation. In the case of the P-type silicon studied the best fit was obtained when, $E = 0.340$ e.v. and $F = 0$. The data are summarized in Table 31.
TABLE 31

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Experimentally Determined Resistance</th>
<th>Resistance Calculated $\Delta E/\kappa T$</th>
</tr>
</thead>
<tbody>
<tr>
<td>-58.56</td>
<td>79,600</td>
<td>80,016</td>
</tr>
<tr>
<td>-50.80</td>
<td>42,250</td>
<td>42,087</td>
</tr>
<tr>
<td>-40.49</td>
<td>19,200</td>
<td>19,157</td>
</tr>
<tr>
<td>-30.91</td>
<td>9,926</td>
<td>9,807</td>
</tr>
<tr>
<td>-20.38</td>
<td>5,052</td>
<td>4,993</td>
</tr>
<tr>
<td>-10.81</td>
<td>2,872</td>
<td>2,842</td>
</tr>
<tr>
<td>0.00</td>
<td>1,600</td>
<td>1,587</td>
</tr>
<tr>
<td>+9.92</td>
<td>980.0</td>
<td>973.8</td>
</tr>
<tr>
<td>+19.99</td>
<td>620.5</td>
<td>620.4</td>
</tr>
<tr>
<td>+30.00</td>
<td>411.7</td>
<td>414.0</td>
</tr>
<tr>
<td>+40.00</td>
<td>285.6</td>
<td>288.7</td>
</tr>
<tr>
<td>+50.03</td>
<td>207.5</td>
<td>210.1</td>
</tr>
<tr>
<td>+60.18</td>
<td>157.0</td>
<td>159.1</td>
</tr>
<tr>
<td>+70.47</td>
<td>124.5</td>
<td>125.4</td>
</tr>
<tr>
<td>+79.98</td>
<td>104.2</td>
<td>104.2</td>
</tr>
<tr>
<td>+90.35</td>
<td>88.5</td>
<td>88.4</td>
</tr>
<tr>
<td>+99.99</td>
<td>78.0</td>
<td>78.0</td>
</tr>
</tbody>
</table>

Standard deviation, $\sigma_f = 0.8\%$

$B_1 = 45.85$
$B_2 = 8.069 \times 10^{-4}$

The fit of the data is very good. In only a few cases do the calculated points vary more than 1% from the observed points. The value of $\Delta E = 0.340$ agrees well with the values determined by independent techniques (13).

The reason for deviation from a linear log $\phi$ vs. $1/T$ plot appears to be due to the saturation of the gold levels at high temperatures. The value of the empirical equation is that it allows the expression of resistance and sensitivity in an explicit manner at temperatures up to 100°C for P-type silicon. It also establishes the gold donor level precisely at 0.340 e.v. above the valence band.
## Time Constant of Response

A study was completed comparing the time constant of response of our thermistors uncoated and coated with various materials to that of the Bendix AMT-4, ML419 radiosonde thermistor. Our thermistors measured 4 x 4 x 1 mm. The gold-doped silicon radiosonde thermistor is on the order of 2 x 2 x 1 mm, hence the time constant would be somewhat less than in the case of this study. The time constant was measured while changing the temperature of the thermistor from 0°C to approximately 26°C. The time constant was taken as the time in seconds for the resistance to change 63% of the total in an air stream of 800 ft/min. A summary of the results of this study is given in the table.

### TABLE 32

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Nature of Coat</th>
<th>Time Constant (seconds)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1176</td>
<td>Uncoated</td>
<td>2.0</td>
</tr>
<tr>
<td>1180</td>
<td>Hand painted with 1 coat of white silicone enamel.</td>
<td>2.8</td>
</tr>
<tr>
<td>1182</td>
<td>Dipped in thinned (~25% in toluene) RTV-731* and then RTV-521**</td>
<td>3.9</td>
</tr>
<tr>
<td>1187</td>
<td>Hand painted with RTV-731 thinned at 50% with toluene</td>
<td>4.3</td>
</tr>
<tr>
<td>1186</td>
<td>Hand painted with RTV-731 thinned at 50% with toluene</td>
<td>4.6</td>
</tr>
<tr>
<td>1179</td>
<td>Dipped in thinned white silicone enamel</td>
<td>5.3</td>
</tr>
<tr>
<td>Bendix, AMT-4, ML419</td>
<td></td>
<td>5.5</td>
</tr>
<tr>
<td>1178</td>
<td>Dipped in white silicone enamel, then hand painted with white silicone enamel to cover corners</td>
<td>7.2</td>
</tr>
<tr>
<td>1184</td>
<td>Dipped in RTV-731 thinned at 50%</td>
<td>7</td>
</tr>
<tr>
<td>1185</td>
<td>Dipped in RTV-731 thinned at 50%</td>
<td>7</td>
</tr>
</tbody>
</table>

* - RTV-731 Dow Corning room temperature vulcanizing rubber.
** - RTV-521 Dow Corning room temperature vulcanizing rubber.
In order to obtain a desirable time constant it is necessary to keep the coating as thin as possible. The silicone enamel is desirable since a thin coating can be applied and it has high opacity. The coating which has the best temperature range is a Dow Corning room temperature vulcanizing rubber, RTV-731. Main problem with this material is that it is very viscous and difficult to apply in a thin coating. The hiding is not as desirable. We attempted to obtain thinner and more opaque coats by adding pigments and thinning with solvents such as xylene. This investigation was not successful because of the tendency of RTV-731 to polymerize when exposed to atmospheric moisture. Hand painting is the best technique for applying these coatings since dipping or spraying tends to leave the corners or edges of the thermistor deficient in paint. Based on the results of the study it was decided to use two hand-painted coats of silicone enamel on each thermistor with a baking period of about one hour at 200°C after the first coat and two hours after the second coat.

Information received later from the U. S. Weather Bureau pointed out that they define the time constant as "the time required for the thermistor to indicate 63% of the total increment in temperature in a stream of air with a speed of 820 ft/min." The increment can be positive or negative and the result should be about the same. The factor measured in this test is the physical ability of the thermistor to accept or dispose of heat to the environment.

To measure the time constant a small wind tunnel was made with two air intakes whose temperature and speed could be independently regulated. The temperatures used were 25°C and -10°C for testing the P-type material and the Bendix AMT-4, ML-419 thermistor; for the N-type thermistor, 90°C and 25°C. The speed of the air was kept constant at 820 ft/min.

Ten readings were taken from each sample. For a given thermistor or a group of thermistors of the same kind, the maximum variation in the measurement was 4 tenths of a second.
The following table summarizes the results:

<table>
<thead>
<tr>
<th>Thermistor Number</th>
<th>Size in mm.</th>
<th>Ohms at 25°C</th>
<th>Time Constant in seconds</th>
</tr>
</thead>
<tbody>
<tr>
<td>2m-3-7</td>
<td>2.5 dia. x 2.5</td>
<td>4K</td>
<td>Minimum (1) 2.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 2.2</td>
</tr>
<tr>
<td>W5-35</td>
<td>2.6 dia. x 2.6</td>
<td>4K</td>
<td>Minimum (1) 2.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 2.4</td>
</tr>
<tr>
<td>W2-34</td>
<td>2.6 dia. x 2.3</td>
<td>4K</td>
<td>Minimum (1) 2.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 2.2</td>
</tr>
<tr>
<td>3253-A-10</td>
<td>2.2 x 2.2 x 2.0</td>
<td>500K</td>
<td>Minimum (1) 2.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 2.4</td>
</tr>
<tr>
<td>3253-A-14</td>
<td>2.2 x 2.3 x 2.2</td>
<td>500K</td>
<td>Minimum (1) 2.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 2.4</td>
</tr>
<tr>
<td>3253-A-16</td>
<td>2.3 x 2.3 x 2.3</td>
<td>500K</td>
<td>Minimum (1) 2.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 2.5</td>
</tr>
<tr>
<td>2m-2-11</td>
<td>2.5 dia. x 3.0</td>
<td>5K</td>
<td>Minimum (1) 2.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 3.1</td>
</tr>
<tr>
<td>2M-2-21</td>
<td>2.5 dia. x 2.9</td>
<td>5K</td>
<td>Minimum (1) 2.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 3.3</td>
</tr>
<tr>
<td>2M-3-15</td>
<td>2.5 dia. x 2.9</td>
<td>5K</td>
<td>Minimum (1) 3.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 3.3</td>
</tr>
<tr>
<td>W-14-8</td>
<td>3.7 dia. x 1.9</td>
<td>.5K</td>
<td>Minimum (1) 2.9</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 3.2</td>
</tr>
<tr>
<td>Bendix M1-419</td>
<td>.7 dia. x 39</td>
<td>50K</td>
<td>Minimum (1) 3.1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 3.4</td>
</tr>
<tr>
<td>W-15-4</td>
<td>3.8 x 3.8 x 2.3</td>
<td>.5K</td>
<td>Minimum (1) 4.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 4.5</td>
</tr>
<tr>
<td>W-6-12</td>
<td>7.8 x 3.8 x 2.5</td>
<td>.5K</td>
<td>Minimum (1) 4.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Maximum (1) 5.1</td>
</tr>
</tbody>
</table>

(1) - Average of ten readings.

This table shows that the time constant of our thermistors, except the bulky 3.8 x 3.8 x 2.3 mm size, is equal or better than the time constant of the Bendix thermistor and well within the specifications of 5 seconds of the present contract.
15. **Dissipation Constant**

The dissipation constant is defined as the amount of power in milliwatts that has to be applied to a thermistor to raise the temperature of the thermistor one degree centigrade above the surrounding medium. For surrounding medium, manufacturers specify well stirred oil baths and still air. Both values are given with the characterization chart of the thermistor.

To measure the dissipation constant of the thermistors in still air, we use a cardboard box with a capacity of about 25 liters. The thermistors were evenly distributed hanging from the top of the box by their own leads. The box was sealed. Then the resistance of all the thermistors was measured using a current through the sample of 5 microamps. The resistance was remeasured increasing the amount of current. This step was repeated until the resistance of every thermistor showed a decrease equivalent to one degree centigrade or more. The measurements in oil were made using Dow-Corning 200-20 silicone oil stirring at constant speed and at 25°C. Again several measurements were taken increasing the current through the thermistor before each measurement until the resistance showed a decrease equivalent to 1° centigrade or more.

With the values obtained in the previous tests we were able to draw a smooth curve for each thermistor and find the exact value of the current that will give the difference of 1° centigrade higher than the surrounding medium. Having the current "I" and the resistance "R" of the thermistor, the power in milliwatts was calculated using the formula: \( P = I^2R \). The following table summarizes the results.

<table>
<thead>
<tr>
<th>Thermistor Number</th>
<th>Size in mm.</th>
<th>Ohms at 25°C</th>
<th>Dissipation Factor in a well stirred oil bath (milliwatt/°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W-6-12</td>
<td>3.8 x 3.8 x 2.5</td>
<td>0.5K</td>
<td>3.7 54</td>
</tr>
<tr>
<td>W-14-8</td>
<td>3.7 dia. x 1.9</td>
<td>0.5K</td>
<td>2.6 37</td>
</tr>
<tr>
<td>W-15-4</td>
<td>3.8 x 3.8 x 2.3</td>
<td>0.5K</td>
<td>2.6 -</td>
</tr>
<tr>
<td>2mm-3-15</td>
<td>2.5 dia. x 2.9</td>
<td>5K</td>
<td>2.8 52</td>
</tr>
<tr>
<td>2mm-2-11</td>
<td>2.5 dia. x 3.0</td>
<td>5K</td>
<td>2.8 60</td>
</tr>
<tr>
<td>W-5-35</td>
<td>2.6 dia x 2.6</td>
<td>4K</td>
<td>2.2 35</td>
</tr>
<tr>
<td>W-2-34</td>
<td>2.6 dia x 2.3</td>
<td>4K</td>
<td>2.1 35</td>
</tr>
<tr>
<td>2mm-3-7</td>
<td>2.5 dia x 2.5</td>
<td>4K</td>
<td>1.9 37</td>
</tr>
<tr>
<td>Bendix MI-419</td>
<td>0.7 dia.x 39</td>
<td>50K</td>
<td>0.8 1.5</td>
</tr>
<tr>
<td>I-27</td>
<td>2.2 x 2.2 x 2.2</td>
<td>500K</td>
<td>0.4 8</td>
</tr>
<tr>
<td>I-22</td>
<td>2.2 x 2.3 x 2.2</td>
<td>500K</td>
<td>0.4 8</td>
</tr>
<tr>
<td>A-14</td>
<td>2.2 x 2.3 x 2.2</td>
<td>500K</td>
<td>0.4 7</td>
</tr>
</tbody>
</table>

This table shows first, a fairly good reproducibility for thermistors of the same size and range; second, that the dissipation constant for the same thermistor is about 20 times higher in the oil bath than it is in air; and third, that all our thermistors excepting perhaps the high resistance, small size, N-type, have a higher dissipation constant than the Bendix thermistor.
16. **Static Temperature Lifetime**

Besides the data that have already been mentioned in previous sections on thermal stability, there are additional data to demonstrate the stability of single-crystal silicon thermistors when held for extended periods of time at a given temperature within the operating range. It is felt that the most severe conditions are those that occur at the upper end of the suggested operating range of 200°C. Any thermistor which is stable at 200°C would be stable at any temperature below 200°C. The table below summarizes aging data at 200°C for various types of thermistors both in the wafer size and in the diced form.

<table>
<thead>
<tr>
<th>Thermistor No.</th>
<th>Type</th>
<th>Original Resistance (Ohms)</th>
<th>Hours at 200°C</th>
<th>Resistance After Treatment at 200°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>N</td>
<td>7305</td>
<td>1765</td>
<td>7302</td>
</tr>
<tr>
<td>2</td>
<td>N</td>
<td>7345</td>
<td>1765</td>
<td>7345</td>
</tr>
<tr>
<td>3</td>
<td>N</td>
<td>5688</td>
<td>1765</td>
<td>5685</td>
</tr>
<tr>
<td>4</td>
<td>N</td>
<td>5641</td>
<td>1765</td>
<td>5636</td>
</tr>
<tr>
<td>5</td>
<td>P</td>
<td>19.1</td>
<td>2199</td>
<td>18.8</td>
</tr>
<tr>
<td>6</td>
<td>P</td>
<td>22.1</td>
<td>2199</td>
<td>22.0</td>
</tr>
<tr>
<td>7</td>
<td>P</td>
<td>19.2</td>
<td>2199</td>
<td>19.1</td>
</tr>
<tr>
<td>8</td>
<td>P</td>
<td>18.6</td>
<td>2199</td>
<td>18.6</td>
</tr>
<tr>
<td>9</td>
<td>N</td>
<td>10,490</td>
<td>2816</td>
<td>10,150</td>
</tr>
<tr>
<td>10</td>
<td>N</td>
<td>12,230</td>
<td>2816</td>
<td>12,090</td>
</tr>
<tr>
<td>11</td>
<td>N</td>
<td>12,650</td>
<td>2816</td>
<td>12,460</td>
</tr>
<tr>
<td>12</td>
<td>P</td>
<td>635</td>
<td>4934</td>
<td>633</td>
</tr>
<tr>
<td>13</td>
<td>P</td>
<td>611</td>
<td>4934</td>
<td>615</td>
</tr>
<tr>
<td>14</td>
<td>P</td>
<td>614</td>
<td>4934</td>
<td>614</td>
</tr>
<tr>
<td>15</td>
<td>P</td>
<td>606</td>
<td>4934</td>
<td>603</td>
</tr>
<tr>
<td>16</td>
<td>P</td>
<td>610</td>
<td>4934</td>
<td>608</td>
</tr>
</tbody>
</table>

These data demonstrate that both the P- and N-type thermistor material are stable for extended periods of time, on the order of several thousand hours at temperatures in the range of 200°C. Frequently the change in resistivity is little or less than 1%.
III CONCLUSIONS AND RECOMMENDATIONS

Single-crystal semiconductor silicon, because of its purity, resistivity, energy gap, and availability, is an outstanding candidate for monocrystalline thermistor development.

Gold-doped monocrystalline silicon can produce thermistors which are highly reproducible, predictable and sensitive over the range of -85° to +200°C.

Float-zone leveling produces a product with less variability than diffusion material.

Forming of ohmic and mechanically sound contacts to gold-doped silicon results in good thermistor stability.

Measurement precision and speed are improved by the use of the comparison or ratio technique.

Final size adjustment is necessary to obtain high yields of devices with ±2% specifications.

Size adjustment on small thermistors is difficult.

The dependence of thermistor resistivity on resistivity of the starting silicon is very useful for production purposes.

Short-term annealing at 210°C can increase the temperature stability of gold-doped silicon.

Yields of 85% or better are possible when using the prescribed 28-step process for P-type silicon and 24-step process for N-type silicon.

Future work should be directed toward smaller-sized thermistors with very fast response times.

Size adjusting should be improved or revised so that it can be used on very small samples.

Other techniques of encapsulation such as canning should be investigated.
IV BIBLIOGRAPHY

9. Ibid., Hannay, p 342.

- 99 -
V APPENDIX
TO: Dr. Wayne T. Barrett
FROM: Blanche B. White
DATE: October 3, 1961
SUBJECT: THERMISTORS - LITERATURE SEARCH 15 - 46

In searching the literature for information on thermistor element properties and thermistor element fabrication, the following sources were consulted:


In addition, at our request, Astia prepared a bibliography, covering the last five years, of all documents in their collection pertinent to this subject. This was supplemented by a visit to Astia to search their older catalogue cards for any information in this field.

Information obtained from manufacturers concerning thermistors manufactured by them is listed in a section of the appended bibliography (p. 50-53).
The greater part of the rather extensive literature on thermistors covers the composition, method of manufacture, and properties of the various ceramic mixed oxide thermistors. Most frequently used of the oxides are Ni, Mn, Ti, Fe, Co, Cu and Cr. Oxides of Sn, Mg, Al, Li, Ta, V, Tb, U, Pb, Ba, Fe, Pr and Nb etc. are also mentioned in the patent literature.

Only two references were found to the use of III-V intermetallics in thermistors, and only a very limited number of references were found to the use of Si and Ge. Other semiconductor materials used for this application were also noted and references will be found in the bibliography.

There are numerous references describing specific applications of thermistors. These have not been included in the bibliography; however, a table showing general areas of application is included (p. 53).
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THERMISTORS
LITERATURE SEARCH L8 - 46

1. Adey, A.W.
REVIEW OF THERMISTOR THERMOMETRY.
AD-153,609, Sept. 1957.
(Astia Bibliography).
The usefulness of a thermistor as a thermometer is related to the following characteristics: (a) Sensitivity, (b) Time constant, (c) Ruggedness, (d) Reliability or stability.

