NBS TECHNICAL NOTE 598

Methods of Measurement for Semiconductor Materials, Process Control, and Devices

Quarterly Report

January 1 to March 31, 1971

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Methods of Measurement for Semiconductor Materials, Process Control, and Devices

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January 1 to March 31, 1971

Edited by W. Murray Bullis

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Institute for Applied Technology
National Bureau of Standards
Washington, D.C. 20234

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FOREWORD

The Joint Program on Methods of Measurement for Semiconductor Materials, Process Control, and Devices was undertaken in 1968 to focus NBS efforts to enhance the performance, interchangeability, and reliability of discrete semiconductor devices and integrated circuits through improvements in methods of measurement for use in specifying materials and devices and in control of device fabrication processes. These improvements are intended to lead to a set of measurement methods which have been carefully evaluated for technical adequacy, which are acceptable to both users and suppliers, which can provide a common basis for the purchase specifications of government agencies, and which will lead to greater economy in government procurement. In addition, such methods will provide a basis for controlled improvements in essential device characteristics, such as uniformity of response to radiation effects.

The Program is supported by the National Bureau of Standards,[*] the Defense Atomic Support Agency,[†] the U. S. Navy Strategic Systems Project Office,[§] the U. S. Navy Electronics Systems Command,[+] the Air Force Weapons Laboratory,[¶] the Atomic Energy Commission,[#] and the National Aeronautics and Space Administration.[×] There is not a one-to-one correspondence between the tasks described in this report and the projects by which the Program is supported. Although all sponsors subscribe to the need for the entire basic program for improvement of measurement methods for semiconductor materials, process control, and devices, the concern of certain sponsors with specific parts of the Program is reflected in planning and conduct of the work.

* Through Research and Technical Services Projects 4251120, 4251123, 4251126, 4252114, 4252119, 4252128, 4254111, 4254112, and 4254115.

† Through Order EA071-801. (NBS Project 4259522).

§ Administered by U. S. Naval Ammunition Depot, Crane, Indiana through Project Orders PO-1-0030, PO-1-0041, and PO-1-0067 and Naval Avionics Facility through Work Request WR-1-1038. (NBS Projects 4259533 and 4254432)

+ Through Project Order PO-1-1057. (NBS Project 4252534)

¶ Through Delivery Order F29601-71-F-0002. (NBS Project 4252535)

Division of Biology and Medicine. (NBS Project 4259425)

× Through Orders S-70003-G, Goddard Space Flight Center, and H-76553A, Marshall Space Flight Center. (NBS Projects 4254429 and 4251449)

METHODS OF MEASUREMENT FOR SEMICONDUCTOR MATERIALS, PROCESS CONTROL, AND DEVICES

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ABSTRACT

This quarterly progress report, eleventh of a series, describes NBS activities directed toward the development of methods of measurement for semiconductor materials, process control, and devices. Significant accomplishments during this reporting period include application of tuning and other related procedures for ultrasonic wire bonders in an industrial environment with a reported reduction of rejection rate at visual inspection by more than a factor of two, more complete evaluation of the photovoltaic method for determining radial resistivity profiles of circular semiconductor wafers, and identification of test conditions in which a d-c calibration curve may properly be used in measurements of thermal resistance. Work is continuing on measurement of resistivity, carrier lifetime, and electrical inhomogeneities in semiconductor crystals; specification of germanium for gamma-ray detectors; evaluation of wire bonds and die attachment; measurement of thermal properties of semiconductor devices, transit-time and related carrier transport properties in junction devices, and electrical properties of microwave devices; and characterization of silicon nuclear radiation detectors. Supplementary data concerning staff, standards committee activities, technical services, and publications are included as appendixes.

Key Words: Alpha-particle detectors; aluminum wire; base transit time; carrier lifetime; die attachment; electrical properties; epitaxial silicon; gamma-ray detectors; germanium; gold-doped silicon; methods of measurement; microelectronics; microwave devices; nuclear radiation detectors; probe techniques (a-c); resistivity; semiconductor devices; semiconductor materials, semiconductor process control; silicon; thermal resistance; thermographic measurements; ultrasonic bonder; wire bonds.

1. INTRODUCTION

This is the eleventh quarterly report to the sponsors of the Joint Program on Methods of Measurement for Semiconductor Materials, Process Control, and Devices. It summarizes work on a wide variety of measurement methods that are being studied at the National Bureau of Standards.

Since the Program is a continuing one, the results and conclusions reported here are subject to modification and refinement.

The work of the Program is divided into a number of tasks, each directed toward a particular material or device property or measurement technique. This report is subdivided according to these tasks. Highlights of activity during the quarter are given in Section 2. Section 3 deals with tasks on methods of measurement for materials; Section 4, with those on methods of measurement for process control; and Section 5, with those on methods of measurement for devices. References for each section are listed in a separate subsection at the end of that section.

The report of each task includes the long-term objective, a narrative description of progress made during this reporting period, and a listing of plans for the immediate future. Additional information concerning the material reported may be obtained directly from individual staff members connected with the task as indicated throughout the report. The organization of the Joint Program staff and telephone numbers are listed in Appendix A.

An important part of the work that frequently goes beyond the task structure is participation in the activities of various technical standardizing committees. The list of personnel involved with this work given in Appendix B suggests the extent of this participation. Additional details of current efforts in this area are given in Section 2.

Background material on the Program and individual tasks may be found in earlier reports in this series as listed in Appendix D. From time to time, publications that describe some aspect of the program in greater detail are prepared. Current publications are also listed in Appendix D.

2. HIGHLIGHTS

Significant accomplishments during this reporting period include successful application of tuning and other related procedures for ultrasonic wire bonding machines in an industrial environment with a reported reduction in rejection rate at visual inspection by more than a factor of two, more complete evaluation of the photovoltaic method for determining radial resistivity profiles of circular semiconductor wafers, and identification of test conditions in which a d-c calibration curve may properly be used in measurements of thermal resistance. Highlights of these and other technical activities are presented in this section; details are given in subsequent sections of the report. This section concludes with a summary of standardization activities being carried out by program staff members.

Resistivity — Principal emphasis was placed on continuation of the study of current and probe-force dependence of resistivity as measured by the four-probe method. Measurements completed on mechanically polished wafers with revised experimental procedures are being evaluated. The wafers were chem-mechanically polished, and measurements on the chem-mechanically polished surfaces were begun. Coordination of two round robins on the four-probe method being conducted in cooperation with ASTM Committee F-1 on Electronics continued. Measurements were made in connection with a round robin on the metal-oxide-semiconductor capacitance-voltage method also conducted by Committee F-1. Experimental work on the diode capacitance-voltage method and further consideration of silicon wafer resistivity standards were deferred. Effort on spreading resistance methods was devoted to further study of procedures for preparing silicon wafers for examination by the scanning electron microscope and initial study of probe impact with a force transducer.

Carrier Lifetime — The level of effort in this area was decreased significantly during the quarter. Additional experiments were conducted in an effort to define appropriate conditions for the measurement of carrier lifetime in bulk crystals by the photoconductive decay (PCD) method. Measurements were made on bulk crystals to compare results obtained by the PCD and surface photovoltage (SPV) methods. The draft of the paper describing the application of the SPV method to measurement of carrier lifetime in epitaxial layers was completed. An experiment on measurement of carrier lifetime by the metal-oxide-semiconductor capacitance method was completed.

Inhomogeneities — Various aspects of the application of the photovoltaic method as a means for measuring radial resistivity profiles in circular semiconductor wafers without contacting the flat surfaces of the wafers were evaluated. Some problems in comparing profiles made by photovoltaic and four-probe methods were traced to the effects of uncertainty in the probe location in a four-probe measurement. It was shown that abrading the wafer rim improved the quality of the rim contacts and permitted measurements to be made closer to the edge of the wafer. An experiment conducted to obtain an indication of the reproducibility of the photovoltaic method yielded a maximum difference between four independent readings of less than 2 percent.

Gold-Doped Silicon — Work continued in preparing and evaluating gold-doped silicon wafers in the extensive series outlined previously. A preliminary study to determine if the retrograde behavior of the resistivity of heavily gold-doped, *p*-type silicon could be explained by a variation of the ionization energy of the gold donor level with gold concentration yielded negative results. The study of the lateral uniformity of diffused gold as determined by x-ray fluorescence with the scanning electron microscope continued.

Specification of Germanium — Standards pertaining to the measurement of lithium-ion drift mobility and lithium-drifted germanium detector characterization are being developed in cooperation with ASTM Committee F-1 and IEEE Nuclear Instruments and Detectors Committee respectively. Additional semiconductor diodes, including a heat-treated germanium specimen, a diode fabricated from a crystal of high-purity germanium, and a lithium-drifted silicon diode, have been examined by the improved infrared response technique. Preliminary studies of the infrared response measurement system have been directed toward determination of specimen temperature during the measurement and of the effects of changing the magnitude of applied bias to the diode on the spectral response.

Die Attachment Evaluation — Experiments were completed which demonstrated that the procedures and equipment used to make thermal response measurements result in adequate reproducibility and that the spread in the measured thermal response of diodes without voids is small compared to the change introduced by the presence of voids. Both these conditions had to be met in order to compare the relative sensitivity to the presence of voids in the die attachment material of transient thermal response measurements and steady-state thermal resistance measurements. It was found that transient thermal response measurements were significantly more sensitive to the presence of voids than steady-state thermal resistance measurements.

Wire Bond Evaluation — At the request of a sponsor capacitor microphones and magnetic pickups were distributed to four semiconductor device manufacturers together with instructions for their use in setting up and tuning ultrasonic wire bonding machines. In one case, use of this equipment and associated technology for process control resulted in a reduction in rejection rate due to faulty bonds at visual inspection by more than a factor of two. A study of wire-bond failures due to slow-power-cycling of transistors indicated that use of a large loop height minimizes such failures. Two new ultrasonic wire bonding machines, one for bonding round wire and the other for bonding ribbon wire, were set up. Extensive modifications were necessary to correct faults in the machines as delivered. Detailed procedures for setting up bonding machines were developed. The wire tester was completed; initial tests showed distinct differences in uniformity of three different wire samples. An apparatus to measure bonding force was designed for the purpose of reducing variability between measurements made by different operators. Initial studies of ultrasonic bonding that may eventually lead to an in-process method for determination of bond quality were made with a self-tuning ultrasonic power supply and transducer.

Thermal Properties of Devices — Conditions under which d-c calibration procedures yield correct values in making thermal resistance measurements have been identified. It was found that the thermal resistance can be calculated from differences in temperature and power between two different power levels on the linear portion of the base-emitter-voltage — transistor-power curve. Preliminary measurements of the reproducibility which can be achieved by the thermal resistance measuring equipment were undertaken. A procedure for electrically measuring junction temperature during the current-constricted operating mode was investigated. A comparative study of several different techniques for measuring thermal resistance was begun in order to determine the general applicability of the various techniques for use as industrial standards.

Microwave Device Measurements — The r-f portion of the X-band mixer measurement system was extensively rebuilt to permit active stabilization of the local oscillator power and improve other aspects of the circuit.

Carrier Transport in Junction Devices — Evaluation of the Sandia-type delay-time bridge continued. Preliminary delay-time measurements were made as a function of emitter current on a silicon *n-p-n* transistor with f_α of about 350 MHz. A theoretical analysis of a delay-time error that might be introduced during rebalancing of the bridge was completed. From a survey of noise properties of semiconductor devices that was completed, it was concluded that the use of noise spectrum measurements should enable one to measure separately device phenomena which otherwise are almost always measured by their sum effect.

Silicon Nuclear Radiation Detectors — Pre-flight bench testing of lithium-drifted silicon radiation detectors continued. A test chamber for carrying out measurements of the effects of ambients that may affect the performance characteristics of semiconductor detectors was constructed and evaluated. Radiation damage measurements on lithium-drifted silicon detectors using 1.5 MeV electrons were begun.

Standardization Activities — Many of the standardization activities undertaken by program staff are broader than the technical tasks described in the following sections. These activities involve general staff support in committees, coordination of efforts which may encompass a variety of tasks, and participation in areas where no direct in-house technical effort is under way. Standardization activities directly related to particular task areas are reported with the appropriate tasks.

Six program staff members attended the regular winter meeting of ASTM Committee F-1. W. E. Phillips was appointed secretary of Subcommittee 4 on Semiconductor Crystals in addition to his other assignment as chairman of the Carrier Lifetime Section. W. M. Bullis acted as temporary chairman of the Resistivity and Conductivity Type Sections. He also reported on the completion of a revised portion of the general information guide for members of ASTM Committee F-1. A broadened scope of the committee to include device as well as material measurement methods was approved by the ASTM Board of Directors. J. C. French and W. M. Bullis proposed activities that might be undertaken in response to this broadened scope to several committee officers. Subcommittee 7 on Hybrid Microelectronics is taking the lead in developing new areas of effort. Considerable interest exists both in the evaluation of wire and die bonds and in the characterization of chips destined for use in hybrid microcircuits. Other areas of interest are radiation effects on and thermal properties of semiconductor devices and integrated circuits.