TEMPERATURE-DEPENDENCE OF THE ELECTRIC CONDUCTIVITY OF THE Cu₂O-Mn₂O₃ SYSTEM.
(C.A. 52, 13342).
The temperature dependence of the conductivity $\sigma$ of samples obtained by sintering a mixture of 25% Ni₂O₃ and 75% Cu₂O was investigated. The linear function of $\log\sigma$=f(1/T) shows 2 breaks at 140 and 230°C. The energy of activation in the region below 140°C is 0.7 e.v.; in the region 140-230°C, 0.907 e.v.; and in the region 230-300°C, 1.106 e.v. The material is suggested for use in the construction of thermistors.

3. Aoki, Ikuo (Chiba Univ.).
ELECTRICAL CONDUCTIVITY OF SEMICONDUCTORS: OXIDES OF COBALT AND MANGANESE.
Specimens were prepared by mixing oxides of Co and Mn in various ratios and sintering the mixtures at 1200°C. The specific resistance $R$ and the temperature coefficient dR/dT were measured at room temperature with the aid of x-ray powder photography. A minimum of $R$ was found at the mole ratio Co:Mn = 2:1. The analysis of the diffraction rings shows that the specimen (I) having this mole ratio has the spinel structure (Mn,Co)-(Co,Mn)₂O₄. I has a comparatively high dR/dT. The specimen consisting only of Co₂O₃ and CoO has larger $R$ and smaller dR/dT than does the mixture of Co and Mn. As Mn content increases, $R$ increases rapidly. I may be the best for a stable thermistor with low $R$ and high dR/dT.

4. Aoki, I.
ELECTRIC CONDUCTIVITY OF MIXTURES OF OXIDES OF COBALT, MANGANESE, AND NICKEL.
Specimens were prepared by mixing oxides of Co and Mn in various ratios and sintering the mixtures at 1200°C. The specific resistance $R$ and the temperature coefficient dR/dT were measured at room temperature with the aid of x-ray powder photography. A minimum of $R$ was found at the mole ratio Co:Mn = 2:1. The analysis of the diffraction rings shows that the specimen (I) having this mole ratio has the spinel structure (Mn,Co)-(Co,Mn)₂O₄. I has a comparatively high dR/dT. The specimen consisting only of Co₂O₃ and CoO has larger $R$ and smaller dR/dT than does the mixture of Co and Mn. As Mn content increases, $R$ increases rapidly. I may be the best for a stable thermistor with low $R$ and high dR/dT.
NiO for O:1, (Ni,Co)O for the ratios between 1:8 and 3:2, and (Ni,Co)O + CoO + Co₃O₄ for 2:1 to 8:1. That of the system Ni₂Mn₃O₈ was NiMn₂O₄ + Mn₃O₄ for the ratio 1:3, NiMn₂O₄ for 1:2, NiMn₂O₄ + NiO for the ratio between 1:1 and 3:1. Specific resistance vs. composition curve of the Co-Mn-O system gave a minimum when x:y was equal to 2:1, and the activation energy of electric conductivity increased with Mn content. Specific resistance of Co-Ni-O system gave a minimum when x:y was between 3:2 and 5:4, and the activation energy was minimum at x:y = 1:1. Specific resistance of the Ni-Mn-O system was minimum at the composition NiMn₂O₄, and the activation energy was minimum at the same composition. These results suggest that the electric conductivity of Co-Mn-O and Ni-Mn-O thermistors is explained by the inverse spinel structure, and the electric conductivity of the Co-Ni-O system is attributed to impurity atoms present in the NiO lattice.

5. Arma Research Foundation.
PHYSICAL MECHANISM OF INFRARED DETECTORS.
(Astia Bibliography).

A research program is in progress to illuminate fundamental conduction mechanism of thermistors. Questions posed are: (1) What are the atoms involved and how are they arranged? (2) How did this arrangement come into being? (3) How does the arrangement lead to certain mechanisms of electronic motion? and (4) How do these mechanisms give rise to the observed properties? The first two questions are answered by X-ray studies of Mn, Co and Ni oxides. Efforts are directed toward the third question, by determining valence states of cations in the lattice. This is done by (a) examining electrical properties of thermistor materials in which normal ratio of cations is varied, and (2) examining by neutron diffraction techniques thermistor materials in which relative proportions of Mn, Co, and Ni have been grossly altered.

THERMISTORS. I. TiO₂-Cr₂O₃ ROD-TYPE THERMISTORS.
(C.A. 42, 8923b).

The manufacturing method is: sintering the mixture of TiO₂ and Cr₂O₃ in 1:3 molar ratio at 1300° for 1 hour, and heating in H₂ at 1000° for 1 hour. The electrical characteristics of the thermistors obtained are given. In spite of many disadvantages, the thermistors of this type could be applied to carrier and repeater telephone systems.

THERMISTORS. II. TITANIUM DIOXIDE-CHROMIUM SESQUIOXIDE BEAD-TYPE THERMISTORS.
(C.A. 46, 1367d).

To obtain side-heater-type thermistors, the elements of the thermistor were improved to small-bead type (diameter = 0.4 mm.) having a small heat content. They were made by heating a mixture of TiO₂ and Cr₂O₃.
(1:3 molar ratio) at 1300° for 30 minutes in vacuo. The bead-type thermostors were superior in power sensitivity to type 22kΩ (0 mw.), 10kΩ (40 mw.). Several characteristics of side-heater-type thermostors, thus prepared, together with the resistance variation owing to aging at 200° were observed. III. NICKEL MONOXIDE-MANGANESE OXIDE-COBALT OXIDE THERMISTORS. Ibid. 295-6. NiO-MnO₄-Co₃O₄ thermistors of higher γ (resistance compressibility of side-heater-type thermistor) were prepared by heat treatment in air. Resistance of the thermistors thus prepared were 10kΩ-2meg.Ω. The constant B, in the equation \( R = \frac{R_0}{e^{(-B/T)}} \), was about 4500, and γ was 4.5-5.5. These results should be compared with those of the TiO₂-Cr₂O₃ thermistors: B = approximately 2500, γ = 4.6-4.9.

   TEMPERATURE MEASUREMENT WITH THERMISTORS.

   The advantages of thermistors for temperature measurement applications, bases for probe selection, and future developments are outlined.

   THERMISTORS-APPLICATION IN TEMPERATURE MEASUREMENT AND CONTROL.
   (Ceramic Abstracts 1960, 453b).

   A review. 8 figures, 14 references.

10. Atkins Technical, Inc.
    ELECTRONIC THERMOMETERS.

    The Thermophil electronic thermometer which utilizes germanium thermistors to measure surface and immersed temperatures between -200 and 450° is announced...

    TEMPERATURE-VARIABLE RESISTORS.
    (C.A. 32, 2823b).

    A resistance having a large negative temperature coefficient comprises a fused mixture of CuO and MnO₂. A mixture of CuO 75% and MnO₂ 25% is fused in an arc between Cu electrodes, terminal wires being inserted in the melt and a globule of resistance material withdrawn and solidified with the wires in position.

12. Bailey, H.T.
    THERMISTOR BASICS.

    RESISTANCE THERMOMETER - SEMICONDUCTING SENSITIVE ELEMENTS.
THERMISTOR INFRA RED DETECTORS. PART I. PROPERTIES AND DEVELOPMENT.
(Astia Bibliography).

Barnes Engineering Co. under license from Western Electric started in 1952 to manufacture thermistor infrared detectors utilizing the technique developed at Bell Laboratories.

Employed as infrared radiation detectors, thermistor elements are made small and thin and attached to a good heat sink to produce fast response. They are known as "solid-backed" thermistor infrared detectors or bolometers. Thermistor materials are metallic oxides, chiefly MnO, NiO, and CoO. They have a negative coefficient of resistance about 4%/°C.

Materials used had resistance (ohm cm.) 2500 and 250 respectively, material constant \( \rho (\text{°K}) \) 3800 and 3400 respectively, and temperature coefficient of resistance \( a = -0.042 \) and -0.038° respectively \( (T = 30^\circ \text{K}) \).

When a pulse of radiant energy \( \Phi \) falls on the blackened thermistor element, a portion is absorbed (about 60%) and heats the thermistor; at the same time heat generated in the thermistor is conducted away to the backing block.

15. Battelle Memorial Inst., Columbus, Ohio.
TEMPERATURE ELEMENTS (THERMISTORS) COVERING THE PRODUCTS OF THE FOLLOWING MANUFACTURERS WW, XX, YY, AND ZZ.
(Astia Bibliography).

Battelle Memorial Inst., Columbus, Ohio.
TEMPERATURE ELEMENTS (THERMISTORS) COVERING THE PRODUCTS OF THE FOLLOWING MANUFACTURERS WW, XX, YY, AND ZZ.
(Astia Bibliography).

Battelle Memorial Inst., Columbus, Ohio.
TEMPERATURE ELEMENTS (THERMISTORS) COVERING THE PRODUCTS OF THE FOLLOWING MANUFACTURERS WW, XX, YY, AND ZZ.

16. Beakley, W.R.
DESIGN OF THERMISTOR THERMOMETERS WITH LINEAR CALIBRATION.
(Physics Abstracts 54, No. 6180 (1951)).

Under certain circuit conditions a current or voltage may be obtained which varies almost exactly linearly with the temperature of a thermistor. Equations are given from which may be calculated: (a) the extent of the range of temperature within which departure from linearity is less than a specified value; (b) the circuit conditions which provide optimum linearity; (c) the sensitivity of the circuit in terms of current change per unit temperature; (d) the maximum permissible current through the thermistor.
STABILITY OF THERMISTORS.
(C.A. 50, 69021).

Thermistors may reach a state when their constants remain stable, however, they may undergo sudden arbitrary changes. This is probably due to subjecting the thermistors to temperatures outside the allowable range. Within certain limits, it is still possible to obtain temperatures accurate to within 0.02°.

18. Becker, Joseph A., and Howard Christensen (to Bell Telephone Laboratories Inc.).
HIGH-TEMPERATURE COEFFICIENT RESISTOR.
(C.A. 47, 6798b).

A rapid-response temperature-sensitive resistor can be made having electrodes fired simultaneously with the semiconductive body. Thermistors of this type are free from noise, have increased power capacity and thermal response, and have lower resistance. The finely divided metal for the electrode (I) is mixed with a temporary binder (II) and spread in a thin film on a surface. Over this is spread a film of finely divided semiconducting material (III), with a high resistance temperature coefficient, with a temporary binder (IV). This double film is removed and heat-treated at 200-600° to remove II and IV and later fused at high temperatures. Another I may be applied to III in film form prior to the fusing operation. Alternatively, there may be interposed between each I film and III layer, films of mixtures of I and III. Finely divided Pt is used as I film to minimize the barrier layer at the I-III interface. III is comprised of one or more of MnO, NiO, CoO, Cu₂O, FeO, and ZnO. II and IV are methacrylates, vinyl polymers, or cellulose acetate butyrate with a volatile solvent. The thickness of the finished flake is on the order of 20Å with 1Å thickness for each electrode. The fusing temperature is 1100° for most oxides and not over 1450°.

PROPERTIES AND USES OF THERMISTORS - THERMALLY SENSITIVE RESISTORS.
(C.A. Index 1946, 370).

New circuit element and control device is made of solid semiconducting materials whose resistance decreases about 4% per degree C.

20. Bemski, G., and J.D. Strenthers (Bell Telephone Laboratories).
GOLD IN SILICON.
(C.A. 52, 19488b).

Heat-treatment of Si, both p-type and n-type, to temperature in excess of 900° frequently results in a decrease of lifetime and is sometimes accompanied by an increase in resistivity. In the present experiments it was found that Au was introduced during such heat-treatments in concentrations sufficient to account for the observed changes in the
electrical characteristics. Au has been observed on the surfaces of all the Si samples examined so far. Heat-treatments in the temperature range 1100-1300° result in a concentration of Au in the bulk material of about $10^{14}$ at./cc. A film of Ni or Cu on the Si surface during the heat-treatment has a gettering action for Au. Heat-treatment in vacuo can also be effective in the removal of Au.


Comparative tests of the thermoelectric properties of Fe-constantan thermocouple and a Thermistor (Victory Engineering Corp. Type 51A1) show greater sensitivity, higher stability up to 580° F., and shorter thermal time lag for the Thermistor...


Experiments were performed which indicate that the present manufacturing techniques will produce thermistors which are capable of being used as precision thermometers. Thermistors will allow temperatures to be measured with an accuracy approaching 0.001°C and appear to be stable over long periods of time. Thermistors can be made into practically any size or shape. Bead thermistors can be manufactured in extremely small sizes (down to 0.015 cm.). Small flake-type thermistors can be made with thermal relaxation times of the order of a few milliseconds and are suitable for handling rapidly changing temperatures. The nonlinearity of the thermistor makes precision calibration involved and the interpretation of the data obtained more difficult. The upper limit of the temperature range of thermistors is controlled by their thermal instability. This can be expected to improve with improved manufacturing techniques. It is probably unwise to use them much above 150°C without extensive preliminary tests of higher temperature stability. The low-temperature limit arises as a result of their extreme sensitivity. At low temperatures, their resistance increases so much that the measuring circuit, which is designed for the higher temperature range, becomes insensitive. Circuits can be specially designed for these low-temperature regions.
23. Bol, Arie (to Hartford National Bank and Trust Co.).
ELECTRICAL RESISTOR.
(C.A. 47, 9850).

In the manufacture of electrical resistors, in which the resistance element consists of a sintered homogeneous spinel phase of Fe$_3$O$_4$ and Zn$_2$TiO$_4$ in a 1:1 ratio, the conducting leads are attached by cementing them into the ends of the element. The leads are inserted in holes at the ends of the element, an aqueous paste of 45% Ag$_2$O and 55% Fe$_3$O$_4$ powder is then applied in the holes. The element is heated at 600° for about 2 minutes to cause the Ag$_2$O and Fe to react exothermically and form a mixture of Fe$_3$O$_4$ and Ag, which is liquid at the high temperature of the reaction. If the conducting leads have a very smooth surface, a small amount of low-melting glass can be used as a flux in the adhesive mixture. Ti powder can be substituted for the Fe powder.

NEGATIVE-TEMPERATURE-COEFFICIENT ELECTRICAL RESISTORS.
(C.A. 52, 18896).

Electrical resistors with negative temperature coefficient are made by sintering a mixture of powdered oxides or carbonates of Ni and Li at 1100-1300°. The electrical uniformity varies with the trace element content of the Ni source, but this variation may be suppressed by incorporation of 1-30 (preferably 10-20) mole % CuO into the sintered mass. For example, powdered NiO and CuO were ground for 16 hours with alcohol in a ball mill with addition of an amount of LiOO$_3$ sufficient to give final proportions of 15 and 1.5 mole % CuO and LiOO$_3$ respectively, the rest being NiO. To 100 grams of the filtered, dry mixture, 5 grams of a binder solution containing 20 grams Me acrylate and 1 ml. di-Bu phthalate per 100 ml. MeOAc was added. The resulting mass was molded into 5 X 5 X 15-mm. rods and sintered at 1240° for 1 hour. Extensive data are given to show the improvement of electrical uniformity resulting from the inclusion of CuO.

A RELATIONSHIP BETWEEN RESISTANCE AND TEMPERATURE OF THERMISTORS.
(C.A. 42, 18871).

The equation $R = A + B/(T + C)$ for the resistance $R$ of a thermistor at $T° K.$ is proposed. Least squares analyses of the most precise resistance-temperature data available for 3 different thermistor materials show that the equation is a considerable improvement over its predecessors...
26. Brackenbury, P.S. 
RESEARCH AND DEVELOPMENT OF SEMI-COAUTHING MATERIALS. PRACTICAL 
APPLICATIONS FOR ULTRA-SENSITIVE TEMPERATURE MEASURING EQUIPMENT AND 
AUTOMATIC CONTROL AND STABILIZING PROBLEMS. INTERROGATION OF ERWIN WEISE. 
Bibliography of Scientific and Industrial Reports 9, 892 (1948).) 

The semi-conductors in which Weise had carried out research were in a 
material known as magnesium titanium spinel (MgO \cdot TiO_2) bearing 
the trade name of Urdox. This material is made in the form of sheet, rod, 
and tube. It is also possible to make them in the form of fine fila-
ments of about 70 microns diameter formed on a thread of silk of about 
30 microns. Method of manufacture, list of publications by Weise or 
in collaboration, and patents made by Weise are given as well as 
remarks to uses and possible use.

27. The British Thomson-Houston Co., Ltd. 
RESISTANCES. 
Brit. 496,210, Nov. 21, 1938. 
(C.A. 22, 28237). 

A resistance having a substantially linear negative coefficient of 
resistance from 0° to 50° consists of Te, alloyed or not with Ag, 
formed in single crystals by progressive solidification from a molten 
state and annealed at 105-25° for a time depending on the annealing 
temperature and the coefficient required.

28. Broom, R.F. 
A LOW-TEMPERATURE RESISTANCE THERMOMETER USING p-TYPE GALLIUM ARSENIDE. 
(Physics Abstracts 62, No. 5402 (1959)). 

The construction and calibration of the thermometer are described. 
The thermometer consisted of a single crystal of copper-doped gallium 
arsenide with copper-plated contacts. The resistivity of the thermo-
meter material was 2.5 X 10^5 ohm-cm. at 4.2° K. and the sensitivity, 
\( \frac{\Delta R}{\Delta T} \) (dR/dT), was approximately constant at 2.5 over the 
range 2 to 25° K. The thermometer was photoconductive and must be 
screened from light for reliable readings.

SEMICONDUCTING PROPERTIES OF IRON OXIDE THERMISTOR MATERIAL. 
(C.A. 50, 1051la). 

The semiconducting properties of iron oxide thermistor material containing 
10% each of copper and titanium ions as impurities have been examined. 
This material, prepared by sintering the oxide powder, is found to have 
the crystal structure of alpha Fe_2O_3 indicating simple substitution of 
copper and titanium for iron ions in the lattice. The resistivity is a 
logarithmic function of temperature having an activation energy of 0.52 
electron volt. Hall effect and Seebeck effect studies of the semicon-
ductor show that this variation is due to thermal activation of carriers 
to conduction levels. The carriers are found to be electrons with
mobility of about 1.0 cm²/volt second. Concurrent with the origin of electrical conduction is ferromagnetic behavior, and a saturation magnetic moment of 2.0 emu/gram at room temperature is observed above magnetic saturation at about 5000 oersteds. A discrepancy between donor concentration determined from extrapolated carrier density measurements and the known concentration of impurity atoms appears to indicate a type of cancellation between the effect of copper and titanium atoms.