J. C. French and W. M. Bullis attended meetings of the sections of ASTM Committee E-10 that are concerned with radiation effects on electronic components. Reviews of Committee F-1 and Joint Program activities were presented. This committee has a significant interest in definitions and test methods appropriate to the study of response of devices to radiation. Program staff members are providing a communication link between Committee F-1, oriented toward electronics and Committee E-10, oriented toward radiation effects.

Activity in connection with JEDEC committees of the Electronic Industries Association was concentrated in the thermal measurements and second breakdown areas. F. F. Oettinger was recently named to a task group on thermal measurements for the newly organized Committee JC-30 on Hybrid Integrated Circuits and was instrumental in the preparation of JEDEC Engineering Bulletin No. 18, Thermal Parameters of Microelectronic Devices, issued through Committee JC-11 on Mechanical Standardization.

D. E. Sawyer was appointed liaison representative to the National Materials Advisory Board ad hoc Committee on Materials for Radiation Detection Devices. W. M. Bullis continued his work in connection with the NMAB ad hoc Committee on Materials and Processes for Electron Devices. In this connection he organized a symposium of experts on metal-insulator-semiconductor structures to provide input to the committee report.

Activity in various IEEE committees increased. J. C. French, H. A. Schafft, and J. M. Kenney continued work with the Standards Committee of the Group in Electron Devices particularly with respect to thermal properties and microwave diode characteristics. W. M. Bullis was appointed to the New Technology Subcommittee of the Technical Committee on Hybrid Microelectronics of the Group on Parts, Hybrids, and Packaging. This committee is beginning to undertake development of a terms glossary and test methodology appropriate to hybrid microcircuits.

3. METHODS OF MEASUREMENT FOR SEMICONDUCTOR MATERIALS

3.1. RESISTIVITY

Objective: To develop methods suitable for use throughout the electronics industry for measuring resistivity of bulk, epitaxial, and diffused silicon wafers.

Progress: Principal emphasis was placed on continuation of the study of current and probe force dependence of resistivity as measured by the four-probe method. Measurements, made with revised experimental procedures, were completed on mechanically polished wafers. The wafers were then chem-mechanically polished, and measurements on the chem-mechanically polished surfaces were begun. Standardization activity this quarter consisted of continued coordination of two round robins on the four-probe method and participation in a third on the metal-oxide-semiconductor capacitance-voltage method, all in cooperation with ASTM Committee F-1 on Electronics. Experimental work on the capacitance-voltage method and further consideration of silicon wafer resistivity standards were deferred. Effort on spreading resistance methods was devoted to further study of procedures for preparing silicon wafers for examination by the scanning electron microscope (SEM) and initial study of probe impact with a force transducer.

Four-Probe Method — Mechanically polished bulk silicon wafers were measured using new experimental procedures in the study of current and probe force dependence of resistivity as measured by the four-probe method. The new procedures require that at each of twelve orientations of the wafer under the probe head, data for all current levels be taken before raising the probe to sample a new orientation. Further, by interchanging probe springs, data for all values of probe loading can be taken in order of increasing load before a slice is replaced by another specimen. In this way, and using a recently constructed slice centering apparatus that rapidly positions a slice such that its center is within 2 mils (50 μm) of the center of the four-probe array, it is possible to make all measurements at the same location at the center of the slice and vary all relevant parameters before there has been any significant change in the surface cleanliness. In addition, probe impressions were recorded at each value of probe loading immediately after measurement on each slice to assure that the probe itself remained essentially unchanged during the data taking.

The results appeared to show greater consistency of trend with current and probe force for each slice resistivity than had been observed heretofore. In general the relative sample standard deviation for the twelve measurements taken on a specimen at each current, probe-force combination was less than 0.35 percent and often was less than 0.2 percent. Exceptions to this were the two specimens in the 100 Ω·cm range where measurements made with low current (10 μA) and probe loading (25 and sometimes 50 g) yielded relative sample deviations that were as high as 1 percent. At the other extreme of the resistivity range (0.001 Ω·cm), measurements made with low current (10-30 mA) yielded relative sample standard deviations that were as high as 0.7 percent but were generally independent of loading. The source of difficulty at high resistivities was mainly due to the large noise susceptibility of the high impedance (up to 1 MΩ) voltage contacts. At the low end of the resistivity scale, scatter in the data was due predominantly to low signal level (as low as 20 μV).

On completion of the measurements on mechanically polished surfaces, the wafers were chem-mechanically polished and a new sequence of measurements was begun. (J. R. Ehrstein and D. R. Ricks)

Standardization Activities — Data has now been returned from all participants in the round robin on four-probe measurement of ultra-high resistivity silicon that is being run in conjunction with ASTM Committee F-1. The results are being tabulated for presentation to the committee.

No further measurement data have been received in the Committee F-1 round robin to measure four-probe resistivity of epitaxial layers with opposite conductivity-type substrates. (J. R. Ehrstein and D. R. Ricks)

A series of measurements was made as part of a Committee F-1 round robin in the metal-oxide-semiconductor capacitance-voltage method for measuring resistivity. (G. N. Stenbakken)

Spreading Resistance Methods — Work on definition of proper specimen preparation in order to obtain high-quality SEM photomicrographs of the contact damage made by spreading resistance probes continued. Although pictures of favorable quality were obtained from some wafers, the procedures tested to date do not give consistent results.
(W. J. Keery and J. R. Ehrstein)

A piezoelectric transducer of lead zirconate-lead titanate capped with an anvil of tungsten carbide has been acquired in an attempt to measure the force of a spreading resistance probe impacting a silicon wafer surface as a function of time. Because the transient response of the transducer structure to a fast event such as probe impact has not yet been determined it has not been possible to separate the probe impact signal from transducer generated "noise" such as ringing or reflection of pressure waves. (J. R. Ehrstein)

Plans: Study of the probe force and current dependence of four-probe resistivity measurements will continue with completion of data collection on chem-mechanically polished wafer surfaces and beginning of remeasurement of lapped wafer surfaces. Efforts to locate a suitable epitaxial reactor so that the wafer surfaces can be vapor etched will continue. Complete results of the round robin on four-probe measurements on ultra-high resistivity silicon and a status report on the round robin on four-probe measurement of resistivity of epitaxial wafers will be presented to ASTM Committee F-1 at its June meeting. Further work will be undertaken on clarifying the conditions necessary to take high-quality SEM photomicrographs of probe damage to the silicon surface and on characterization of the piezoelectric transducer intended for use in measuring the impact momentum of spreading resistance probes. Work on the capacitance-voltage method will resume with measurement on 15 Ω·cm *n*-type wafers diffused with boron.

3.2. CARRIER LIFETIME

Objective: To determine the fundamental limitations on the precision and applicability of the photoconductive decay method for measuring minority carrier lifetime and to develop alternative methods for measuring minority carrier lifetime in germanium and silicon which are more precise, more convenient, or more meaningful in the specification of materials for device purposes.

Progress: Reprogramming led to a significant decrease in the level of effort in this area. The experiment to study the effect of specimen current on specimen heating during photoconductive decay (PCD) measurements was completed. It was found that the current required to increase the surface temperature of the specimen by 0.5°C depended on both the resistivity and shape of the specimen but no universally applicable relationship could be developed. Analysis of this result and the results of previously conducted experiments (NBS Tech. Note 592, pp. 15-16) to establish specifications for specimen current and signal level was not completed. The experiment to compare the time mark and null methods for determining the voltage decay time was terminated prior to completion.
(R. L. Mattis and A. J. Baroody)

Several measurements were made on bulk crystals to compare the PCD and surface photovoltage (SPV) methods. Measurements made by the two methods on one specimen gave similar values while for another specimen the SPV value was only 1/40th the PCD value. The significance of these results cannot be determined until additional measurements are made.

The draft of the paper describing the application of the SPV method to measurement of carrier lifetime in epitaxial layers was completed.
(W. E. Phillips)

Experimental work on a preliminary study of measurement of carrier lifetime by the metal-oxide-capacitance method was completed. Details of this work are reported elsewhere [1]. (R. L. Mattis)

Plans: Work in this area, with the exception of investigation of the applicability of the SPV method to measurements of carrier lifetime in gold-doped silicon, will be suspended temporarily. Results of the SPV study will be reported as part of the task on gold-doped silicon.

3.3. INHOMOGENEITIES

Objective: To develop improved methods for measuring inhomogeneities responsible for reducing performance and reliability of germanium and silicon devices and, in particular, to evaluate a photovoltaic method as a means for measuring radial resistivity gradients in germanium and silicon circular wafers without contacting the flat surfaces of the wafers.

Progress: Study of the effect of uncertainty in the probe location on radial resistivity profiles determined by the four-probe method was completed. The asymmetry that results from applying off-center correction factors [1] to measurements on a mis-centered wafer as though it had been centered has been reported previously (NBS Tech. Note 520, pp. 8-9). Similar asymmetry was noted in several four-probe profiles made in connection with the present study. It was found that shifting the position variable a small amount improved both the symmetry of the four-probe profile and the agreement between the four-probe and photovoltaic profiles. In the example shown in figure 1, the original four-probe profile on a *p*-type silicon wafer was corrected by a shift in position variable 0.3 mm to the right and reapplication of the off-center correction factors. The photovoltaic profile along the same diameter is also shown for comparison.

The influence of the electrical quality of the measurement contact on the measurement of photovoltaic profiles was also studied. Photovoltaic profiles were measured along the same diameter of the *p*-type

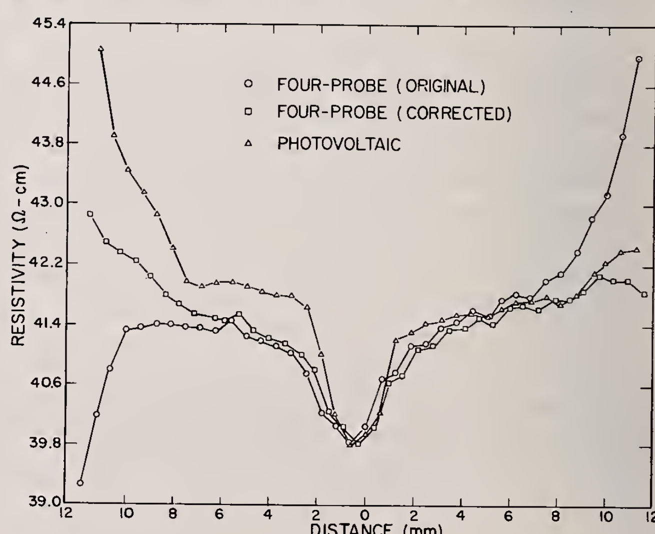

Figure 1. Four-probe and photovoltaic resistivity profiles along a diameter of a *p*-type silicon wafer showing effect of uncertainty in the probe location on the four-probe profile.

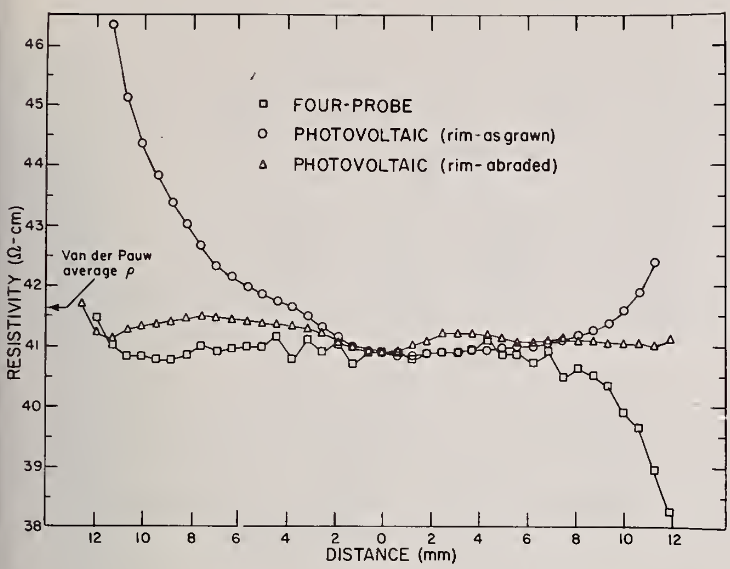

Figure 2. Four-probe and photovoltaic resistivity profiles along a diameter of a *p*-type silicon wafer showing the effect of contact quality on the photovoltaic profile.

silicon wafer with knife-edge contacts pressed into the as-grown wafer rim and into an abraded wafer rim. The former contacts were apparently less ohmic in nature than the latter as can be seen from the profiles in figure 2. The anomalously large resistivity gradient to the left of the wafer center indicated by the photovoltaic measurements made with the as-grown rim is a result of the barrier photovoltage generated by carriers which diffuse to the large barrier present at the measurement contact. A similar, but smaller, effect can be seen to the right of the wafer center, but only beyond about a half a wafer radius. This indicates that while both contacts were very nonohmic, the contact on the left was much less ohmic than the one on the right. In the measurements made with the abraded rim, the barrier photovoltage is significantly smaller; again the contact on the left appears to be somewhat less ohmic than the one on the right. The asymmetry of the four-probe profile may be due to slight miscentering of the wafer as noted above.