30. Brouwer, G.P.
THERMISTORS.
(C.A. 48, 9257d).
A review dealing with properties and uses of semiconductors of high negative temperature coefficient.

COBALT-MANGANESE OXIDE SEMICONDUCTOR BOLOMETERS.
(Physica Abstracts 65, No. 8836 (1960)).
The basic parameters characterizing the performance of thermistor bolometers are defined and classified, and 3 methods are given for their measurement. The construction of 3 types of bolometer is discussed in detail and their performance is described.

KMT-14 THERMISTOR.
Elektricheskto No. 2, 71-3 (1960).
(Monthly Index of Russian Accessions 13, 3018 (1960)).

TEMPERATURE ELEMENTS, THERMISTORS.
(Astia Bibliography).
The electrical performance was investigated of rod, disk, and bead thermistors as affected by low-temperature storage, moisture resistance, temperature cycling, shelf life, and continuous-load life. The program also included an investigation of temperature hysteresis and steady-state-current time characteristics of galvanic action, terminal and body strength, and the effects of soldering on the terminals. Specimens used were purchased from 4 specified manufacturers (WW, XX, YY, and ZZ as indicated in the manufacturers' code which was previously transmitted to the Signal Corps Engineering Laboratory). The values of material constants (β) for the scheduled specimens were calculated from the initial measurements and varied between 1400 and 4600. Stable thermal characteristics were noted in the specimens. Resistance changes under increasing and decreasing temperature were normally between the limits of 0.12 and 2.0%. Very slight changes in β were noted as a result of temperature-cycling, cold-storage, and shelf-life and load-life operations. Moisture-resistance exposure resulted in average changes up to 2% in β, and the presence of galvanic action was noted in 1 of 24 types of elements. Negative resistance values are graphed. The spread of the current-voltage characteristics among the 10 specimens comprising a
group of the same make, type, and resistance was very narrow. Strength
tests applied to the terminals of the specimens provided a wide range
of results from 1 ounce to 15 pounds. The changes in resistance as a
result of terminal soldering were consistent and very small. Terminal-
twist tests indicated that all terminal leads can be expected to fail
in a range between 1 and 5 twists.

34. Christensen, Howard, and Joseph J. Kleimack (to Bell Telephone Labs, Inc.).
METALLIC-OXIDE RESISTOR.
(C.A. 43, 4401a).

In making resistors of sintered metallic oxides, the oxides are permitted
to come to equilibrium with an O-containing atmosphere at the temperature
at which the equilibrium O content of the body is such that the desired
resistance is obtained, and then the resistor is sealed off from the
surrounding atmosphere.

35. Christensen, Howard (to Bell Telephone Labs, Inc.).
THERMISTOR BY OXIDATION OF A NICKEL-MANGANESE ALLOY.
U.S. 2,636,012, April 21, 1953.
(C.A. 47, 7700a).

Thin, thermally sensitive conductive flakes suitable for use as thermis-
tors (thermally sensitive resistive elements) are prepared from thin
foils of an alloy containing 80 atomic % Mn and 20 atomic % Ni. The
foils, which have a thickness of 0.5-25 μ, are heated in an oxidizing
atmosphere to 600-700° and kept in the furnace for at least 16 hours,
during which time the temperature is allowed to drop to 450-500°. The
alloy is completely oxidized by this treatment without separation of
the two oxides. To produce a spinel-type crystalline structure, the
oxidized material is heated from 450-500° to 875-1150° in 2 hours.
The final product consists of smooth-surfaced flakes, a relatively
uniform mixture of Ni and Mn oxides. It has stable electrical pro-
properties and is relatively free from mechanical irregularities. The
starting material may contain 40-90 atomic % Mn and 10-60 atomic % Ni.

36. Christensen, Howard (to Bell Telephone Labs, Inc.).
HIGH-TEMPERATURE-COEFFICIENT RESISTORS.
U.S. 2,674,583, April 6, 1954.
(C.A. 48, 11683h).

Thin-film resistors whose resistance varies greatly with temperature
are formed by first mixing a long-chain-polymer temporary binder with
particles of resistor material of such dimensions relative to the length
of the polymer chain that a gel mixture forms. Resistor particles of a
larger size are then added to the mixture. They are held in suspension
by the gel, thereby providing a film of uniform density. In a typical
example, the resistor flakes are composed of Mn and Ni oxides combined
in an Mn:Ni atomic ratio of 2:41. In preparing a mixture for resistor
flakes, 6 grams of a thermistor material having a particle size of 0.5-
1 μ is milled with 6 grams of a temporary binder and 12 cc. of solvent
in a high-speed ball mill to about 1% of the original particle size in
6 hours. An additional 7 grams of resistor material (2 μ) is added to
the mixture, which is then milled an additional hour. A thin film of
this mixture is spread on a smooth, flat surface to a thickness of several thousandths of an inch. The film is dried, removed from the flat surface, and cut into the desired sizes and shapes. The binder is removed from the uncured flakes by heating to 400°F. The temperature is then raised to 1000-3000°F in about an hour, cooled gradually to 860°F, and cured for 16 hours at this temperature. After cooling, electrodes are attached to the flakes. As binders, isobutyl methacrylate and cellulose acetate butyrate can be used.

37. Clark, Layton E., Jr. DETERMINATION OF EXACTNESS OF FIT OF WESTERN ELECTRIC TYPE 14B THERMISTOR TO THE EQUATION \( \log R = A + \frac{B}{T+E} \) AND \( \log R = A + \frac{B}{T+E} + CT \). AD-156,261, Feb. 26, 1958. (Astia Bibliography).

The closeness of fit of the two formulas over the -65 to +50°C temperature range was tested against the calibration of 47 Western Electric Type 14B thermistors, comprising samples for four production lots. Both formulas describe semiconductors of the Western Electric Type 14B within an accuracy of +0.1°C, except below -55°C where a correction to the formula must be applied. The four-constant equation describes the 14B thermistor with inaccuracies not greater than +0.04°C. with no correction to the formula at temperatures above -55°C, and within 0.04°C below -55°C. if corrections are made to the formula.

38. Clark, Layton E. REPORT ON DETERMINATION OF EXACTNESS OF FIT OF THERMISTORS TO THE EQUATIONS \( \log R = A + \frac{B}{T+E} \) AND \( \log R = A + \frac{B}{T+E} + CT \). AD-251,845, Jan. 1961. (Astia Bibliography).

The exactness of fit of the equations \( \log R = A + \frac{B}{T + \Theta} \) and \( \log R = A + \frac{B}{T + \Theta} + CT \) has been tested on a well-diversified selection of rod, disk, and wafer thermistor types, over all or portions of the temperature range of -60°C to +180°C. Data of these calibrations as well as tabulations of closeness of fit, expressed in temperature units, are presented in tabulated form for the convenience of those who may be considering a given thermistor type for use in a specific temperature-measurement application.


Plastic particles are coated with conductive compounds, e.g., Ag₂S, Fe₃O₄ or Cu₂O. Moldings exhibit electrical conductivity intermediate between that of metals and untreated plastics. The physical properties of the composition are substantially unchanged. Thus, 80 grams 20-mesh polystyrene beads and 20 grams Ag₂S (average nominal particle size 0.0005 inch) were ball milled 16 hours with steel balls, 90% of the surface of the beads being thus covered with Ag₂S. The particles were compression molded at 175° for 10 minutes at 10,000 pounds/sq. in. and cooled under pressure. The products had a specific resistance of 7 × 10⁶ ohm-cm. at 20° and 4 × 10⁸ ohm-cm. at 30°. They are useful in making thermistors.
Literature search cont'd.

40. Collins, C.B. (General Electric Co.).
SILICON CURRENT CONTROLLING DEVICES.
(Semiconductor Electronics 3 (1), No. 3687 (1959)).

An iron-doped silicon thermistor which is highly sensitive to temperature changes in the range from about -100 to 100° C. and an iron-doped silicon photoconductive cell which is highly sensitive to radiation, particularly infrared radiation, in the temperature range 0 to -200° C. are described. The magnitude of the resistance of these devices over the temperature ranges is such that they are useful in electrical control circuits.

41. Compagnie générale d'électroceramique S.A.
ELECTRICALLY CONDUCTING CERAMIC COMPOSITION FOR RESISTANCES.
Fr. 1,094,342, May 18, 1955.
(C.A. 52, 20976a).

In the manufacture of ceramic resistors, the scattering of the resistivity values caused by the varying degree of oxidation or reduction of the metal oxide in the furnace is eliminated by using compositions of clay containing Ti, Sn, or Ce as a plastifier and Si as an internal reducing agent. The necessity for controlling the atmosphere and (or) the addition of special additives is also avoided. The conductivity of such products increases with increasing Si content until a TiO₂/Si ratio of 3.85 is reached. Higher Si content has little effect. Preparation of the compositions and firing is done by the usual methods. The scattering of the resistivity is weakest when the conductivity is low and when the firing is done rapidly. The resistances thus obtained function normally at 800-900°. Typical compositions (TiO₂, Si, clay, and kaolin, respectively, in %) and their resistivities (r) at 20°, 300°, and 800°, respectively, are: 52, 15.5, 21.5, 15, r = 4.6, 0.8, 0.2; 42, 11.5, 27.4, r = —, 4, 2.5; 36.3, 9.4, 34.4, 19.9, r = 110, 25, 9; 7.25, 40.6, 24.35, r = 9000, 1500, —. The oxides of Sn and Ce behave similarly. Addition of these oxides to TiO₂ modifies the temperature coefficient and the expansion coefficient.

42. Compagnie générale de télégraphie sans fil.
ELECTRIC RESISTORS WITH HIGH NEGATIVE TEMPERATURE COEFFICIENT (THERMISTORS).
Fr. 1,052,015, Jan. 20, 1954.
(C.A. 52, 143971).

The thermistors described can be used at ambient temperatures and have permanent electrical characteristics reproducible within ±5%. This is achieved by controlled processing and careful choice of metallic oxides. Processing consists of homogenization by milling of the oxides for 400 hours with WC carbide balls and by long mixing or precipitation of easily decomposed organic salts. Minor components can be introduced by impregnation of the powder with solutions of their salts. Compression to very high pressures and heating to >1000° follows. At least a portion of the metallic oxides belongs to groups with the same number of electronic orbits, the same number of electrons in the outer-most orbit, but a variable number of electrons in the orbit immediately below.
For example, pure oxides are mixed in the proportions $\text{Mn}_2\text{O}_3$ 78, $\text{NiO}$ 14, and $\text{Fe}_2\text{O}_3$ 4%. Then 2% $\text{Al}_2\text{O}_3$ and 2% $\text{SiO}_2$ are added. The mixture is processed as above and the 10-mm. pills obtained are covered with an Ag-base glaze and baked once more at a temperature higher than the one at which they are to be used. Connections are soldered on and the thermistor painted with a silicone-base varnish. Resistivity at 25° is 100,000 ohm-cm. and temperature coefficient at 25°, 0.053 ohm/ohm/°C.

43. Compagnie générale de télégraphie sans fil.

**THERMISTORS.**


New compositions of metallic oxides provide improved reproducibility of resistivity and more lasting stability. The mixture consists of 3.5% $\text{Al}_2\text{O}_3$, a suitable amount of $\text{MnO}_2$ (at least 47%) and at least one of the oxides of Ni, Co, and Cu. Typical compositions are:

1. $\text{MnO}_2$ 80-6%, $\text{Co}_2\text{O}_3$ 0.5-15, and $\text{Al}_2\text{O}_3$ 3.5-5%;
2. $\text{MnO}_2$ 52-6, $\text{Co}_2\text{O}_3$ 32-6, $\text{NiO}$ 8-10, and $\text{Al}_2\text{O}_3$ 3.5-5%;
3. $\text{MnO}_2$ 47-52, $\text{Co}_2\text{O}_3$ 27.5-36, $\text{NiO}$ 7-9, $\text{Al}_2\text{O}_3$ 3.5-5, and CuO 10-12%. Resistivities were 50-150, and 90-150 kohm-cm./sq. cm., respectively. Cf. Fr. Addition 63,332.

44. Compagnie générale de télégraphie sans fil (by A. Vassiliev).

**ELECTRIC RESISTORS.**


Electric resistors are composed of metal oxide ions in an amount of Mn 75-95, Ni 4-25, and Cu 1-10%, based upon $\text{MnO}_2$, $\text{NiO}$, and $\text{CuCO}_3$-$\text{Cu(OH)}_2$. The oxide mixture is ball milled, presintered at 600-1100°, and ground. To the resulting frit, up to 15% of clay is added. The mixture is fired, ground, mixed with an organic binder, such as starch, and extruded. The final firing takes place at 1200° for 2 hours. Within the indicated composition range, values of $\rho$ may be varied between 50 ohm-cm. and 100,000 ohm-cm., and the coefficient $B$ between 2800 and 4300. In the equation $\rho = \rho_e e^{B/T}$, $\rho$ is the resistance at 25°, $\rho_e$ is the resistance at a very high temperature, and $T$ is the temperature in degrees Kelvin. It is shown that, with the ratio $\text{MnO}_2$:$\text{NiO}$ constant, $\rho$ is a function of the amount of CuO present and can thus be predetermined. The addition of clay has several important advantages. It has a stabilizing effect upon the changes of $\rho$ occurring with aging. This is explained by the assumption that during the heating of $\text{MnO}_2$, lower oxides, such as $\text{Mn}_2\text{O}_3$ or $\text{Mn}_3\text{O}_4$, are produced which subsequently undergo reoxidation. The latter is inhibited by the glassy phase formed by clay around the oxide particles. Clay also improves the values of $\rho$ and $B$ and renders firing conditions, such as control of temperature and rate of heating, less critical. By promoting better cohesion during extrusion, the addition of clay also permits the shaping of units of >10 mm. in diameter.
45. Compagnie industrielle des céramiques électroniques (Humbert and Masson, inventors).

**THERMISTOR MATERIALS.**

Fr. 1,112,965, Mar. 21, 1956.  
(C.A. 52, 8574f).

Substances having a negative temperature coefficient of resistance for thermistors that resist high energies (>2 w.) are manufactured by mixing 80-97% of a powdered thermistor material obtained by known methods, e.g., as in Fr. 1,052,015 (C.A. 52, 14398a) and Fr. 1,052,361, with 3-20% \( V_2O_5 \), shaping the mixture with an organic binder under pressure, and heating the article at 600-700°. Thus, a mixture of powdered \( MnO_2 \), \( NiO \), \( CoO \), and \( Al_2O_3 \) 40 grams was agglomerated with 100 grams urea and sintered for 24 hours at 1200°. The thermistor obtained was crushed, screened through 60-mesh, mixed with 115 grams \( V_2O_5 \), shaped at 1 ton/sq. cm. with 60 grams ceresin as a binder, and fired a second time at 700° for 1 hour.

46. Council of Scientific and Industrial Research.

**CERAMIC CONDUCTING BODIES.**

Indian 53,608, Jan. 18, 1956.  
(C.A. 50, 9709e).

Anatase-type \( TiO_2 \) is wetted with \( H_2O \), granulated to 20-mesh, and dried to 10% moisture. It is then pressed into wafers or rods, dried at 100° for 24 hours, and stacked over blocks of \( ZrO_2 \) and separated by \( ZrO_2 \) granules. The temperature of the oven is raised in 3 stages: overnight to about 900°, 1 day to 1200°, and 8 hours at 1380-450°. The semiconductors thus obtained may be used as thermistors up to 1000° for rectification or amplification.

47. Counts, William E., Robert W. Smith, and Karl Schwartzwalder (to General Motors Corp.).

**CERAMIC SEMICONDUCTOR COMPOSITIONS.**


A ceramic composition is formed of 15 to 60 \( TiO_2 \); 0 to 50 \( SnO_2 \); up to 15 \( Ta_2O_5 \), \( Nb_2O_5 \) or \( V_2O_5 \); 20 to 40 \( Al_2O_3 \); magnesia, millite, zircon, or chrome oxides; up to 10 parts \( MoO_3 \), \( WO_3 \), or mixtures thereof; and trace impurities, the \( Ta_2O_5 \), \( Nb_2O_5 \), and \( V_2O_5 \) being present in small but effective amounts to reduce the resistance to the desired value, and the \( MoO_3 \) and \( WO_3 \) being present in small but effective amounts to reduce the voltage coefficient of resistivity to the desired value.

48. Dearborn, Ernest F. (to Bell Telephone Labs, Inc.).

**ELECTRIC RESistor WITH NEGATIVE TEMPERATURE COEFFICIENT.**

(C.A. 26, 4437f).

Oxides are used together, such as \( NiO \), \( Mn_3O_4 \), and \( Co_2O_3 \) 30%.
49. Doucet, Y.

USE OF THERMISTORS.

(Physics Abstracts 55, No. 970 (1952)).

Comments are given on thermistors. The best are the type "R" in which sporadic fluctuations limit the precision to about ± 0.002° C. The curve log RVf(1/T) is not linear, as suggested by theory...

50. Dul’nev, G.N., and V.P. Savinov.

PRECISION OF TEMPERATURE MEASUREMENT BY SEMICONDUCTIVE THERMOSENSITIVE RESISTANCES.


The absolute temperature error is σ=PxF, where P is the dispersive power of the semiconductor thermosensitive resistance, and F is a coefficient dependent on the construction of the resistance, on the thermal conductivity of the material, and on the operating conditions. The coefficient F can be determined theoretically for a series of simple semiconductive thermosensitive resistances.

51. Estermann, I., A. Foner, and J.A. Randall (Carnegie Institute of Technology).

RESISTIVITY OF GERMANIUM AT LOW TEMPERATURES AND THE EFFECT OF ADDITIONS.

Physical Reviews 71, 484 (1947).