To obtain an indication of the reproducibility of the photovoltaic method, four resistivity profiles were made by the photovoltaic method along the same diameter of an *n*-type silicon wafer. The maximum difference between any of the four profiles, as shown in figure 3, was less than 2 percent of the average specimen resistivity. Since the wafer had a large non-radial gradient along the chosen measurement diameter and since the apparent resistivity profile of the wafer varied considerably as the measurement diameter was changed slightly, the reproducibility is considered to be indicative of that which would be obtained under less than ideal conditions.

A summary of the work to date on the photovoltaic method is being prepared for presentation [2]. (D. L. Blackburn)

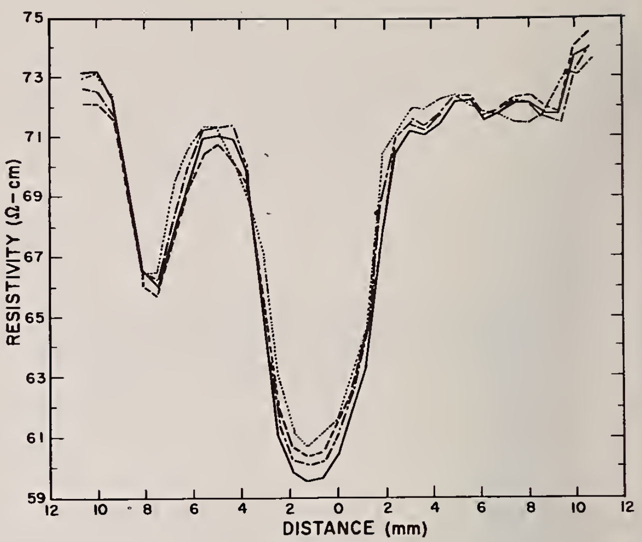

Figure 3. Four independently made photovoltaic resistivity profiles along the diameter of an *n*-type silicon wafer showing the degree of reproducibility that can be achieved.

Plans: Next effort in this area will be directed toward determination, in cooperation with ASTM Committee F-1, of the suitability of this technique as an alternative to the existing standard four-probe method for measuring resistivity variation. Because of the close relationship with resistivity measurements, results of future work will be included in the report of the resistivity task.

3.4. GOLD-DOPED SILICON

Objective: To characterize *n*- and *p*-type silicon doped with gold and to develop a model for the energy level structure of gold-doped silicon which is suitable for use in predicting its characteristics.

Progress: Gold was diffused into additional 10- and 20-Ω·cm, boron-doped silicon wafers to complete the range of gold concentrations for these resistivities. Also 90-Ω·cm, *p*-type wafers were diffused in an oxygen atmosphere at temperatures from 850 to 1250°C. After diffusion, 0.12 mm was lapped from each side of the wafers. A part of each slice was reserved for the determination of gold concentration by activation analysis and a Hall bar for electrical measurements was cut from the remainder.

Sets of *n*-type silicon wafers with resistivities of 5, 80, 400, and 2200 Ω·cm were prepared for gold diffusion at five temperatures from 850 to 1250°C. The present procedure is to clean the slices and evaporate 40 nm of gold on each side just prior to inserting them in the furnace. Control wafers without gold are also being included at two temperatures to determine if the heat treatment alone affects the electrical properties. (W. R. Thurber, T. F. Leedy, and W. M. Bullis)

To determine if the retrograde behavior of the resistivity in boron-doped material (NBS Tech. Note 592, p. 21) could be explained by a variation with gold concentration of the ionization energy of the gold donor level, Hall effect and resistivity measurements were made as a function of temperatute on two 20-Ω•cm, boron-doped specimens which had gold concentrations of 3.1 and 9.9 × 10^{16} atoms per cubic centimeter. The specimen with the higher gold concentration had the slightly larger ionization energy, but the difference was not considered significant. To serve as an explanation for the retrograde behavior of the resistivity, the specimen with the lower gold concentration should have had the larger ionization energy. (W. R. Thurber)

Work continued on study of the lateral uniformity of the diffused gold as determined by x-ray fluorescence with the scanning electron microscope. A Hall bar was cut from a diffused wafer to use in this study. The complex shape of the Hall bar facilitates examination of the various regions of the specimen after successive surface treatments. Aqua regia was used to remove any excess gold on the surface. Examination of the Hall bar in four areas showed it to be farily uniform except for occasional small clusters of gold 2 to 5 μm in diameter on the surface. A layer 5-μm thick has been removed from the surface of the bar preparatory to making x-ray fluorescence measurements at the locations previously examined to determine if the gold clusters seen on the surface extend into the bulk of the material. (W. J. Keery and W. R. Thurber)

Plans: After the gold diffusions are completed, a portion of each wafer will be sent for activation analysis and a Hall bar will be cut from the adjacent material. Hall coefficient and resistivity measurements will be made at room temperature on each of the bars. The investigation of the lateral uniformity of the gold diffusion process in the Hall bar specimen will continue.

3.5. SPECIFICATION OF GERMANIUM

Objective: To measure the properties of germanium crystals and to correlate these properties with the performance of germanium gamma-ray detectors in order to develop methods for the early identification of crystals suitable for fabrication into lithium-compensated gamma-ray detectors.

Progress: Standards pertaining to the measurement of lithium-ion drift mobility and Ge(Li) detector characterization are being developed in cooperations with ASTM Committee F-1 and IEEE Nuclear Instruments and Detectors Committee respectively. Additional semiconductor diodes were examined by the improved infrared response (IRR) technique. Response spectra were obtained for a heat-treated germanium specimen and a diode fabricated from a crystal of high-purity germanium. The infrared response of a lithium-drifted silicon diode was measured. Thermocouples were added to the IRR cryostat and preliminary measurements performed to determine the specimen temperature. The effects of the magnitude of applied bias on the IRR of germanium diodes were studied.