The resistivity of several germanium samples was measured between room temperature and 13° K. The purest sample shows a very steep increase of resistivity with cooling at temperatures below 30° K, giving a linear relation between log ρ and 1/T. The samples with low impurity concentration (both N and P type), e.g., 0.006 atmosphere percent Al, show a slight decrease in resistivity on cooling from room temperature to about 150° K, followed by an increase to about 2½ times the minimum value at 13° K. The sample with higher impurity concentration (0.04 atmosphere percent Al) shows practically no change in resistivity over the whole temperature range. In this sample, however, the resistivity at all temperatures increased substantially after the first four cooling and warming up operations; the same effect, but to a much lesser degree, was observed in other samples. Typical results are given in table below.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Type</th>
<th>273°K</th>
<th>180°K</th>
<th>90°K</th>
<th>60°K</th>
<th>30°K</th>
<th>20°K</th>
<th>14°K</th>
<th>13°K</th>
</tr>
</thead>
<tbody>
<tr>
<td>&quot;pure&quot;</td>
<td></td>
<td>17.5</td>
<td>19.2</td>
<td>11.2</td>
<td>9.2</td>
<td>7.3</td>
<td>1840</td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>P added</td>
<td>N</td>
<td>0.0242</td>
<td>0.0234</td>
<td>0.0301</td>
<td>0.0384</td>
<td>0.0551</td>
<td>0.0560</td>
<td>0.0560</td>
<td></td>
</tr>
<tr>
<td>0.006 at % Al</td>
<td>P</td>
<td>0.0324</td>
<td>—</td>
<td>—</td>
<td>0.0330</td>
<td>0.0641</td>
<td>0.0723</td>
<td>0.0712</td>
<td></td>
</tr>
<tr>
<td>0.04 at % Al (first run)</td>
<td>P</td>
<td>0.00510</td>
<td>0.00472</td>
<td>0.00520</td>
<td>0.00585</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td></td>
</tr>
<tr>
<td>0.04 at % Al (fourth run)</td>
<td>P</td>
<td>0.00937</td>
<td>—</td>
<td>0.0086</td>
<td>0.0086</td>
<td>0.0088</td>
<td>0.0087</td>
<td>0.0087</td>
<td></td>
</tr>
</tbody>
</table>

The resistivity of several germanium samples with different amounts of added impurities was measured between 4°K. and 20°K. Samples with high impurity content, in which the electron gas is degenerated below 100°K., do not change their resistivity in this temperature range. Another sample, which has a degeneracy temperature of 20°K., computed from Hall Effect measurements, showed the same behavior. Two other samples, which did not become degenerate above 20°K., exhibited a moderate increase in resistivity (20 and 80 percent respectively) between 20°K. and 4°K. Two "pure" samples, i.e., without added impurities, showed a very strong increase of resistivity below 20°K., increasing from about 2 ohm-cm. at 20°K. to more than 3 X 10^3 ohm-cm. at 4.7°K. At these low temperatures, however, the resistance depends strongly on the applied electric field, and this effect requires further study.


Beads are made by daubing a discrete quantity of a suitably prepared mixture on a pair of parallel wires. The wire is strung onto a frame which allows for adjustment of the wire tension and spacing. The beads are fired 4 hours at 2700°F. Three mixes were used: Mix A - 6.3 grams Cr₂O₃, 1.85 cc. Cr(NO₃)₃·9H₂O solution (specific gravity 1.2), H₂O added; Mix B - To 5.0 grams Cr₂O₃, Cr(NO₃)₃ was added (specific gravity 1.2), and as mix dried more Cr(NO₃)₃ was added; Mix C - To 5.0 grams Cr₂O₃, Cu(NO₃)₂ was added (specific gravity 1.2) and as the mix dried more Cu(NO₃)₂ was added. Mix A is most promising. It is felt particle size plays a part in ease with which bead forms. Beads are tested at 77° and 200°F. for resistance. It is felt furnace used for firing is faulty leading to low bead resistance.


The quantity of Cr(NO₃)₃ in the bead mix (Cr₂O₃/Cr(NO₃)₃) can be used to control the amount of spread in the resistance distribution of the fired beads. A small amount of densifier in the bead mix will increase the mean resistance and strength of the beads but may result in a cracked bead structure. Firing in the new furnace takes 16 minutes (in contrast to earlier 6 hours heating, 4 hours soaking and 6 hours cooling). The beads are well sintered and green in color. Bi₂O₃ was used as the densifier.
HIGH TEMPERATURE THERMISTORS.
(Astia Bibliography).

This report is concerned with testing of stability of chromic oxide bead thermistors. The firing technique was studied as a cause of poor stability.

56. Fisher, Joseph R. (to Bell Telephone Labs, Inc.).
ELECTRIC RESISTANCE ELEMENTS.
U.S. 2,082,102, June 1.
(C.A. 31, 4910a).

A method of producing resistors having a high negative temperature coefficient of resistance, for use in electric circuits, consists in placing a quantity of Ag sulfide powder between a pair of dies, applying sufficient pressure to dies to compress the Ag sulfide powder into a coherent mass, removing the resistance from the lower die, heating it in an atmosphere of S vapor for approximately 1 hour at 300-400° and then heating it in a stream of pure dry N at 200-300° for approximately 1 hour.

57. Fisher, Joseph R. (to Bell Telephone Labs, Inc.).
ELECTRIC RESISTORS.
U.S. 2,091,259, Aug. 31.
(C.A. 31, 7344f).

Resistors having a high negative coefficient of resistance for use in electric circuits are produced by subjecting a mass composed substantially of pure Ag to S vapor heated to 300° until the mass is converted into Ag sulfide.

58. Föllx, M.
CERAMIC BODIES FORMED FROM Al₂O₃ AND V₂O₃.
Silicates Ind. 17, 326-9 (1952).
(C.A. 47, 1940a).

V₂O₃ is, in many respects, different from Al₂O₃, comparable to the differences in the ionic constants of Al³⁺ and V⁴⁺. Its powder can be pressed and sintered to compact bodies which have a high electric conductance (similar to graphite), and also a high thermal conductance, like corundum. The inversion at -100° is characterized by a great change in properties: the modification which is stable at lower temperatures is a good insulator. Mixed powders of Al₂O₃ and V₂O₃ have been pressed and sintered at 1450-1800° in an atmosphere of H. There are two series of crystalline solutions: (I) in the concentration range from 100 to 50 molecular % V₂O₃; (II) from 100 to 90 molecular % Al₂O₃. The products II are easily made from Al₂O₃ and V₂O₃ powder, the latter, after fusing, acting as a bond; V₂O₃ is reduced to V₂O₃ in the subsequent sintering in H. With increasing Al₂O₃ content, the products I show decreasing elementary cell dimensions, but V₂O₃ is chemically stabilised by the Al₂O₃ addition. The crystalline solution
phases are highly refractory, with fusion points near 2000°, and an indistinct eutectic in the unmixing gap. The electric conductance of the bodies was measured in the range from -100 to +200°; the resistance, $\rho$, is a function of temperature. A graph with log $\rho$ plotted vs. molecular Al$_2$O$_3$ concentration shows two different systems of curves, for bodies fired at 1450° and at 1800°, with lower values for those fired at 1800°. Even a body with 95 molecular % Al$_2$O$_3$ fired at 1800°, is still a conductor, but with 1450° as the firing temperature the log $\rho$ is above 7 in a sample with 70 molecular % Al$_2$O$_3$. The temperature coefficient of $\rho$, as a function of the Al$_2$O$_3$ content, increases gradually over a wide range in composition, but above 90% Al$_2$O$_3$ it increases suddenly. Products fired at 1450° are friable, and oxidize slowly in air, while those fired at 1800° are stable, hard, and reproducible.

Bodies of type II are useful as "thermisters," i.e., electric resistors sensitive to temperature changes, and with a negative temperature coefficient of $\rho$. In this respect, they are similar to thermisters of magnetite, to which spinellides are added (e.g. Cr spinel). The thermal shock resistivity of the Al$_2$O$_3$-V$_2$O$_3$ bodies is satisfactory, their thermal expansion moderate, the thermal conductance high. Products II are hard, but still grindable. Theoretically, the hardness of corundum must be raised by the admixture of V$^{+++}$ ions in the crystal structure. It is possible that Al$_2$O$_3$-V$_2$O$_3$ bodies may be used as electric heating elements, up to 1800°, either in massive bodies, or as surface-treated Al$_2$O$_3$ ceramics, with V$_2$O$_3$ introduced by diffusion. An addition of Cr$_2$O$_3$ to Al$_2$O$_3$-V$_2$O$_3$ bodies may increase the stability of V$_2$O$_3$ against atmospheric oxidation.


A previous survey of semiconducting samples indicates that materials with about 2 X 10$^{17}$ impurity centers/cm$^3$ and room temperature resistivities of about 0.05 ohm cm. should have large enough temperature coefficients of resistivity at liquid helium temperatures to make good secondary thermometers. Experiments on p-type samples cut from a melt of germanium and 0.001 atomic % indium are described. The influence of the original location of a sample in the melt on the magnitude and reproducibility of its change of resistivity with temperature is discussed. Satisfactory thermometers have been obtained from such a melt by Surveying the resistivities between 4.2°K. and 1.6°K. of samples from slices taken perpendicular to the vertical axis. Strips from the uppermost layer of the melt are found to have about 1.5 X 10$^{17}$ impurity centers/cm$^3$ and resistivities at 300°K. of about 0.08 ohm cm. From this group, a thermometer has been selected having a temperature coefficient of resistivity of -1 (degrees K.)$^{-1}$ at 4°K. and -3.70 (degrees K.)$^{-1}$ at 2°K. Its use in temperature measurement, reproducible to better than 0.01°K. throughout the helium range, is discussed.

61. Genser, Milton, and Worth B. Allred (Battelle Development Corp.).
SEMICONDUCTOR MATERIAL.
(C.A. 52, 9789d; see also Semiconductor Abstracts VI, No. 1516 (1958)).

AlSb semiconductors are prepared by incorporating Ta in AlSb by treating
Al and Sb in a Ta-lined container. The method produces semiconductor
material having a resistivity of 10^3-10^6 ohm-cm., which can be used as
thermistors, photoconductors, or infrared detectors. The semiconductor
has a carrier concentration of 10^{12}-10^{13} carriers per cm^2-cm. and can
be used in the band between 0.8 and 4.0 μm.

62. Gibson, W.T. (Standard Telephones and Cables, Ltd.).
THERMISTOR PRODUCTION.
Electrical Communication 30 (4), 263-70 (1953).

Methods of manufacture of bead, pellet, and rod type thermistors by
Standard Telephones and Cables, Ltd., are described. Raw materials
used are Ni, Mn and Cu oxides.

63. Gildart, Lee W.
SEMICONDUCTOR MATERIALS FOR THERMISTORS.
(C.A. 52, 14350d; see also Semiconductor Abstracts VI, No. 1515 (1958)).

A thermistor material is made from 96% by weight Sb₂S₃ and 4% As₂S₃,
both of at least 99.9% purity, by heating to about 650°C and then cooling.
The resistivity of the resulting alloy is 140 ohm-cm. at 25°C. An alloy
containing 75% Sb₂S₃ and 25% As₂S₃ has a resistivity of about 12,000
ohm-cm. at 25°C. These materials can be incorporated into thermometer
probes by melting a granular form of the composition and allowing it
to fuse around a pair of electrode tips.

64. Gizzi, A.J. (General Electric Co.).
THE GROWING THERMISTOR FIELD.
(Semiconductor Electronics 2, No. 2635 (1958)).

Present and future applications of thermistors are discussed and the
results of a thermistor market survey are presented. The present limita-
tions of thermistors are outlined.

65. Goodyear, Robert S.
THERMAL RESISTOR ELEMENTS FOR ELECTRICAL CIRCUIT APPLICATIONS.
Product Eng. 16, 93-5 (1945).
(C.A. 52, 26754a).

Graphs show characteristics and performance of thermal resistor ele-
ments formed from metallic oxides. These resistor elements, which con-
sist of metallic oxides pressed into disks, extruded rods, or formed
into tiny beads, can be used as small electric-circuit elements,
resistance of which changes with temperature. Some thermistors are
produced with resistance values ranging from a few ohms to several
megohms. The temperature of a thermistor may be changed by external
heating, or by heating due to passage of electric current, or by a separate heating coil in close contact with the thermistor element. Static characteristics of a directly heated thermistor relating voltage, current, resistance, and power are given. A directly heated thermistor can be used as a time delay device. Resistance thermometers, having a thermistor as the measuring element, are far more accurate than are thermocouples. Thermistors in similar bridge circuits can be used as flow meters or vacuum gages, or wherever the physical quantity to be measured can affect the flow of thermal energy from a hot body. Externally heated thermistors can be used as variable-resistance devices. Standard relays can be protected against high-voltage surge in the circuit by placing a directly heated thermistor in series with the winding.


A stable, resistor unit of high negative resistance-temperature coefficient comprises a self-supporting plate of consolidated, finely divided, heat-treated resistance material, the major constituent of which is one or more of the oxides of Fe, Ni, and Co, the plate having a thickness less than approximately 1/12 of its smallest surface dimension, and being provided with contact means including at least one heat-distributing member on opposite faces of the plate.


The temperature-sensitive electrical resistor (thermistor) is a recent development of the Bell Telephone Laboratories. Its most important property is an unusually high temperature-resistance coefficient as compared to conventional temperature-sensitive wire. It is particularly useful in problems involving measurement and control of temperature and related quantities. Report contains descriptions of various models of thermistors, their uses and associated circuits. A parts and accessories list for thermistor electronic thermometer, illustrations, a chart and a diagram are included.


The temperature coefficient of resistance of semiconductors is derived for materials of varying degrees of purity and for different temperature ranges. The temperature effects of p-n junctions are determined from the diffusion currents of the minority carriers.
69. Haayman, Pieter, Arie Bol, Frans C. Romeyn, and Evert J. W. Verwey
(to Hartford National Bank and Trust Co.).
**METHOD OF MANUFACTURING SEMICONDUCTIVE MATERIAL.**
U.S. 2,735,824, Feb. 21, 1956.
(Ceramic Abstracts 1956, 119j).

A resistor having a given value of specific resistance consists essentially of substantially homogeneous mixed crystals produced by heating a mixture of about 99 molecular % of ferric oxide and about 1 molecular % of titanium dioxide in air at a temperature of about 1200°.

**THERMISTORS IN TRANSISTOR CIRCUITS.**
Semiconductor Products 3 (8), 31-35 (1960).
(Eng. Index 1960, p. 1497).

Temperature compensation of transistor circuits can be accomplished inexpensively and with relative simplicity through the use of negative temperature coefficient resistors, thermistors. Thermistors are available in a wide range of resistance values and temperature coefficients. Time constants similar to transistor time constants are available. Characteristics of available thermistors are discussed together with methods for determination of characteristics and a description of how they should be specified. Some typical applications of thermistors in compensating transistor circuits are also discussed. Thermistors for temperature compensation are available in 3 basic types. All 3 are manufactured in a range of resistivities and temperature coefficients. The highest coefficient (steepest slope) is that of cobalt-Mn-Ni oxide types which are available in disc, rod, and washer form. These types possess negative temperature coefficients of approximately 3-5.2% per °C. Thermistors possessing intermediate slope are composed of Ti and Fe oxides and are available primarily in rod form. The negative temperature coefficient of these types can be from 1.3-2.5% per °C. The least change with temperature is that exhibited by the SiO types which are available in rod form and possess coefficients of 0.25-0.35 per °C. The resistance-temperature characteristic of a thermistor is specified by β value: \( R_T - R_0 = R_0 \beta (T - T_0) \), where \( T_0 \) = reference temperature in °K, \( R_0 \) = resistance at \( T_0 \), \( T \) = temperature of thermistor in °K, \( R_T \) = resistance at temperature \( T \), and \( \beta \) = beta value or resistance-temperature constant. Since the resistance-temperature characteristic of a thermistor is non-linear, \( \beta \) varies with temperature, generally increasing with temperature.

71. Inutuka, Hideo, and Syuziro Kawase (to General Electric Co.).
**SINTERED METALLIC COMPOSITIONS SUITABLE FOR TIME-DELAY RELAYING DEVICES.**
(C.A. 27, 8652).

CuO 95-60 and FeO or chromic oxide 5-40% are used in forming a sintered composition which has a negative coefficient of resistance and which is substantially nonporous and free from change with respect to negative temperature coefficient of resistance after having been heated to high
temperatures. U.S. 2,294,756 relates to a process of manufacturing resistances, which involves mixing a powdered oxide of a metal such as Cr₂O₃ with one or more powdered metals such as Cu and shaping the mixture and sintering it at 1000-1300° in the presence of an oxidizing medium such as O₂ to effect oxidation of the metal or metals.

SINTERED ELECTRICAL RESISTORS.
(C.A. 52, 6842f).
Resistors having a specific resistance range of 0.2-5750 ohm cm., and a temperature coefficient between -0.5 and -4.3%/°C. are described.

The composition of these resistors corresponds approximately to ABₙDₙ₋₁O₃ in which A is a mixture of at least 1 ion of the group La⁺⁺⁺, Nd⁺⁺⁺, and Pr⁺⁺⁺ and at least 1 ion of the group Ca⁺⁺, Sr⁺⁺, Ba⁺⁺, and Pb⁺⁺; B is at least 1 ion of the group Fe⁺⁺⁺, Cr⁺⁺⁺, and Co⁺⁺⁺; D is at least one ion of the group Fe⁺⁺, Cr⁺⁺, Co⁺⁺, and Mn⁺⁺; and n is >0, but <1. Co³⁺ and Co⁴⁺ are present only when at least 1 of the other metal ions besides those represented by A in the above formula is present, the elements Co, Fe, and Cr being present in both the tri- and quadrivalent states. Resistors are claimed in which the conductive phase contains: (1) Co in an amount up to 90 atomic % of the total of Fe, Cr, Co, and Mn; and (2) Al, Ti, and (or) Ni in a total amount up to 20 atomic % of the total of Fe, Cr, Co, and Mn. Also claimed is a resistor containing up to 10% flux.

73. Kaganov, M.A.
DETERMINING CHARACTERISTICS OF THERMISTORS USED IN MEASURING EQUIPMENT.
Priborostoenie No. 3, 5-7 (1960).
(Monthly Index of Russian Accessions 13, 2084 (1960)).

74. Kamiyama, Masahide, and Ziro Nara (Univ. Tokyo).
PROPERTIES OF Mn₃O₄-NiO THERMISTORS.
Ôyû Butsuri 21, 400-2 (1952).
(C.A. 47, 8505f).