Standardization Activities — In cooperation with the Germanium Section of Committee F-1, two specimens from each of two different germanium crystals are being prepared according to a procedure outlined for the second round robin on carrier trapping measurements. The measurement of lithium-ion drift mobilities of these four specimens has been completed, and the diodes are undergoing further drifting prior to the detector trapping measurements. (H. E. Dyson and A. H. Sher)

Standard test procedures for characterizing germanium gamma-ray detectors have been approved by the members of the Nuclear Instruments and Detectors Committee [1] and have been submitted to ANSI Committee N·42 for approval as an American National Standard.
(A. H. Sher and J. A. Coleman)

Ge(Li) Detector Measurements — Work on the development and evaluation of an improved infrared response (IRR) technique has proceeded on two fronts. Measurements have been continued on different types of semiconductor diodes in order to further the qualitative understanding of details observed in the IRR spectra. Work has also begun on the study to determine the effects of various factors such as specimen temperature and applied bias on the results obtained. This is intended to aid in determining the capability of the method for obtaining quantitative information about the concentration of defect or impurity levels in the semiconductor specimens examined.

The IRR of a Ge(Li) diode fabricated from a *p*-type germanium crystal which had been heated to 800°C for 15 min and then cooled rapidly to room temperature has been measured. The spectrum of this diode, Ge(Li) 83-4, is shown in figure 4 together with spectra from three diodes previously discussed (NBS Tech. Note 592, pp. 22-26). Diode Ge(Li) 83-3 was fabricated from a specimen of the same germanium crystal as Ge(Li) 83-4 but was subjected only to the typical lithium-drifted detector fabrication process. Diodes Ge(Li) 13 and Ge(Li) 13Cu were fabricated from two specimens of another *p*-type crystal that had more than 10,000 dislocations per square centimeter; diode Ge(Li) 13Cu also contained approximately 10^{14} copper atoms per cubic centimeter. The spectra were taken with the specimens at a temperature of about 100 K. A 1-mm thick germanium filter was placed in the radiation path. For higher energies, to the right of the vertical dashed line in figure 4, a 640-line per millimeter grating was used in the monochromator. For lower energies to the left of the dashed line, a 240-line per millimeter grating was used.

In a study of thermally-induced acceptors in germanium, Logan [2] found from Hall effect data that the levels introduced into *p*-type

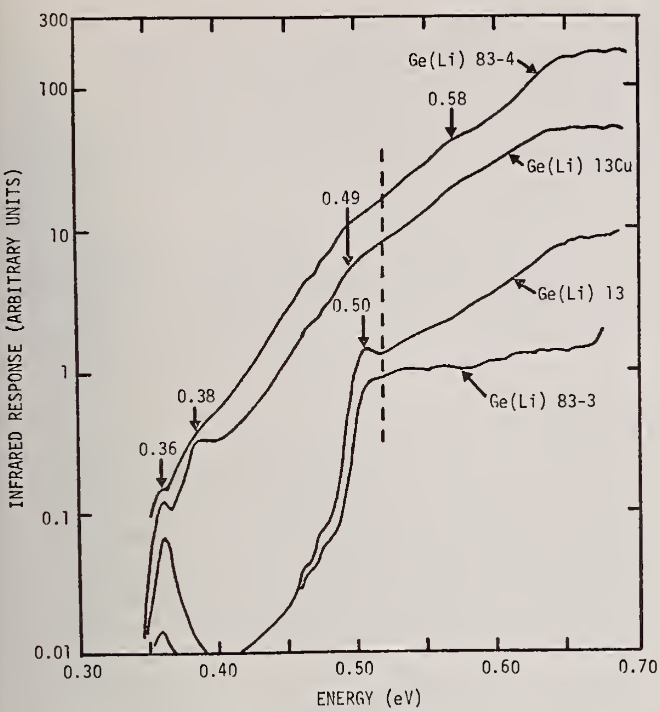

Figure 4. Infrared response spectra of four lithium-drifted germanium diodes.

crystals by heat treatment and subsequent quenching to room temperature were indistinguishable from those of copper. For a temperature of 800°C, the concentration of added acceptors was found to be about 10^{14} cm^{-3}. Miles [3] has studied acceptors arising from edge dislocations in plastically deformed germanium. Using measurements of specimen photoconductivity, he interpreted the observed photoresponse in terms of a band of dislocation acceptor states extending in energy from about 0.62 eV to 0.38 eV with a peak response at approximately 0.58 eV. The observed IRR spectrum of diode Ge(Li) 83-4 as shown in figure 4 is consistent with these models. A "knee" is observed at 0.58 eV on the broad response that extends to below 0.40 eV. Features observed at 0.38 and 0.49 eV that are similar to but less intense than those observed in the copper-doped specimen are attributed to the copper levels at E_v + 0.33 eV and E_c - 0.22 eV. No special precautions were taken to exclude copper from the furnace during the heat treatment of Ge(Li) 83-4; therefore, it is possible that some contamination might have occurred.

Measurements of IRR were also obtained from a diode fabricated from a specimen of high-purity germanium which initially contained a net donor concentration of approximately 7×10^{10} cm^{-3}. This diode was prepared at another laboratory* with the use of an indium-gallium regrowth technique for the p^+-contact and a lithium diffusion for the n^+-contact. Such a diode can be depleted to a depth of several millimeters. The IRR spectra obtained were similar to those of lithium-drifted germanium diodes of high

*Supplied by R. D. Baertsch, Semiconductor Branch, General Electric Company, Research and Development Center, Schenectady, N. Y. 12301.

quality such as Ge(Li) 83-4 except that the level at about 0.50 eV attributed to the lithium-defect interaction was not observed. This is consistent with the fact that lithium-drifting was not used to obtain the compensated or depleted region that is the only portion of the diode sensitive to either optical or nuclear radiation.

Preliminary IRR measurements were also made on a germanium alloy transistor and a lithium-drifted silicon diode. A special right-angle mount for the IRR cryostat cold finger was designed and fabricated for use with the transistor. Because of the small size of the device and the resultant low signal level, only the band-edge peak could be observed in the IRR spectrum. The signal-to-noise ratio was further degraded by the high leakage current characteristic of this type of device at 100 K. Suitable large area devices with low leakage current are required to continue further measurements of this type. The IRR spectrum of the silicon diode at approximately 100 K exhibited a band-edge peak at approximately 1.15 eV and a broad IRR distribution, peaked near 1.00 eV. On the low energy side of the band-edge peak, a shelf-like response was observed at approximately 1.12 eV, similar to that observed in the germanium diodes (NBS Tech. Note 555, pp. 16-21).