Seven samples of thermistors were prepared from MnO₂ and NiO by sintering the mixtures of various ratios at 1300° (3 hours) and annealing at 500° (24 hours). MnO₃ changed to Mn₃O₄ on sintering. The electric resistance R was measured at various temperatures to calculate the activation energy E, which is for samples of MnO₂:NiO ratio in weight, (I) 1:0 (pure Mn₃O₄), 0.48 e.v., (II) 4:1, 0.37, (III) 3:2, 0.35, (IV) 2:3, 0.36, (V) 1:4, 0.46, and (VI) 0:1 (pure NiO), 0.61. There is a minimum of E at 713 corresponding to a minimum of R. The thermoelectric-power measurements at temperature difference 5 ~ 6° showed that I, II, and IV were of p-type and III, IV, and V were of n-type. The sample (VII) was of n-type below 200° and p-type above that temperature. X-ray powder photography showed that VII was composed of II and III and relative magnitude of R of II and III determined the type of VII. Mn₂NiO₄ is contained in every sample except I and VI, and plays an important role in these thermistors.
Literature search cont'd.


SEMICONDUCTOR THERMISTORS.

Izv. vys. ucheb. zav. energ. 2 (6), 133-4 (1959).

(Monthly Index of Russian Accessions 15, 316 (1960)).


76. Kasperovich, A.S.

METHODS FOR DETERMINING THE THERMAL CONSTANT OF TIME OF A THERMISTOR.

Trudy Inst. energ. ANSRR 6, 222-7 (1958).

(Monthly Index of Russian Accessions 15, 680 (1960)).

77. Keonjian, E., and J.S. Schaffner. (General Electric Co., Syracuse, N.Y.)

SHAPING THE CHARACTERISTICS OF TEMPERATURE-SENSITIVE ELEMENTS.

Communication and Electronics 1924, 396-400.

A method for shaping the characteristics of temperature-sensitive elements is described. The proposed method can be applied to any temperature-sensitive element provided the coefficient of the temperature-sensitive element is larger than that of the desired characteristic and the characteristic of the temperature-sensitive element does not change with the applied voltage.


TEMPERATURE MEASUREMENT BY SEMICONDUCTOR RESISTANCE THERMOMETERS.


(Physica Abstracts 52, No. 1438 (1949)).

Resistance-temperature characteristics are given for UO₂ over the range 0-100°C. Thermistor resistance thermometer elements are described and rate of response demonstrated. Sensitivity and scale distribution are tabulated for various temperature ranges, obtained in conjunction with the usual circuit bridge.


HOT CONDUCTORS.

Elektrichesctvo 1947, No. 3, 20-7; Chem. Zentr. (Russian Zone Ed.)

1948, II, 1158.

(C.A. 44, 8801f).

Methods of preparation are given for hot conductors (with a negative temperature coefficient) of the following materials and the properties of such conductors are discussed: U oxide (obtained by heating UO₂-(NO₃)₃-xH₂O to 500°C), CuO (from NaOH + CuSO₄ solution, heated to 90°C), a mixture of Cu and Mn oxides (addition of NH₃ to a solution of CuSO₄ and MnSO₄ at 80°C), a mixture of Ti and Mg oxides, and Ag₂S (from AgNO₃ + H₂S). The conductors were formed from the compressed powders, which were then heated for varying periods depending upon the resistance values. Electrical connections were made (except with the CuO conductor) by reduction of an applied Ag₂S solution. Tables are given which report the resistance values obtained, the relation between conductivity and temperature, and the manner in which this latter relation varies with the duration and temperature of calcination (minutes to hours and 500-1300°C). Within a definite range the conductivity of the CuO-MnO conductor scarcely varied with the composition.
of the mixture or with the temperature of calcination. At a conductivity of $10^{-1}$ reciprocal ohms per cm, the temperature coefficient of this hot conductor corresponded to that of an ordinary hot conductor at a conductivity of $10^{-3}$ to $10^{-4}$ reciprocal ohms per cm. Other materials recommended as suitable for hot conductors were CuOCrO$_3$, Mn$_3$O$_4$, NiO, and PbSe.


Germanium samples purified by high vacuum treatment have been alloyed by adding from 0.001% to 1.0% of metallic impurities. The electrical conductivity and transverse Hall effect of these samples have been investigated over temperatures ranging from -180°C to 650°C. Plotting log $\rho$ (resistivity) and log $R$. (Hall constant) against $1/T$ shows that the resistivity at low temperatures decreases with increasing temperature, increases around room temperature and then drops sharply with a slope identical for the various samples. The Hall curves indicate electron (N type) or hole (P type) conduction, depending upon the type of impurity. P type samples show reversal of Hall effect, and the slope at high temperatures is identical for all samples, P and N alike, indicating that germanium behaves at low temperatures as an impurity semiconductor, but is at high temperatures an intrinsic semiconductor with energy level separation of about 0.76 volt. Hall values show that the number of current carriers ranges from about $10^{13}$ up to $10^{19}$ per cc. The temperature behavior of mobility, determined by $R/\rho$, cannot be explained as due to lattice scattering alone, but indicates the existence of another scattering mechanism, especially at low temperatures.

82. Lark-Horovitz, and V.A. Johnson (Purdue University). THEORY OF RESISTIVITY IN GERMANIUM ALLOYS. Physical Reviews 69, 258 (1946).

The temperature dependence of resistivity of germanium alloys is considered in three ranges: (1) the impurity range of low temperatures with conduction due to impurity electrons or holes, (2) the transition range with contributions from both impurity and intrinsic electrons and holes, and (3) the intrinsic range of temperatures so high that the numbers of electrons and holes are equal. Resistivity in the impurity range is the sum of resistivity due to lattice scattering (proportional to $T^2$) and resistivity due to impurity scattering. Mean-free-path calculations indicate that the impurity mean-free-path increases and the lattice mean-free-path decreases with rising temperature. At a given temperature the lattice mean-free-path
Literature search cont’d.

is the same for all germanium samples, but the impurity mean-free-path varies widely with impurity content.

83. Loman, Geo. T. (to Bell Telephone Labs, Inc.).

THERMISTORS.
U.S. 2,694,040, Nov. 9, 1954.
(C.A. 49, 2226d; Semiconductor Abstracts 3, No. 858 (1955)).

Stable thermistors with a large temperature coefficient of resistance are made from combinations of oxides of Ni, Mn, and Fe in the system
NiMn$_3$-$_4$ oxides: NiFe$_2$-$_4$ oxide by heating at about 1000° for 16 hours a typical mixture contains NiO 14.94, Fe$_2$O$_3$ 15.97, and Mn$_2$O$_3$ 15.79 grams. The oxides are mixed in a colloid mill with distilled H$_2$O acidified with AcOH for better wetting. After filtering, the resulting cake is dried and screened and a binder, e.g. 5 grams iso-Bu methacrylate/100 grams of oxides, is added together with a solvent, e.g. 1 gram xylene/gram of oxide. Dimethyl phthalate in a ratio of 5 cc./100 grams of oxides can be added before a second screening to control the consistency of the mixture. Resistor bodies of the disk type are prepared from the screened cake by pressing approximately 1 gram of material in a 1/2 inch diameter die at about 12,700 pounds/sq. inch. The pressed disks are heat treated in air at 500° for about 4 hours to remove binder after individually burying each disk in a finely divided refractory, e.g. 120-mesh Alundum. Sintering takes place at 1000-1400° in 16 hours. Each face is covered with a layer of conductive material, e.g. a paste of finely divided Ag or Pt mixed with the above binder and cured at 600°.

84. Meyer-Hartwig, E., and H. Federspiel (Terlano, Italy).

MANUFACTURE, REGULATING ACTION, AND APPLICATION OF ELECTRIC RESISTORS.
(C.A. 45, 4628a).

Resistance materials with negative temperature coefficients in which the current flow is due to electrons and not to ions, which with direct current cause decomposition, are described. The best materials for the manufacture of thermistors are oxides of U, Cu, Cr, Ni, Mn, and mixed oxides with a spinel basis, and products on a metalloceramic basis of a special composition. The maximum temperature of operation is for pure U oxide in a protective tube at 600°, pure Cu oxide at 220° in air, mixed oxides of Cr, Ni, Mn, Co at 300° in a protective tube and at 150° in air, spinels (Mn, Ti) 500° in a protective tube, and for capillary thermistors, i.e., mixtures of metals and nonmetallic substances, 600° in a protective tube and 450° in air. Methods of manufacture, properties, current control, and fields of application are described. Fifteen references.


PRESSURE COEFFICIENT OF RESISTANCE OF THERMISTORS.
(Physics Abstracts 55, No. 6473 (1952)).

The pressure coefficient of resistance for one type of thermistor thermometer was determined up to pressures of 2000 pounds per inch.
at temperatures of 0°, 25° and 50°. The pressure coefficient at any given pressure appeared to be independent of temperature but changed rapidly with pressure, decreasing with increased pressure from \(-3 \times 10^{-6}\) per pound per sq. inch at atmospheric pressure to \(-1.5 \times 10^{-6}\) per pound per inch\(^2\) at 1000 pounds per inch\(^2\) and approaching zero value at 2000 pounds per inch\(^2\)...


Resistors with negative temperature coefficient are made out of TiO\(_2\), partially decomposed to Ti metal by vacuum firing near the melting point. Hard, blue-to-black colored ceramic bodies are obtained by compressing pure TiO\(_2\), in vacuo, heating 1500-1640°, and later in an inert gas such as He, Ne, or Ar.


A thermistor film was obtained by evaporating a rod thermistor (baked mixture of Co\(_3\)O\(_4\) and CuO and of Mn\(_3\)O\(_4\) and NiO) onto a plate of fused quartz at 1500° and 10\(^{-3}\) mm. Hg and heat-treating in air (500-600°, 3-5 hours for the Co-Cu system and 250-350°, 3-5 hours for the Mn-Ni system). The temperature dependence of the electrical resistance of the films had the same trend as that of the original rod. Electron diffraction studies made after growing the particles in the film by sintering showed that the oxides had the spinel structure. The use of the films as infrared bolometers is suggested, their response being 0.07-0.12 volt (r.m.s.) per watt (at 15 cycles per second) and the time constant 0.05-0.09 second.


Thermistors covering a wide temperature range and with a reproducible temperature coefficient of resistance are manufactured from stable, spinel-type compounds, such as NiMn\(_2\)O\(_4\) - NiFe\(_2\)O\(_4\) or NiMn\(_4\)O\(_6\) - NiFe\(_4\)O\(_6\), which are mutually soluble in all proportions. The concentration of O varies with the partial pressure and sintering temperature. NiO 18.67, Mn\(_2\)O\(_3\) 19.73, and Fe\(_2\)O\(_3\) 19.96 grams (1μ particles) are mixed in a colloid mill with distilled H\(_2\)O and AcOH (or make slightly basic). After thorough mixing petroleum wax in triethanolamine is added; the binder is precipitated onto particles. The mixture is then filtered and pressed to the desired form, wax is evaporated at 300°, and the body is sintered in air at 1200° for 16 hours. The recommended atomic ratio for (Mn plus Fe)/Ni is 2-4; this gives a specific resistance of 3 \(\times 10^3\) to 2 \(\times 10^7\) ohm cm. at 25°; when the ratio is 2, the temperature-dependence factor is approximately 3500-4600°, while at 4, it is approximately 3700-3800°.
Three general types and shape factor are represented in the 205 thermistors described: rods, discs, and threads. They were prepared in 18 different batches with varying heat treatments; the electrical properties varied over wide limits. The resistance at 25°C. ran from 15 ohms to 44 megohms and the temperature coefficients of resistance at 25°C. from -1.0 to 5.5%/°C. The material used is magnesium orthotitanate (2 MgO).\(\text{TiO}_2\) \(\text{(I)}\). I is an insulator with specific resistivity of \(10^{12}\) ohm-cm. but is converted to a semiconductor with a room temperature specific resistivity of \(10^4\) ohm cm. by heat treatment in a reducing atmosphere. Since O is given off the O atoms may leave vacancies in the crystal lattice which act as impurity centers or reduction may take place on the surface with excess metallic atoms subsequently diffusing into interstitial positions in the lattice. At low temperatures all of the electrons are bound and resistance is high; as the temperature increases more electrons break their bonds and the resistance decreases. \(R = Ae^{-\frac{b}{T}}\) where A is a constant containing the shape factor; T is temperature in °K., and b is a constant proportional to the activation energy. For our thermistor the activation energy averages about 0.3 electron volts. Temperature coefficient of resistance \(\alpha = \frac{R}{T}\). Mg orthotitanate was prepared from MgO and TiO\(_2\) (50.16% MgO and 49.84% TiO\(_2\)) mixed by tumbling 30 minutes then run through a hammer mill. The powder was pressed into square shapes and heated to 1050°C., ground to pass a 120 mesh screen and formed into thermistor shapes. Disks were formed by the dry-press method, rods by tamping in a brass tube and threads by application of a mixture with nitrocellulose to a nylon thread. Each was fired at 1000-1200°C. They were then reduced in H at 1350-1450°C. Contacts were fixed before or after firing depending on type used.

90. Nechev, G.N.
TEMPERATURE STABILIZATION OF THERMISTORS.
Atom i telen. 21 (4), 548-54 (1960).
(Monthly Index of Russian Accessions 13, 2084 (1960)).

91. New York University.
INVESTIGATION AND RESEARCH TOWARD DEVELOPMENT OF A SEMI-CONDUCTING CERAMIC MATERIAL.
AD-147,452.
(Astia Bibliography).

This study concerns a semiconducting ceramic material with temperature coefficient of resistance as near zero as possible from minus 40°C. to plus 35°C. Materials used are porcelain (flint), steatite (talc) and zircon porcelain.
This investigation was made in view of preparation of thermistor thermoelements for the measurement of high temperatures. ZnO and Al₂O₃ were investigated to 1300°C. The powders were pressed into a steel tube, together with a Nichrome wire serving as a second electrode, at a pressure of 5000 atmospheres. The resistance of Al₂O₃ consists of two parts: volume resistance with electronic conductivity and barrier-layer resistance with hole conductivity. Aging increases the resistance by an increase of barrier-layer resistance. At 1500°C the resistance of Al₂O₃ is of the order of 10⁴ ohms. ZnO has a low resistance above 1000°C, and it is thus unsuitable for thermistors at high temperatures.


Pressed rods of Al₂O₃, MgO, ZnO, and MgO + Al₂O₃ were tested for use as high-temperature pyrometers at temperatures up to 1570°C. The results obtained with Al₂O₃ were readily reproducible with a scattering of ± 10°C for the different samples, but the values obtained with MgO were not dependable.


The semiconductor resistance thermometer (S.R.T.) consisted of Al₂O₃ elements between SiC electrodes. It was shown that although the conductivity of the measuring elements decreased (sometimes by a factor of 10) during the first 50 hours at 1500°C the stability after this preliminary annealing was quite satisfactory. It is concluded that SRT's of this type are suitable for temperature measurements at 1000-1600°C.


Resistance elements are prepared by sintering a mixture of Mg and Ti oxides at 1700-2000°C in a gas stream containing H and O, the O concentration being in excess of 0.003% of the H concentration...
96. Patent-Treuhand-Gesellschaft für elektrische Glühlampen m.b.H.
ELECTRIC RESISTANCES HAVING A NEGATIVE TEMPERATURE COEFFICIENT.
Ger. T12,538, Sept. 25, 1941.
(C.A. 17, 4310*).

The resistances are made of a mixture of nonconducting oxides and conducting lower oxides. At the most 75% of the mix is TiO₂ and (or) Cb₂O₃ and (or) V₂O₅. The nature of these oxides is such that they do not reduce to metal at 800-1500° in an atmosphere of H₂. The rest of the mix is made up of an alkaline earth oxide, especially MgO. The alkaline earth oxide should not decompose during the preparation nor in operating the resistance and should have a specific resistance 10⁵ ohms per cm. The mix is shaped and fired first in an atmosphere containing O₂ and then in a neutral or reducing atmosphere. The firing is such that the Ti, Cb and (or) V oxides are reduced to lower oxides having a specific resistance of 10 ohms per cm.

97. Paterson, W.L.
CHARTS GIVE THERMAL CHANGES OF THERMISTORS.

A chart showing the relation of resistance to temperature for thermistors, based on the equation \( R = A e^B \) where \( R \) is resistance, \( A \) is a constant having the same dimensions as \( R \); \( B \) is a constant depending on activation energy and \( T \) is absolute temperature.

98. Pearson, G.L., and J. Bardeen (Bell Telephone Labs, inc.).
ELECTRICAL PROPERTIES OF PURE SILICON AND SILICON ALLOYS CONTAINING BORON AND PHOSPHORUS.
Physical Reviews 75, 865-883 (1949).
(Physics Abstracts 52, No. 6199 (1949)).

Measurements of electrical resistivity and Hall effect over a temperature range from 0.0005 to 1% \( \beta \) (p-type impurity) or \( \rho \) (n-type impurity) were used. X-ray measurement showed that the impurity centers occupy lattice positions. The width of the forbidden band was found to be 1.12 e.v. The activation energy associated with acceptor impurity levels is 0.08 e.v. for very low impurity contents, increases with increasing impurity content and vanishes for concentrations above 5 X 10¹⁸ centers per cm³. Activation energies associated with donor levels are generally smaller than those of acceptor levels...

RESULTS OF AN EXPERIMENTAL INVESTIGATION OF THERMOELECTROMOTIVE FORCE IN THERMISTORS.
(Nuclear Science Abstracts 14, No. 20723 (1960)).

Results are given of an experimental investigation of the thermal and electrical properties of disc thermistors made from AgCl, CuO, Ag₂S, Mn₂O₃ and MnO. Results lead to the conclusion that it is possible to use these thermistors for the measurement of heat flow...
100. Pereleshina, A.P.
PHYSICOCHEMICAL PROPERTIES OF THERMISTORS MADE OF MANGANESE DIOXIDE.
AD-257,908.
(Astia Bibliography).

The results of experimental investigations of the electrical and thermal properties of thermistors made of Mn oxides show them to be solid electrolytes. Theoretical formulas are derived for temperature dependence of coefficient of heat conductivity and thermal e.m.f. and dependence of resistance on temperature and of charge mobility on activation energy are empirically characterized. In thermistors of Mn oxides, oxygen ions are the charge carriers. In the presence of a temperature gradient in a thermistor semiconducting substance a non-uniform concentration of O ions appears which leads to the appearance of an e.m.f. in the thermistor.

101. Perrone, Guy
PROPERTIES AND APPLICATIONS OF THERMISTORS.
Chaleur et Ind. 36, No. 354, 3-13 (1955).
(C.A. 42, 4909c).

A review with 20 references.

102. Peters, Melville F. (to Petcar Research Corp.).
THERMISTOR FOR HIGH-TEMPERATURE MONITORS.
U.S. 2,596,284, May 13, 1952.
(C.A. 46, 7914e).

The skeleton of the thermistor is made by sintering a mixture of steatite and flour at 1800°F. (cf. U.S. 2,495,867, C.A. 44, 2724g). This skeleton is impregnated with a temperature-sensitive material, such as e.g. I sulfide dissolved in alcohol, after which it is dried at 200-300°F. Other soluble metal compounds which may be used to impregnate the skeleton are Mn, Fe, or Ni acetates, FeCl₂, ZnCl₂, Fe sulfate, Na₂Cr₂O₇, or any other soluble metallic compound which upon heating changes into an insoluble oxide. The temperature responsiveness of the thermistor will depend upon the used impregnating material, the concentration of the solution, and the number of impregnations. Increasing the concentration of the impregnating solution lowers the resistance of the thermistor and causes it to become conductive at a lower temperature.

103. Petrov, I.N.
THERMISTORS.
Poluprovodnikovye Pribory (Semiconductor Devices) Moscow, 1957.
Chapter 2 Section 5. LC or SLA mi.-P2.40 pH 3.30, No. 60-13552.
(Translation).
(OTS Selective Bibliography - Semiconductors SB-429).

Information is reviewed on properties, types, and practical uses of thermistors.
A sintered electrical resistance having a high negative temperature coefficient consists of mixed crystals of Fe$_3$O$_4$ and a spinel $R'0_2$, in which $R$ is a bivalent element and $R'$ a quadrivalent element, such as Mg$_2$TiO$_4$, Zn$_2$TiO$_4$, Ni$_2$TiO$_4$, and Zn$_2$SnO$_4$, or their mixed crystals, less than half the molecules being Fe$_3$O$_4$. To obtain a resistance having a low negative temperature coefficient, more than half the molecules should be Fe$_3$O$_4$, in which case a second phase, consisting of an insulating bivalent oxide can be present to raise the specific resistance and regulate the temperature coefficient, which oxide dissolves badly in the spinel mass and is distributed evenly therein, such as MgO and ZrO$_2$. If the spinel phase is not supersaturated with a second phase at about 500°, separation of a second phase above 500°, effecting undesired changes of the resistance properties, is impossible. At lower temperature the velocity of separation is so low that no trouble can be caused. In an example 0.376 mole Fe$_2$O$_3$, 1.5 mole NiO, and 0.75 mole TiO$_2$ are ground with alcohol in a ball mill for 16 hours. The dried mass is worked with a 20% solution of the Me methacrylate in methyl glycol acetate to a moldable mass and pressed into bars of 4 mm. diameter, which are sintered at 1300° for 1 hour in an atmosphere of pure N (O content about 0.1%) and then are rapidly cooled in a N current. At 22° the specific resistance is 22,000 ohms cm., at 138° 900 ohms cm., at 227° 200 ohms cm., and at 332° 50 ohms cm.

A resistance having a negative temperature coefficient and formed from a sintered semiconductive mixture containing ceramic material and a preponderating amount of Si or Si alloy is simultaneously provided with end contacts by assembling together the resistance material and carbonaceous contact masses, the whole unit being heated at a temperature sufficient to sinter the mixture but not fuse the Si or Si alloy and cause the mixture and the contact masses to adhere. In an example, Fe-Si, clay and tragacanth are powdered and mixed into a paste with H$_2$O and pressed into rods and small graphite blocks are pressed into the ends; the rods are dried and sintered at 1300° in a reducing gas atmosphere. Small rods of Ni may serve as leads.

A heated thermistor comprises a rod-shaped ceramic thermistor, a layer of ceramic insulation surrounding the thermistor, and a layer of graphite surrounding the insulation layer.
107. Prigent, J.
INVESTIGATION OF URANIUM OXIDE THERMISTOR.
(Physics Abstracts 52, No. 2513 (1949); C.A. 42, 7346h).

Amorphous UO$_2$ is preferred, as it makes better contact than the
crystalline form; details of preparation for uranyl nitrate are
given. Resistance varies with pressure according to $1/R = c\beta C$,
c and C being constants for a given thermistor. Resistance in
opposite directions vary by about 1 to 500. Resistance decreases
with time at first, taking 30 minutes to reach a constant value
5% less than the original value. Resistance at 50 volts is approxi-
mately 1/2 that at 1 volt. Resistance at 100°C. is approximately
1/4 that at 50°C. Thermistors may be used as resistance thermometers
reading to 1/160°C., or as sensitive infrared bolometers. Their
high resistance makes them unsuitable for cm. and wave detectors.

108. Rodgers, G.B., and F.A. Raal (Diamond Research Lab., Johannesburg,
South Africa).
SEMICONDUCTING DIAMONDS AS THERMISTORS.

LOW-RESISTANCE THERMISTORS AS ULTRA-COLD THERMOMETERS.
Electronic Inds. Tele-Tech 16 (1), 55, 142-3, 146-7 (1957).
(C.A. 52, 1695h).

At liquid-O temperatures new thermistors exhibit resistance
characteristics as low as 300 ohm-cm. and temperature coefficients
as high as 35% per degree.

110. Sanborn, Paul H.
FERRIC OXIDE-TITANIUM DIOXIDE MIXTURES AS ELECTRICAL CONDUCTORS.
(C.A. 46, 5288a).

Fe$_2$O$_3$-TiO$_2$ with clay is formed into a ceramic body, leaving a
negative temperature coefficient of resistance, by the usual methods.
The articles are useful in the measurement of temperatures, tempera-
ture control of relays, for voltage regulation, as thermoresponsive
resistors for motor starting, as lightning arrestors, etc.

111. Sapoff, Meyer.
THE THERMISTOR - A SPECIALIZED SEMICONDUCTOR SENSOR.

Although the structure and mechanism of conduction in monocristalline
semiconductors is fairly well understood, their use as thermistors
has been very limited to date. At present almost all commercial
thermistors are fabricated from oxide semiconductors. Unfortunately
no quantitative theory exists which adequately explains the behavior
of oxide semiconductors. Some qualitative insight may be obtained
by comparing their properties with those of single-crystal doped
elements...Oxides of NiO and Cu$_2$O are called "p" type semiconductors,
of ZnO and TiO$_2$, "n" type.
PROCESSING OF POSITIVE TEMPERATURE COEFFICIENT THERMISTORS.
J. Am. Ceramic Soc. 43, 297-301 (1960).
(Ceramic Abstracts 1960, 163d).

The controlled addition of impurities such as La in ceramic Ba titanate and BaSi titanate semiconductor systems results in thermistor materials having large positive temperature coefficient of resistance over controlled temperature ranges (9-6-12.5% per °C. at 25-50°C). The resistivity in these systems is sensitive to the concentration of the trace additives, to undesirable impurities in the raw materials and to the sintering and maturing conditions. Ceramic procedures well adapted to prepare these thermistors reproducibly have been developed.

THERMISTORS, THEIR THEORY, MANUFACTURE AND APPLICATION.

114. Schiff, R.I.
THERMISTORS.

THERMONEGATIVE RESISTORS.
(C.A. 74, 2264g).

A resistor with a descending characteristic may comprise a wire- or ribbon-like conductor of material such as CuO or UO₂ embedded in a refractory insulating material.

MANGANESE OXIDE THERMISTORS. Special Translation from "Physico Chemical Properties of Thermistors Made of Manganese Oxides".
AD-257,908, June 22, 1961 (OTS 3.60).
(TAB 61-2-4, p. 23 (1969)).

Experimental investigation of the electrical and thermal properties of thermistors made on manganese oxides confirms the correctness of the hypothesis which states that the investigated specimens are actually solid electrolytes. Theoretical formulas are derived for the temperature dependence of the coefficient of heat conductivity and thermal e.m.f. Empirical laws are determined to characterize the dependence of the resistance of specimens on temperature, and the dependence of the charge-carrier mobility in the specimens on activation energy. In thermistors made of manganese oxides, O ions, which are the charge carriers, also take an active part in the process of heat transport. In the presence of a temperature gradient in a thermistor semiconducting substance, a nonuniform concentration...
of O ions appears which leads to the appearance of an e.m.f. in
the thermistor.

117. Schwartzwalder, Karl, Alexander S. Rulka, and Robert W. Smith
(to General Motors Corp.).
RESISTOR FOR TEMPERATURE INDICATORS.
(C.A. 42, 94071).
A ceramic resistor having a large and uniform temperature
coefficient of electrical resistance and other desirable properties
is composed of magnetite 71.1, Fe₂O₃ 8.9, borosilicate glass
(Pyrex) 10 and Mg borate glass 10%. It is chemically stable and
unaffected by oil or grease, but slightly soluble in H₂O and must
be protected from it. Elimination of Mg borate glass from the
recipe improves stability to H₂O.

118. Shashkov, A.G.
POLARIZED THERMISTOR.
(Monthly Index of Russian Accessions 12, 680 (1960)).

119. Sheftel, I.T.
SEMICONDUCTORS AS THERMALLY SENSITIVE RESISTORS (THERMISTORS).
1, 249-90 (1957).
(C.A. 54, 61h).

120. Silver, Roland (Electronics Corp. of America).
RESEARCH IN THERMAL-RESISTANCE EFFECTS.
AD-130,844, June 1957.
(Astia Bibliography).
A study has been made to investigate the properties of film-type
thermistors and their application to computer circuitry. An ex-
tensive bibliography, and a discussion of theory derived from these
references, are presented together with basic circuitry for various
applications. A few thermistors of the thin-film semiconductor
type were constructed, including an array of ten elements. Tests
made on these experimental units corroborated the theoretical data
and computations, and established the feasibility of using therm-
istor as medium-speed switching units of very small size and little
weight in computer circuitry. Further studies of many more poten-
tial thermistor materials, and of practical applications of them
are recommended.

121. Skaupy, F. (Berlin-Lichterfelde, Germany).
THE HISTORICAL DEVELOPMENT OF THE SEMICONDUCTOR PROBLEM AND OF
SEMICONDUCTOR RESISTORS.
(C.A. 44, 9832f).
Historical data on the development of semiconductors in Germany.
Skaupy proposed as early as 1920 the use of ZnO as resistor with
negative temperature coefficient.
122. Smith, H.A.
TEMPERATURE MEASUREMENT BY THERMISTORS AS APPLICABLE TO TELEMETRY.
AD-123,600, Dec. 1956.
(Astia Bibliography).
This memo describes the theory, and practical application, of thermistors used in potential dividing devices to give voltage outputs related to temperature. These, unamplified, can be tele-metered, and recorded by ground or remote equipment.

123. Smith, Otto J.M. (University of California, Berkeley).
THERMISTORS. PART I. STATIC CHARACTERISTICS.
The fundamental thermistor equations are presented as functions only of electrical quantities and constants, and independent of temperature measurements. The universal curves allow one to predict the static characteristics of thermistors as circuit elements. The two most significant characteristics are the power coefficient of resistance and the negative incremental resistance. These can be computed or read from the universal curves, if the resistance and 4 parametric constants are known. These 5 values can be determined from 6 convenient measurements: the resistance and change of resistance with current at very low current, at the maximum voltage point, and at maximum current.

124. Smith, Otto J.M.
THERMISTORS. DYNAMIC CHARACTERISTICS. PART II.
A thermistor is a resistance with a high negative temperature coefficient of resistance. They are semiconductors similar to Thyrite except that their resistance changes with temperature instead of applied voltage. They are usually made of fused oxides of Ni, Mn and Co, and are extremely stable for long lengths of time. They can be used for sensitive temperature inducing devices because the resistance change is about 4% per °C. They can be used as sensitive bolometers for microwaves or visible light if connected in a bridge current to balance out ambient temperature changes. A thermistor can be used as a non-linear circuit element in many of the same applications as a C-filament light bulb. They can be used as voltage regulators, amplitude stabilizers, volume expanders and volume control devices. The thermistor sinusoidal and transient response characteristics are presented as a function of the dissipation constant and incremental resistance. Equations are given for time constant, critical frequency, equivalent inductance, and Q. The construction of a low frequency oscillator is discussed, as well as possible non-linear filter applications.
125. Smith, R.A.

**THERMISTORS, VARISTORS AND OTHER NON-LINEAR RESISTORS.**


The thermistor consists of a small bead of semiconducting material used as a bolometer. In commercial units a mixture of semiconductors chosen to give a high temperature coefficient of resistance but not too high a resistance is used. The bolometer element generally consists of a mixture of the oxides of Ni, Mn and Co. Non-ohmic resistors from granulated SiC are widely used. These are commonly called "varistors".

126. Smith, R.W. (AC Spark Plug Division, General Motors Corp.).

**THERMISTORS - THE STORY OF THERMALLY SENSITIVE SEMICONDUCTORS.**

Ceramic Ind. 74 (5), 79-81; (6), 84-7; 75 (8), 74-7 (1960).

127. Smith, Robert W., and Karl Schwartwalder (to General Motors Corp.).

**AN ELECTRIC RESISTOR WITH A NEGATIVE TEMPERATURE COEFFICIENT.**

U.S. 2,786,819, March 26, 1957.

A resistor was prepared which can be used as a thermistor having a negative temperature coefficient. Partially oxidized metal oxides, such as NiO, Ni$_2$O$_3$, Fe$_2$O$_3$, FeO.Fe$_2$O$_3$, Co$_3$O$_4$ and Ti$_2$O$_3$ in various ratios, were used, with a glazing substance to exclude O from the individual particles. Such a thermistor composed of Mg borate glass 15, No. 774 Pyrex 25, Sierra talc 22, and FeO.Fe$_2$O$_3$ 38%, when cycled between room temperature and 1100°F. for 48 hours at 40 cycles per hour, underwent a 12% decrease in resistance.

128. Société Provilis.

**SEMICONDUCTORS.**

Fr. 991,891, Oct. 11, 1951.

129. Standard Telephone and Cables, Ltd.

**ELECTRIC RESISTANCES.**


A resistor having a high negative temperature coefficient comprises a granule consisting of a crystal of B or a cluster of B crystals, permanently joined to lead wires, e.g., of Pt, Pd, or Fe, by a compound of B and the metal lead wire...
A resistance element consisting of a metallic compound having a negative temperature coefficient is provided with metal contacts that are secured initially to the metal body, which is subsequently treated to form the compound, the contacts being formed of a metal or alloy unaffected by the treatment. Thus, Ag$_2$S resistances are produced by binding contact wires of W or a Ni-Cr alloy, e.g., "Brightray," "Nichrome," tightly around grooves in a Ag rod, the assembly being then exposed to S vapor at 300-400° for 15 minutes or longer to convert the Ag to Ag$_2$S after which the element is stabilized by being heated to 200-300° in N or other inert gas.

A material of negative temperature coefficient of resistance comprises the combined oxides of Ni, Mn and Co. The specific resistance of the material depends upon the relative proportions of Ni, Mn and Co in the combination.

Thermistors having resistances of the order of 100 ohms/cm. at 300-1000° are described. They consist of a fritted, homogeneous mass of finely divided metallic oxides of Ti, V, Cr, Mn, Fe, Co, Ni, or Cu, along with 2-10% by weight of a refractory oxide, e.g., Al$_2$O$_3$ and/or SiO$_2$. Preferably, the metallic oxide mixture consists partly of Mn$_2$O$_3$. These mixtures should also contain Mn 40-80, Ni 5-14, Co 10-17 and Fe 1-10%. If Cu oxides are present, the amount of Cu should be 8-30%. The thermists must be coated with a refractory, gas-tight, chemically resistant material, e.g., a silicate or borosilicate of Pb or Zn. For example, 67% Mn$_2$O$_3$ and 15% NiO were mixed with 18% Al$_2$O$_3$ in a jar made of nonoxidizable alloy having great hardness. Balls of tungsten carbide were put into the mixture and the jar was partially filled with liquid. After tightly closing the jar, it was rotated for about 400 hours. The powder obtained was molded under very high pressure into small rods and then fired in a special furnace. Connections were then fixed to the rods which were then stabilized. The material had a resistivity of 100 ohms/cm. and a temperature coefficient of 0.014 ohms/ohm/°C. at 430°. For use at high temperatures, a mixture of PbO 55, ZnO 20, Al$_2$O$_3$ 10, SiO$_2$ 5, and B$_2$O$_3$ 10% was ball-milled for 50 hours, made into a paste with water, applied as a coating, and fired at 650°.

The variation in resistance, expressed as the fractional change \( \Delta R/\rho_0 \) of a thermistor kept at a constant temperature and subjected to various pressures \( P \) up to 5000 kg cm\(^{-2}\), is represented by the equation \( \Delta R/\rho_0 = 4.6 \times 10^{-6} P \). This relation holds almost exactly for any given temperature in the range 30-70°C. The thermistor-controlled thermostatic bath used in the experiments is described.


A Ge resistance thermometer with high sensitivity and stability in the absolute zero temperature range has been developed by J.E. Kunzler, T.H. Geballe, and G.W. Hull of Bell Telephone Labs...

A very small "bridge" cut from a single crystal of As-doped Ge is the basic element...Ge can be doped with As to produce a high and fairly constant temperature coefficient of resistance of about 1 ohm at room temperature, 14 ohms at 10°K. and 216 ohms at 2°K. Both the temperature coefficient and the actual resistance vary widely with minute changes in the amount of doping. This makes it possible to fabricate a thermometer having any of a wide range of characteristics...

135. THERMISTOR DATA CHART. Electronic Design 6, 20-25, Apr. 30, 1958. (Semiconductor Electronics 2, No. 2634 (1958)).

A tabulation of thermistors produced by several manufacturers is presented. The characteristics (cold resistance, dissipation constant, time constant, and temperature coefficient) listed in the table are described.


Measurements of the temperature dependence of electrical resistivity in p- and n-type iron-doped Ge crystals indicate that Fe introduces impurity levels in Ge at 0.34 ±0.02 e.v. from the conduction band. Such samples show very high resistivity at 77°K...
137. Tyler, W.W., R. Newman, and H.H. Woodbury (General Electric Research Lab, Schenectady).
PROPERTIES OF GERMANIUM DOPED WITH COBALT.

Measurements of temperature dependence of electrical resistivity in n- and p-type cobalt-doped Ge crystals indicate that Co introduces acceptor levels in Ge at 0.31 ± 0.01 e.v. from the conduction band and 0.25 ± 0.01 e.v. from the valence band...

BISTABLE SEMICONDUCTOR DEVICES.
(Semiconductor Electronics 2, No. 3535 (1959)).