(A. H. Sher, W. J. Keery, and H. E. Dyson)

The IRR cryostat was modified to accept two chromel-constantan thermocouples. One thermocouple, mounted in the end of the cold finger above the point where the semiconductor diode is mounted, is to be used to monitor the temperature in all subsequent IRR measurements. The second thermocouple, mounted in a hole ultrasonically machined into a block of germanium of the same approximate dimensions as typical test specimens, is being used in the study of the temperature characteristics of the system. Preliminary measurements indicate that, as expected, the temperatures measured by the thermocouples depend upon the quality of the vacuum maintained in the cryostat. Typically, equilibrium temperatures about 95 K are obtained. In some instances, the germanium crystal was found to be as much as 4 K warmer than the cold finger. (W. J. Keery)

A series of measurements was made on several devices in order to ascertain the effects of the magnitude of applied reverse bias on the IRR spectral distribution. In general, it was observed that only the magnitude of the output signal depends on the magnitude of the applied bias. The same spectral features are observed when no external voltage is applied to the device as when a potential of several hundred volts is applied. Typically, the largest increase in output signal occurred for voltages ranging from 0 to perhaps 20 V, whereas from 20 to 200 V the increase was much smaller. In one germanium device, however, an approximately linear increase of signal with applied bias was observed over the range of 0 to 200 V. (W. J. Keery and A. H. Sher)

Plans: The interpretation of results obtained using the improved IRR technique on semiconductor diodes will be emphasized. This will entail the continuing examination of different types of radiation-damaged or specially doped crystals. Measurements will be carried out to study the effects of the germanium filter window on the energy of the spectral features observed as well as the effects of specimen temperature. Further IRR studies will be carried out on diodes fabricated from high-purity germanium. One such diode that will be investigated was intentionally doped with approximately 10^{11} gold atoms per cubic centimeter.

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4. METHODS OF MEASUREMENT FOR PROCESS CONTROL

4.1. DIE ATTACHMENT EVALUATION

Objective: To evaluate methods for detecting poor die attachment in semiconductor devices with initial emphasis on the determination of the applicability of thermal measurements to this problem.

Progress: Work reported previously (NBS Tech. Note 555, pp. 25-27) indicated that a measurement of transient thermal response of a semiconductor diode is, in principle, more sensitive to the presence of voids in the die attachment material between the semiconductor chip and header than is a measurement of the thermal resistance or steady-state temperature rise. In order that a valid comparison can be made, it is necessary to satisfy two conditions: 1) the procedures and equipment used to make the measurements must give reproducible results and 2) the spread in the measured thermal response of diodes without voids must be small compared to the change introduced by the presence of voids. The experiments reproted herein indicate that both these conditions have been met.

Long-term, single-operator measurements were made to check the reproducibility of the die attachment evaluation equipment. Measurements both of steady-state temperature rise and transient thermal response with a heating power pulse width of 5 ms, were made on commercial silicon mesa diode chips bonded to TO-5 headers. Over a six-week period, 25 measurements of forward voltage drop were made at times, $t_{meas.}$, 15 and 50 μs after removal of the heating power on each of four diodes. The temperature difference between the diode junction and case was calculated from the forward voltage temperature coefficient generated for each of the four diodes.

The average values and sample standard deviations of the junction-to-case temperature, listed in table 1, indicate a degree of reproducibility that was adequate, but not as great as was desired. Several ways to improve the reproducibility were considered. The reproducibility of the data for a given diode appeared to be limited by the meter used to

Table 1 — Results of Repetitive Junction-to-Case Temperature Difference Measurements to Demonstrate Reproducibility of Method

Device No.	Steady State, I_F = 0.3A		Pulse Width = 5ms, I_F = 0.6A	
	$t_{meas.}$=15μs	$t_{meas.}$=50μs	$t_{meas.}$=15μs	$t_{meas.}$=50μs
1	17.4 ± 0.41°C	16.9 ± 0.53°C	10.6 ± 0.37°C	9.4 ± 0.42°C
2	16.9 ± 0.55	16.4 ± 0.64	9.6 ± 0.44	8.2 ± 0.53
3	17.9 ± 0.54	17.6 ± 0.69	10.6 ± 0.40	9.5 ± 0.46
4	16.1 ± 0.47	15.8 ± 0.69	8.4 ± 0.47	7.2 ± 0.54

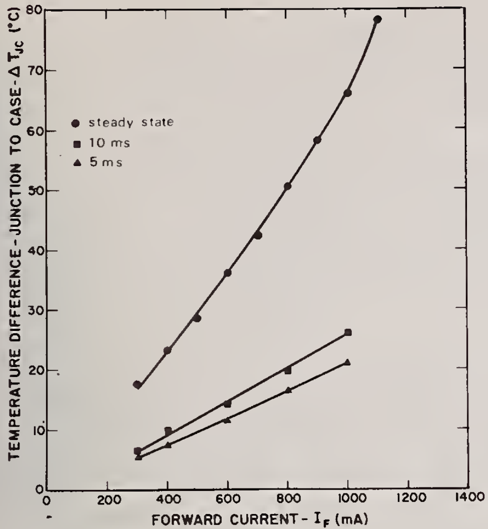


Figure 5. Junction-to-case temperature difference as a function of diode heating current for steady-state heating and for 5- and 10-ms wide heating-power pulses.

measure the forward voltage drop. Increasing the sensitivity of the meter would be expected to reduce the observed sample standard deviation. Increasing the magnitude of the observed temperature difference would also be expected to permit greater reproducibility. Measurements of junction-to-case temperature difference, ΔT_{JC}, were made as a function of diode heating current for steady-state heating and for 5- and 10-ms wide heating power pulses. The forward voltage of the diode was measured 15 μs after switching to the 1-mA measuring current. Case temperature was held at 25°C. Typically, as depicted in figure 5, it was found that doubling the diode heating current at least doubles ΔT_{JC}.

To compare the magnitude of the spread in thermal response of diodes without voids with the change in thermal response introduced by the presence of voids, measurements were made on groups of diodes bonded to TO-5 headers with 0-, 20-, and 40-percent void areas. The 20- and 40-percent void areas were produced by 20- and 29-mil (0.51- and 0.74-mm) diameter dimples ultrasonically machined into the bonding surface of gold-plated TO-5 headers (NBS Tech. Note 560, pp. 27-29). Ten control devices without voids were bonded with each group of 10 to 15 devices with voids. Average values and sample standard deviations of junction-to-case temperature difference for the two groups of control devices used in this experiment are summarized in table 2. Group H controls were associated with the diodes with 20-percent void area; group I controls, with diodes with 40-percent void area. Measurement of steady-state temperature rise and transient thermal response with heating-power pulse widths of 5, 7, and 9 ms were made 50 μs after removal of the heating power. In view of the previous experiment, the heating current pulses were increased to 800 mA for the measurement of transient thermal response. The observed sample standard deviations in these measurements made under a given set of conditions on a group of devices are not much greater than those previously observed in measurements on a single device made over an extended period of time.