Semiconductor devices which utilize the process of injection breakdown to provide 2 alternative values of resistance within a given range of operating values are described. The semiconductor crystal is doped with Fe, Co, Au, Zn or Cu to provide deep-lying energy levels within the forbidden band. Injection breakdown in the semiconductor is believed due to the cumulative effect of minority carriers which are injected at a junction. At liquid air or liquid nitrogen temperatures the resistance of these devices can be made to change by a factor of $10^9$ to $10^{10}$ by a pulse of voltage or light sufficient to initiate the breakdown. Both 2-terminal and 3-terminal devices are described...These devices are suitable for switches, relays, voltage regulators, over-voltage detectors, light detectors and temperature sensors.

139. Tyler, W.W., and R. Newman (to General Electric Co.).
GERMANIUM CURRENT CONTROLLING DEVICES.
(Semiconductor Electronics 2, No. 3686 (1959)).

A thermosensitive device and a photo-conductive cell which are highly sensitive in the temperature range from 0 to -200°C. and a high-voltage rectifier which can operate at frequencies up to 1/2 megacycle are described. All of these devices utilize Ge doped with a trace of Fe, which introduces deep-lying trapping levels and thereby radically alters the resistivity vs. temperature curve of the germanium...

140. Udalov, N.P.
MORE ON THE CALCULATION OF THERMISTOR CHARACTERISTICS.
Automatica 20 (6), 825-7 (1959).
(Monthly Index of Russian Accessions 13, 316 (1960)).

141. Udalov, N.P.
SEMICONDUCTOR THERMAL RESISTORS.
Inzh.-fiz. zhur. No. 1, 125-7 (1960).
(Monthly Index of Russian Accessions 13, 1465 (1960)).

Review (by L. Voloshin) of book of this title.
142. Ullman, Eleanor M. (Thermistor Corp. of America).
HOW TO USE THERMISTORS.
Electronic Equipment 1956, June.

A brief history of thermistors and recent developments in their manufacture, as well as design information, are given.

TEXACO THERMISTOR TESTS AND RESULTS.
(Astia Bibliography).

Six boron thermistors, 3 bead type and 3 cylindrical, were produced and evaluated. At 25° their properties are as follows:

<table>
<thead>
<tr>
<th>Temperature</th>
<th>TPJ-3</th>
<th>TPJ-4</th>
<th>TPJ-5</th>
<th>TRR-2</th>
<th>Vecco 51A22</th>
</tr>
</thead>
<tbody>
<tr>
<td>Voltage</td>
<td>9.32 v.</td>
<td>9.32 v.</td>
<td>9.32 v.</td>
<td>1.58 v.</td>
<td>0.40 v.</td>
</tr>
<tr>
<td>Current</td>
<td>066 μa</td>
<td>075 μa</td>
<td>062 μa</td>
<td>615 μa</td>
<td>3.10 μa</td>
</tr>
<tr>
<td>Resistance</td>
<td>141 meg</td>
<td>124 meg</td>
<td>150 meg</td>
<td>2.5 meg</td>
<td>1.29 K</td>
</tr>
<tr>
<td>dT/dT</td>
<td>0037 4°C</td>
<td>0052 4°C</td>
<td>0033 4°C</td>
<td>033 4°C</td>
<td>034 4°C</td>
</tr>
<tr>
<td>α</td>
<td>5.6%/°C</td>
<td>6.9%/°C</td>
<td>5.3%/°C</td>
<td>5.4%/°C</td>
<td>43%/°C</td>
</tr>
<tr>
<td>β</td>
<td>5000° K</td>
<td>6100° K</td>
<td>4700° K</td>
<td>4800° K</td>
<td>3800° K</td>
</tr>
</tbody>
</table>

144. Vander Beck, Roland R., Jr. (Carborundum Co.).
SILICON CARBIDE RESISTANCE BODIES AND METHODS OF MAKING.
(Ceramic Abstracts 1960, 90b).

A method of making recrystallized silicon carbide thermistor bodies comprises forming a body from a mixture of SiC particles and a small amount of a boron compound to provide 0.20 to 0.34% (weight) boron in the lattice of the SiC when recrystallized and firing the shape at 2000° to 2500° C. to recrystallize the SiC and introduce the boron into the SiC crystal structure.

145. Van Dover, James, and Norman F. Bechtold (U.S. Army Signal Research and Development Lab., Fort Monmouth, N.J.).
SURVEY OF THERMISTOR CHARACTERISTICS.

Industry is finding more and more use for thermistors...To make electronic circuits reliable, thermistors compensate for temperature changes, regulate current or voltage and control remote circuits. In the medical, meteorological and mechanical fields, thermistors are used for accurate measurement of temperature, pressure and liquid levels.
### TABLE I  NEGATIVE-TEMPERATURE-COEFFICIENT THERMISTORS

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ohms</td>
<td>watts</td>
<td>mw/°C.</td>
<td>°C.</td>
<td>°/°C.</td>
<td>sec.</td>
<td>inches</td>
<td></td>
</tr>
<tr>
<td>Glass-coated Bead</td>
<td>100-12 MΩ</td>
<td>0.2</td>
<td>0.09-0.8</td>
<td>500Ω</td>
<td>-3.1</td>
<td>0.5-3</td>
<td>0.006-0.110 diam</td>
<td>wind velocity, temp. gas analysis, liquid level, power control.</td>
</tr>
<tr>
<td>Bead in Container(a)</td>
<td>100-5.3 MΩ</td>
<td>0.2</td>
<td>1</td>
<td>500</td>
<td>-3.1</td>
<td>1-25</td>
<td>0.1 diam</td>
<td>time delay, medical probe, voltage control, very low temperature.</td>
</tr>
<tr>
<td>Disc</td>
<td>4 to 10 kΩ</td>
<td>4</td>
<td>3-800</td>
<td>150Ω</td>
<td>-3.8</td>
<td>2-200</td>
<td>0.1-0.1 diam</td>
<td>temp. comp., fire alarms, osc. ampl. stabilization, temperature control.</td>
</tr>
<tr>
<td>Rod</td>
<td>2%100Ω</td>
<td>2</td>
<td>2.5-6</td>
<td>150Ω</td>
<td>-3.8</td>
<td>20-95</td>
<td>1/4-2 long</td>
<td>filament protection, voltage control and reg. meteorological temp. measurement.</td>
</tr>
<tr>
<td>Washer</td>
<td>10-1,100</td>
<td>10</td>
<td>100-850</td>
<td>150</td>
<td>-3.8</td>
<td>4-24</td>
<td>1/2-4 diam</td>
<td>higher-power temp. comp., surge suppression.</td>
</tr>
<tr>
<td>Wafer</td>
<td>10-1 MΩ</td>
<td>0.5</td>
<td>2.5-7.8</td>
<td>150</td>
<td>-3.9</td>
<td>7-35</td>
<td>1/16 to 1/2 square</td>
<td>temperature measurement and control, high-temperature alarm.</td>
</tr>
</tbody>
</table>

(a) M = 10^6; (b) Container is glass probe or bulb; (c) Special units go to 1,200°C; (d) K = 10^3; (e) 125°C with soldered leads.

### TABLE II  POSITIVE-TEMPERATURE-COEFFICIENT THERMISTORS

<table>
<thead>
<tr>
<th></th>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ohms</td>
<td>watts</td>
<td>°C.</td>
<td>°/°C.</td>
<td>sec.</td>
<td>inches</td>
<td></td>
</tr>
<tr>
<td>Rod</td>
<td>100-1,000</td>
<td>1/4</td>
<td>100</td>
<td>+0.7</td>
<td>35-54</td>
<td>0.4-0.6 long</td>
<td>Transistor temperature comp. temperature measurement.</td>
</tr>
<tr>
<td>Sealed metal case</td>
<td>100-1,000</td>
<td>1/8</td>
<td>125</td>
<td>+0.7</td>
<td>54</td>
<td>0.350 diam</td>
<td>Transistor temperature comp. in high-humidity ambients.</td>
</tr>
<tr>
<td>Glass probe</td>
<td>100-1,000</td>
<td>200</td>
<td>+0.7</td>
<td>9</td>
<td>0.078 diam</td>
<td>0.5 long</td>
<td>Temperature measurement and control.</td>
</tr>
</tbody>
</table>

(a) Hermetically sealed.
146. Vepeck, J.
MAIN PRINCIPLES OF THE DESIGN OF THERMISTOR THERMOMETERS.
(Physica Abstracts 64, No. 4503 (1961)).

The principal characteristics of bead-type thermistors are discussed; in particular, the dependence of their resistance on temperature and current is analyzed. It is pointed out that a bead thermistor can be used as a temperature transducer and, by connecting it in a resistance-bridge circuit, a thermistor thermometer can be designed. The operating conditions of the thermistor in the bridge are considered, and design formulae for the following cases are derived: (1) multipurpose thermometer with a direct-reading meter (the diagonal resistance is much lower than the resistances of the bridge arms), (2) single-purpose thermometer with direct indication, (3) multipurpose thermometer with an indirect-indicating instrument (the diagonal resistance is much higher than the resistance of the bridge arms), and (4) single-purpose thermometer with indirect indication. It is shown that the thermometers are stable and accurate, if the thermistor operating current is suitably chosen. Two practical thermometers for temperatures of 35-42°C and 30-60°C are described.

147. Verwey, Evert J.W., Pieter W. Haayman, and Arie Bol (to Hartford National Bank and Trust Co.).
ELECTRIC RESISTANCE ELEMENT.
(C.A. 42, 8920a).

A resistance element with a negative coefficient of resistance is prepared by sintering FeO, Fe₂O₃, MgO and CrO₃ in the molecular ratio of 1:1:2:2, respectively, to form a homogeneous mixed crystal stable at temperatures above 500°C. At temperatures below 500°C, separation into 2 phases takes place, but the separation is very slow. The FeO-Fe₂O₃-MgO-CrO₃ bodies may be sintered at 1400°C, while the corresponding FeO-Fe₂O₃-MgO-Al₂O₃ bodies must be sintered at 1500-1600°C to obtain a homogeneous and dense product.

ELECTRIC RESISTOR.
(C.A. 44, 3691b).

A resistor with negative temperature coefficient is prepared by sintering at 1400-1500°C in an atmosphere of N₂ a mixture of MgO, FeO, Al₂O₃ and Fe₂O₃ to proportions adjusted to form a spinel phase with 5 to 10 molecular % of MgO and FeO dispersed in the phase.
An element is prepared which has high electric resistance, with a low temperature coefficient which is highly resistant to chemical attack, and which can endure high temperature. The element is made of MgO with approximately 3\% TiO$_2$ or a lower oxide. Anatase or Me cellulose is used as a binder. The rod for the elements is dried and sintered at 1700-2000\°C in an atmosphere of H or H and N, with a trace of O.

A new type of semiconductor is described which contains a fraction of ions of deviating valency without the simultaneous formation of lattice defects. Such a situation is promoted by incorporation into the lattice of foreign ions of such a charge that they balance the charge of the ions of deviating valency. X-ray diffraction data and electrical conductivity of Li$_2$O binary systems with CoO and NiO; of Fe$_2$O$_3$ with TiO$_2$, alkaline-earth systems with TiO$_2$ and La$_2$O$_3$ are given. La$_2$O$_3$ greatly reduces the resistance of SrTiO$_3$, CaTiO$_3$, and BaTiO$_3$. The following additional systems were studied: Li$_2$S-MnS, La$_2$O$_3$-CaMnO$_3$, SrTiO$_3$-SrZrO$_2$-La$_2$O$_3$, CaO-LaMnO$_3$, SrO-LaMnO$_3$, SrO-LaFeO$_3$, TiO$_2$ with Ta$_2$O$_5$, ZnFe$_2$O$_4$, MgFe$_2$O$_4$, NiFe$_2$O$_4$, CoFe$_2$O$_4$, Fe$_2$O$_3$, and Cr$_2$O$_3$-Fe$_2$O$_3$, SnO$_2$-Fe$_2$O$_3$, WO$_3$-Fe$_2$O$_3$, Sb$_2$O$_3$-SnO$_2$, MgWO$_4$-Cr$_2$O$_3$, NiO-MgO-Li$_2$O, and Cr$_2$O$_3$-Fe$_2$O$_3$-TiO$_2$. Applications include ceramic resistors with negative temperature coefficient.

Electrical resistance elements consist of a sintered mass of mixed crystals of FeO, Fe$_2$O$_3$, MgO, and Al$_2$O$_3$ in which the total amount of Fe oxides is 50 molecular \% or less, and the bivalent and trivalent oxides are present in equimolecular amounts. The latter is achieved by the proper choice of sintering temperature and atmospheric composition.

**ELECTRICAL RESISTORS.**
U.S. 2,616,859, Nov. 4, 1952.
(C.A. 49, 7923b).

FeO$_2$ 0.375, NiO 1.5, and TiO$_2$ 0.75 gram-molecules are ball-milled 16 hours with EtOH, worked with a solution of 20% Me methacrylate in Me glycol acetate to form a kneadable mass, extruded as 4-mm. rods, sintered 1 hour at 1300° in pure N (containing 0.1% O), and cooled rapidly in an N stream to give rods having a specific resistance of 2200 ohms/cm. at 22°, 900 at 138°, 200 at 227°, and 50 at 332°. Fe$_2$O$_3$ 4.5, ZnO 10.2, and TiO$_2$ 5.1 gram-molecules are ground with Et acetate for 16 hours, extruded with 10% nitrocellulose in ethylene glycol to form tubes 6.3 mm. long, with an external diameter of 8 mm. and an internal diameter of 4.5 mm., and heated at 1320° as above. The sintering temperatures of combinations of Fe$_2$O$_3$ with spinel compounds, such as Mg$_2$TiO$_4$, Zn$_2$TiO$_4$, Ni$_2$TiO$_4$, Zn$_2$SnO$_4$, or their mixed crystals, may thus be reduced to 1300°.

153. Voloshin, I.F.

**THEORY OF SEMICONDUCTING THERMAL RESISTANCES.**
Trudy Inst. energ. ANSSR No. 6, 193-205 (1958).
(Monthly Index of Russian Accessions 13, 680 (1960)).


**SEMICONDUCTOR THERMISTORS.**
Inzh.-fiz. zhur. No. 1, 124-6 (1960).
(Monthly Index of Russian Accessions 13, 1465 (1960)).

A review, by B.S. Sotskov, of book of this title.

155. Voltz, Sterling E. (to Houdry Process Corp.).

**TREATING CHROMIC OXIDE.**
(C.A. 49, 12955a).

A semiconducting resistor with negative temperature coefficient is made by the following process: Cr$_2$O$_3$ is precipitated from Cr(NO$_3$)$_2$ solutions with NH$_4$OH, washed, and dried at 110° for 8 hours. The precipitate is heated in N at 350° and then in O (5 liters/hour flow) at 350-500° for 2 hours. A further optional treatment in H at 350°, which increases the resistance and decreases the temperature coefficient, may be used to adjust the resistance.

156. Walter, Paul

**NOVEL OXIDE LAYER RESISTOR WITH NEGATIVE TEMPERATURE COEFFICIENT.**
Elektrotech. Z. A78, 500-4 (1957).
(C.A. 22, 12355b).

Solutions of hydrolyzable Cd, In, or Sn salts decompose upon contact with a hot ceramic carrier body and form semiconducting coatings (Ger. 208,882; U.S. 2,564,706 (C.A. 46, 228b)). The resistor bodies consist of a ceramic tube, the conductive coating, and the end leads (or sleeves), the latter tightened upon a silver paint layer. The resistive coating is a mixed oxide of Sn$^4+$ and Sb$^3+$ (Glang, Thesis,
This layer has excellent adhesion and the hardness of quartz. It is also protected through an insulating lacquer. The carrier must be above 400° to coat it through spraying, dipping, or vaporizing. Pure SnO$_2$ layers have about 200 ohms/sq. cm. resistivity in an arbitrary standard, which decreases to about 12 ohm/sq. cm. with 1.3 weight % Sb$_2$O$_3$ to increase nearly linearly with more Sb$_2$O$_3$ (about 300 for 14% Sb$_2$O$_3$). Temperature coefficient can be controlled between +0.1 and -1.0%/degree.

157. Weichman, F.P.
OPTICAL AND ELECTRICAL PROPERTIES OF CuO.
PB-140,973 (1958).
(OTS Selected Bibliographies - Semiconductors).

158. Weil, L., J. Peretti, and A. Lacaze.
A ZINC OXIDE RESISTANCE THERMOMETER.
(Physics Abstracts 57, No. 2377 (1954)).

A layer of Zn is applied to a ceramic base by a vacuum technique and connected to the oxide by heating at 400-450° C. in air or oxygen. Stability is said to be better than that of colloidal graphite and the accuracy 0.01 against 0.1° for graphite. In the region of 2° K. the resistance varies by about 10% per degree.

ABOUT THE EQUIVALENT CIRCUIT OF THERMISTORS.
(Astia Bibliography).

An equivalent circuit for thermistors was developed and the elements were determined for a commercial bead type, both for alternating current and for direct current transients. The circuit contains an inductance for which values of several thousand henries were found. The dependence of the circuit elements upon the position of the operating point on the characteristic, upon frequency, and upon outside parameters, as voltage and series resistance, was determined. For a particular bead type thermistor critical frequencies of about 1 cps at an operating point with a current of 1 ma and of about 3.5 cps at 2 ma respectively were found. At these frequencies the thermistor displayed the behavior of a pure inductance in producing a phase shift of exactly 90° between voltage and current. Other investigators used the same equivalent circuit previously. Their considerations and data were compared with the findings of this work which shows progress over the earlier investigations especially in demonstrating the limits of the applicability of the equivalent circuit assumed.
GRAPHICAL EXTRAPOLATION OF VOLTAGE CURRENT CHARACTERISTICS OF THERMISTORS.
(Astia Bibliography).

It was found that at each point of the voltage current characteristic of a thermistor the rise of temperature above the environmental temperature is directly proportional to the wattage. Using this relation, a simple graphical extrapolation method was derived. By it, any number of characteristics at arbitrary environmental temperatures can be found if two characteristics and the respective environmental temperatures have been measured. The limits of the method are discussed.

161. Weise, Erwin K.
PRINCIPLES OF THERMISTOR APPLICATION.
(Astia Bibliography).

Log R vs. 1/T curves are plotted for CaTiO$_3$ treated in a special way of gradual removal of a certain amount of O from the lattice by holding the samples for about 30 minutes in H at various temperatures from 1000-1500° C. The properties can also be varied by using different raw materials; e.g., by mixing several oxides and sintering at elevated temperature. Non-linear curves can arise from using mixture of different raw materials not forming compounds.

162. Weise, E.K.
ALIGNMENT CHART FOR RESISTANCE-TEMPERATURE CHARACTERISTICS OF THERMISTORS.
(Astia Bibliography).

The relation of resistance (R) ohm and temperature T (°C) is a straight line for many commercial thermistors if plotted as log R vs. 1/T or log R = $K_1 + K_2T$ and if $K_1$, log A and $K_2 = B$, this becomes log R = log A + B/T, $R = A e^{B/T}$. A plot of log resistance vs. reciprocal absolute temperature gives Β (slope) and A (vertical axis intercept). The temperature coefficient of resistance α (% per degree) = $\frac{A}{R} X \frac{dR}{dT} X 100$ or $\alpha = \frac{\Delta R}{R} X 100$. A plot giving this at various temperatures is given.

THERMISTORS AS TOOLS IN RESEARCH AND DEVELOPMENT.
PB-100,655 (mi. $\$2.25, Ph $5.00).
(Bibliography of Technical Reports 14, 73 (1950)).

This report introduces the reader to the physical properties, theoretical considerations, applications and current international production possibilities of thermistor type of semiconductors...
164. Weise, Erwin.
TECHNISCHE HALBLEITER-WIDERSTÄNDE (Technical application of semi-
conductor resistances).
PB-52,381 (mi. $2.50).
(Bibliography of Scientific and Industrial Reports 5, 199 (1947)).

This book discusses "semi-conductor resistances." The first chapter
elaborates on various types of such resistors that are already in
practical use, to wit: (1) The "Urdox resistor" made by Osram A.G.
of Berlin; (2) the "Hot conductor" made by Siemens & Halske A.G. of
Berlin-Siemensstadt; (3) the "Starto tube" made by N.V. Philips of
Eindhoven, Holland; and (4) the "Thermistor" made by the Western
Electric Co. of New York. The second chapter deals with the physical
properties of semiconducting resistances, whereas the third chapter
discusses in detail their practical application. The following uses
are listed and described by the author: (1) Resistance thermometers,
(2) damping and retardation resistances, (3) cathode- and capacitor-
protecting resistances, (4) starting resistances, (5) retardation
relays, (6) regulation of electric machines, (7) regulation of arc
lamps, (8) bridge resistors, etc. Drawings, graphs, and a biblio-
graphy are included. In German.

165. Weise, E.K. (University of Illinois, Urbana).
BASIC AND APPLIED RESEARCH ON SEMICONDUCTORS.
PB-134,716 (mi. $4.80, Ph $13.80, order from Library of Congress).
(U.S. Government Research Reports 30, 351 (1958)).

...Sintered samples of Mg$_2$TiO$_4$, MgTiO$_3$, MgTi$_2$O$_5$, CaTiO$_3$, SrTiO$_3$, and
BaTiO$_3$ were measured at temperatures between -190° C. and +1500° C.
in inert atmospheres. The electrical conductivity was measured between
-900° and +1500°. Between +20° C. and -190° C. the Hall effect was
measured...Investigations of the resistance-temperature characteristics,
the voltage-current characteristics, and the equivalent circuit of
thermistors are discussed.

166. Weise, E.K.
FINAL REPORT ON BASIC AND APPLIED RESEARCH ON SEMICONDUCTORS.
(Semiconductor Abstracts VI, No. 587 (1958)).

Electrical conductivity from -190° to +1500° was measured for Mg$_2$TiO$_4$,
MgTiO$_3$, MgTi$_2$O$_5$, CaTiO$_3$, SrTiO$_3$, and BaTiO$_3$ in inert atmosphere; Hall
effect was determined from -190° to +20°. N-type donors were intro-
duced by partial H$_2$ reduction. Magnetic susceptibilities, conduc-
tivities, Hall data, and electron mobilities were determined for the
berthollides. Thermistor properties are discussed.
167. Wejnarth, Axel R.
COMPOSITION OF SILICON CARBIDE ELECTRICAL-RESISTANCE UNITS.
(C.A. 41, 1059a).

Addition agents incorporated into Si carbide mixes before sintering may be one of the group Cr carbide, Cr nitride, Cr silicide, and Cr boride, together with one of the following metals, combined with any of the above anions: Mo, W, Ti, and V. The resulting electrical-resistance units have improved resistance at high temperatures. The composition also affects the resistance-temperature characteristics.

168. Western Electric Co., Inc.
ELECTRIC CONTROL SYSTEM.
Brit. 465,923, May 19, 1937.
(C.A. 32, 77732).

AggS resistance units are used to control the gain of an amplifier or compensate for variations in the resistance of transmission lines. The units have high negative temperature coefficients.

169. Westinghouse Electric Corp.
THERMISTOR (CONSISTING OF CERAMIC BODIES HAVING A HIGH POSITIVE TEMPERATURE COEFFICIENT OVER A RANGE OF TEMPERATURES).

Thermistor consisting of a ceramic body and electrodes applied thereto is described, the body comprising essentially the fired product of (a) a total of 1 mole of at least 1 oxide selected from the group TiO₂, ZrO₂, Nb₂O₅ and Ta₂O₅, and (b) a combined total of (1) a total of up to 0.999 mole of at least 1 oxide from the group BaO, PbO, and SiO₂, and (2) a total of from 0.0001 mole to the limit of solubility of a trivalent substitutional impurity of at least 1 rare earth metal fitting the crystal lattice, the body being initially fired in an oxidizing atmosphere at a temperature of at least 1000° C. for a period of hours. A typical fired product comprises YₓBa(1-x)TiO₃ (where x has a value of 0.005-0.02).

170. Westinghouse Electric Corp.
CERAMIC MEMBER HAVING A MARKED POSITIVE TEMPERATURE COEFFICIENT OF ELECTRICAL RESISTANCE OVER A SELECTED RANGE OF TEMPERATURES.

Ceramic member comprising the fired product having the general formula: MₓBa(1-x)ZrₓTi(1-y)O₃ is described, where M represents at least 1 rare earth metal from the group consisting of Y and Ce, x has a value of 0.003-0.03 and y, a value of 0.01-0.25, the member being initially fired in an inert atmosphere to sinter it and thereafter fired in an oxidizing atmosphere. Compositions of the formula Y₀.₀₁₅Ba₀.₉₈₅ Zr₀.₂₀Ti₀.₈₀O₃ were found to have a resistance of 1000 ohms at 45° C., the positive temperature coefficient being so large that in the succeeding 25°, the resistance rose to over 10,000 ohms.
171. Wisely, Harriet R.
AN EXHAUST GAS TEMPERATURE THERMISTOR BODY FOR THE RANGE 1500°F. TO 3500°F.
(Astia Bibliography).

Materials tested having melting points in excess of 3500°F. were
Al₂O₃, spinel (MgO.Al₂O₃) with and without addition of stabilized
ZrO₂, CaZrO₃, MgO with addition of Ce₂O₃, BeO with Al₂O₃ and ZrO₂ or
ThO₂ additions. Among the compositions tested, the 99% Al₂O₃ body
most nearly followed the desired resistivity vs. temperature values.

RESISTIVITY CHARACTERISTICS OF SOME CERAMIC COMPOSITIONS.
(C.A. 51, 13338d).

The electrical resistivity of a number of ceramic compositions were
determined, 1000-2000°F., in an effort to obtain a thermistor material
suitable for temperature measurements in the exhaust gases of jet
engines. Promising materials were subjected to tests of stability
on prolonged heating in air at 1800°F. and under alternately oxidizing
and reducing conditions at temperatures up to 2000°F. Compositions
in the system K₂O-Al₂O₃-SiO₂ (K₂O 5-15, Al₂O₃ 40-75, SiO₂ 20-48 weight
%) showed linear relations between log ρ and 1/T and resistivities
varying from 10⁵ to 10⁻⁶ ohm-cm. at 1000°F. to 10³ to 10⁴ ohm-cm. at
2000°F. Compositions in the system Li₂O-Al₂O₃-SiO₂ showed lower
resistivities but higher temperature coefficients.

PROPERTIES OF GERMANIUM DOPED WITH MANGANESE.
Physical Reviews 100, 659 (1956).

The temperature dependence of the electrical resistivity and Hall
coefficient in p- and n-type manganese doped Ge crystals indicates
that Mn introduces 2 acceptor levels in Ge at 0.16 ±0.01 e.v. from
the valence band and 0.37 ±0.02 e.v. from the conduction band...
Comparison is made with other fourth row metals (Fe, V, Co, and Ni)
as impurities in Ge.

TEMPERATURE DEPENDENCE OF THE RESISTIVITY, HALL COEFFICIENT, AND
THERMAL POWER IN Ti₂O₃.
(Solid State Abstracts 1, No. 7309 (1961)).

* Photocopy available.
Information on the following thermistors, supplied by the manufacturers at our request, is available:

(1) Atkins Technical Inc., Cleveland, Ohio.

"Thermophil" thermistors offering stability, interchangeability, accuracy of 0.2% to 0.7% of total overlapping scale range, a temperature range of -148° to +845° F. or -100 to 450° C. on overlapping scales are based on "the change in electrical resistance of a special Germanium alloy (thermistor)." All probes are interchangeable between instruments of the same type. The heat sensing thermistor has a resistance of 50,000 ohms at room temperature.


Globar thermistors B, F and H are non-linear (negative temperature coefficient) resistors which offer a range of temperature coefficients from 0.3% per degree C. to 5.1% per degree C. at 25° C.

Type B have a resistance at 25° of 10-100,000 ohms, a temperature coefficient β of 225-470% and % change per degree C. of .25-53; type F, resistance 10-100,000, β = 1200-2150, % per degree C. = 1.35-2.40; type H, resistance 5150-500,000, β = 3000-4600, % per degree C. = 3.40-5.20. The thermal time constant is 15 seconds to 3-5 minutes; stability is good.

Globar also makes a positive temperature coefficient thermistor, sensitive between -30 and 100°C. for which temperature vs. percent resistance at 37.8°C curve is given.

(3) Fenwal Electronics, Framingham, Massachusetts.

"Thermistors are semiconductors made by sintering mixtures of metallic oxides such as Mn, Ni, Co, Cu, Fe and U. Various mixtures of these metallic oxides are formed into useful shapes. Their electrical characteristics may be controlled by varying the type of oxide used and the physical size and configuration of the thermistor."

β value for Fenwal Electronics Thermistors is approximately 4000, α, the temperature coefficient of resistance, is as high as -5.8% at room temperature, resistance values of 300 ohms to 100 megohms for beads, 5 to 10,000 ohms for discs, and 1000-150,000 ohms for rods can be obtained.

(4) Ferroxcube Corp. of America, Saugerties, New York.

"NTC" resistors have a high negative temperature coefficient of resistance (-3 to -6 1/2 % per °C.). The resistors consist of a disc of semiconductive ceramic material, both faces of which are covered with a layer of silver serving as an electrode. They are available in five different nominal values of resistance at 25° ranging between 4 and 1300 ohms. Value of β varies from 3300-5700.

Ferroxcube also makes "VDR" or voltage-dependent resistors.
Magnetic Materials Section, General Electric Co., Erdmore, Michigan.

General Electric thermistors are made in two material grades and three main types: rods, discs and washers. The raw materials—Mn, Ni, and Co oxides—are milled and mixed in accurate proportions. Suitable binders are added and the units are pressed or extruded to the desired shapes. Thermistor elements are then sintered under carefully controlled atmospheric and temperature conditions to produce a hard ceramic-like material. The properties are as follows:

<table>
<thead>
<tr>
<th>Material</th>
<th>Sp. Resistance at 25° C.</th>
<th>Thermal Resistivity at 25°-75° C.</th>
<th>Thermal Conductivity</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>2100 ±10%</td>
<td>3965 ± 55</td>
<td>29.4</td>
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<tr>
<td>2</td>
<td>300 ±10%</td>
<td>3550 ± 80</td>
<td>20.2</td>
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</table>

Temperature coefficient of resistance at 25° C. = -4 to -4.5% per °C.


"Glennite Thermistors" are ceramic temperature-sensitive resistors. Exhibiting high negative coefficient of resistance, these semiconductors possess resistance values which may vary by a ratio of 10,000,000 to 1 from -100°C. to +450°C. They are available in a resistance range from ohms to megohms and their thermo-resistive characteristics, coupled with excellent stability and high sensitivity in a small, rugged unit, present endless possibilities in circuit design. The temperature coefficient of resistance given varies from -3.9 to -6.8% for wafers, -3.9 to -5.4% for beads, β = 3400-4115.


NTC Resistance Units are formed of metallic compounds, heat treated, contacted and aged for maximum stability and possess the unique characteristic of a large negative change in resistance with respect to temperature. The temperature coefficient is -1.2 to -5% per degree C. They operate at -80 to 150° C.

Kidde Thermistors.

Type K — β = 3100°K., time constant = 18 seconds, resistance at 25° = 1-100 K; Type D — β = 3100°K., time constant 26 seconds, maximum operating temperature 150°C., resistance at 25°C. 100 ohms to 100 K ohms.

Sperry Microwave Electronics Co., Clearwater, Florida.

Miraline thermistors are actually small high resistance ceramic beads supported between two parallel wires. For greater rigidity the bead is generally enclosed in glass. The temperature range is -35 to 800°. These thermistors are used for c-w or average power measurement.
Victory Engineering Corp., Union, New Jersey.

Veco thermistors are manufactured in three distinct forms: beads, discs or washers, and rods. All of these types are made of various mixtures of the oxides of Mn, Ni, Co, Cu, U, Fe, Zn, Ti, and Mg. These materials are mixed in proper proportion to provide the required specific resistance and temperature coefficient of resistance for particular applications. These mixtures of oxides are formed into the desired shapes and sintered under accurately controlled atmospheric and temperature conditions. The finished product is a hard ceramic material which may be mounted in a variety of ways depending upon the mechanical, electrical, and thermal requirements.

All Veco thermistors are produced by carefully controlled processes which give them the following characteristics:

1. Extremely high stability.
2. Mechanical ruggedness and excellent shock resistance.
3. Permanent electrical contacts.
4. Wide range of resistance, temperature coefficient, and power dissipation.
5. Unlimited life when operated within maximum temperature ratings.

Thermistor material No. 1 is composed of oxides of Mn and Ni and has a temperature coefficient of resistance of -4.4% per °C. at 25°C. and is recommended when comparatively high resistance and an extremely high temperature coefficient are desired.

Thermistor material No. 2 is composed of oxides of Mn, Ni, and Co and has a temperature coefficient of resistance of -3.9% per °C. at 25°C. and is used in applications where comparatively low resistance and a moderate temperature coefficient are desired.

There are many other special Veco thermistor mixes now being used for thermistors requiring exceptionally high or low resistance or where special temperature coefficients of resistance are required.

Specific resistances ranging from 100 to 450,000 ohm-cm. at 25°C. have been produced which gives engineers a wide latitude in the design of equipment utilizing Veco thermistors. For example, Veco thermistors are now being produced with a temperature coefficient of resistance as high as -5.8% per °C. at 25°C., while that of platinum is only +0.36° per °C.

Other companies contacted were:

(11) Bart Labs, Newark, New Jersey — no reply.

(12) Bendix Corp., Freig Instrument Division, Baltimore, Maryland — who no longer manufacture thermistors.


(14) Electro-Impulse Laboratory, Inc., Red Branch, New Jersey — who do not make thermistors.


Thermistor Applications

A large number of abstracts on specific applications have been collected and are available on request. However, it is felt that the following tabulation, abstracted from the Veco "Data Book" indicates adequately the variety of these applications.

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<th>APPLICATIONS FOR VECO THERMISTORS IN VARIOUS INDUSTRIES.</th>
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<td>Local and Remote Temperature Measurement and Control</td>
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Transportation
Engine Temperature Measurement and Control
Flow Meters
Switching and Signalling Devices
Differential Temperature Controllers
Fire Protection and Safety Devices
Test Equipment
Aircraft Cabin Temperature Control
Meteorological Equipment
Wing Temperature Measurement for Aircraft
Aircraft Anti-icing Control

Public Utilities
Voltage Regulation
Time Delay Devices
Warning Devices
Gas Detectors
Flow Meters
Anemometry
Chemical Analysis and Control
Power Indicators
Temperature Compensation of Instruments
Switching Devices
Calorimetry
Pyrometry

Housing and Household Appliances
Automatic Switches
Fire Protection Devices
Automatic Room Temperature Control
Air Conditioning Systems Control
Gas Detectors
Furnace Controls
Oven Temperature Control
Refrigeration Control

Medical and Scientific
Temperature Measurement of Blood in the Human Body
Subcutaneous Skin and Muscle
Temperature Measurement

Instrumentation
Power Indicators
Pyrometry
Meteorological Equipment
Vacuum Gauges
Temperature Compensation

In addition to the uses mentioned here, attention should also be called to the interest in thermistors as bolometers, indicated by a large number of references in various government reports.
**DISTRIBUTION LIST**

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ADDRESSEE

Servo Corporation of America
Attn: Mr. Frank Kocsis
111 New South Road
Hicksville, L. I., New York

Sperry Rand Corp.
Sperry Semiconductor Div.
Wilson Avenue
S. Norwalk, Connecticut

Sylvania Electric Products, Inc.
333 W. First Street
Dayton 2, Ohio

Texas Instruments, Inc.
3300 S. Dixie
Dayton 39, Ohio

Textron Electronics, Inc.
G. C. Electronics Co.
Attn: A. C. Valiulis, Chief Engineer
400 S. Wyman Street
Rockford, Illinois

Trionics
Attn: Dr. G. D. Hedden, Technical Director
P. O. Box 548
Madison 1, Wisconsin

Tung-Sol Electric, Inc.
1 Summer Avenue
Newark 4, New Jersey

Tyco Semiconductor Corp.
Attn: G. Freedman, President
Bear Hill
Waltham 54, Massachusetts

Victory Engineering Corp
118 Springfield Avenue
Springfield, New Jersey

Westinghouse Electric Corp.
32 N. Main Street
Dayton 2, Ohio

Yellow Springs Instrument Co., Inc.
Attn: R. A. Gordman, Chief Engineer
P. O. Box 106
Yellow Springs, Ohio
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Gold-doped monocrystalline silicon exhibits temperature-resistivity behavior suitable for making highly reproducible, predictable and sensitive thermistors. Two types of thermistors were developed with reproducibilities of ±2% & operable ranges incl. -85 to +200°C. Manufacturing methods were developed and demonstrated on an unbalanced pilot line.

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Gold-doped monocrystalline silicon exhibits temperature-resistivity behavior suitable for making highly reproducible, predictable and sensitive thermistors. Two types of thermistors were developed with reproducibilities of ±2% & operable ranges incl. -85 to +200°C. Manufacturing methods were developed and demonstrated on an unbalanced pilot line.

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