Table 2 — Results of Junction-to-Case Temperature Difference Measurements on Control Diodes

Heating Current	Pulse Width	Group H	Group I
300mA	steady state	14.09±0.52°C	15.0±0.52°C
800	5ms	8.7 ±0.44	9.2±0.58
800	7	9.9 ±0.50	10.4±0.49
800	9	10.08±0.53	11.3±0.55

Measurements were also made under the same conditions on the devices with voids and the results were compared with the appropriate results on control devices. The percent increase in ΔT_{JC} of the devices with voids over the average ΔT_{JC} of the controls for the various heating power pulse widths (including steady-state temperature rise) was, in every case, greatest when measuring the transient thermal response as opposed to the steady-state temperature rise measurement. The percent increase in sensitivity to voids of the transient thermal response measured for power pulses of 7 and 9 ms over that of the steady-state thermal response ranged from 30 to 700 percent. For all but two of the devices measured the percent increase in sensitivity to voids was above 90 percent for the readings made with power pulses 7- and 9-ms wide.

The measurements thus far indicate that a heating power pulse width between 7 and 10 ms should be used for maximum sensitivity to voids. It was also found that although the spread in ΔT_{JC} was small for the devices with no intentional voids, this was not the case with the intentionally voided devices. No conclusions regarding the relationship between void size and the change in ΔT_{JC} can be drawn from these data until the actual void sizes are determined by direct measurement.

(F. F. Oettinger and R. L. Gladhill)

Plans: Measurements of thermal response on diodes with controlled voids of various diameters will continue. The effect of introducing a more sensitive meter into the measuring circuit will be studied.

4.2. WIRE BOND EVALUATION

Objective: To survey and evaluate methods for characterizing wire bond systems in semiconductor devices and where necessary to improve existing methods or develop new methods in order to detect more reliably those bonds which eventually will fail.

Progress: Two new ultrasonic wire bonding machines were recieved during the quarter. The first was for bonding round wire and is presently being set up for statistical analysis of bonding parameters. The set-up procedure, and typical bond pull strength data are given in this report. The second machine was designed to handle ribbon wire. Initial modifications and set-up procedures have been carried out. The wire tester was completed; initial tests showed distinct differences in uniformity of three different wire samples. A special bonding-force measuring apparatus is described that gives reproducible non-operator-dependent results. The self-tuning ultrasonic power supply and transducer were received and initial studies of the ultrasonic bonding process were made with this equipment.

At the request of a sponsor, a study was made of wire bond failures in transistors subjected to cyclical applications of power. A calculation indicated that use of as large a loop height as is permitted by device and package geometry minimizes such failures.

Four different semiconductor manufacturers and one Government laboratory were visited at the request of sponsors. Capacitor microphones and magnetic pickups were distributed to the four manufacturers together with instructions for their use. In one case, use of this equipment and associated technology for process control developed as a part of this task resulted in a reduction in rejection rate due to faulty bonds at visual inspection by more than a factor of two.

Initial Set-up Procedures for Ultrasonic Bonding Machines — A new ultrasonic bonding machine, designed to bond round wire, was acquired to replace the borrowed machine that had been used previously for the statistical analysis of various bonding parameters. The new machine was delivered with a number of problems involving the motor speed control, the wire clamps, the wire feed system, and the loop height control. These problems had not been completely solved at the end of this reporting period.

Initial experiments were performed to establish the optimum operating parameters for the new machine. These are the values of force, time, and ultrasonic power which yield the most repeatable pull strengths for a group of consecutively made bonds. The results of this initial work are reported primarily to indicate the methods that are presently used to set up the bonding machine. From previous experience, acceptable force values for 1-mil (25-μm) diameter aluminum wire lie between 20 and 30 gf (196 and 294 mN). Initial experiments included four such values: 21, 24, 27, and 30 gf (206, 235, 264, and 294 mN). At each of these force settings, four values of time were selected: 47, 75, 100, and 130 ms. For each of the combinations of force and time, bonds were made at five or six different power settings, from the lowest to the highest power setting that is possible to make a bond. At a power level that is too low, the wire does not stick to the pad. At too high a power level, the bond is so greatly deformed that it is severed at the heel during bonding. The The reported data lie within these two extremes.

Bonds were made in groups of 10 at each of the different combinations of force, time, and power for the first bond and then repeated separately for the second bond. Bonds were tested by pulling to destruction. In order that the first bond be tested independently of the second, epoxy was placed over the second bonds of the bond loop to prevent breakage at that point. The first bonds were similarly epoxied when testing the second bonds. In all, 186 groups of 10 bonds each were tested.

Typical results for first and second bonds are shown in figures 6 and 7, respectively. In each case the bonding force was 27 gf (264 mN); the bonding time was 47 ms for the first bond and 100 ms for the second bond. Each point on the graph represents the average pull strength for 10 bonds made on a single level with a ratio of bond separation to loop height, d/h, equal to 2.5. The measured pull strength should be multiplied by approximately 0.8 to give the actual force in the wire at breakage. In general, the shape of the curves for the first and second bonds differed, particularly in the low-power regions. The pull strength of the second bond drops to a lower value before lift off occurs than is the case for the first bond. In the high-power region, however, the pull strength for the first bond falls off more rapidly than that of the second bond. The reasons for this behavior are not fully understood but may be related to the crack that exists in the heel of the first bond. The error bars in both figures, which represent the 95 percent confidence interval for the mean, are at least twice as large as desired, apparently resulting from the problems with the wire clamp and loop height control mentioned earlier.

The next step in the optimizing procedure is to select a specific force setting and choose reduced power and time ranges based on the data obtained. The experiment is then repeated and further reduced ranges are

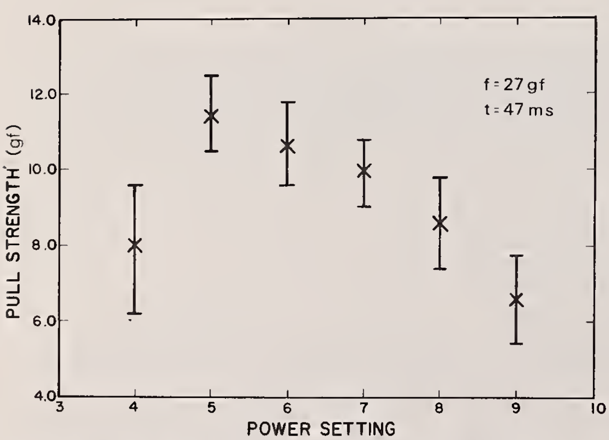


Figure 6. Measured bond pull strength for a first bond as a function of machine power setting.

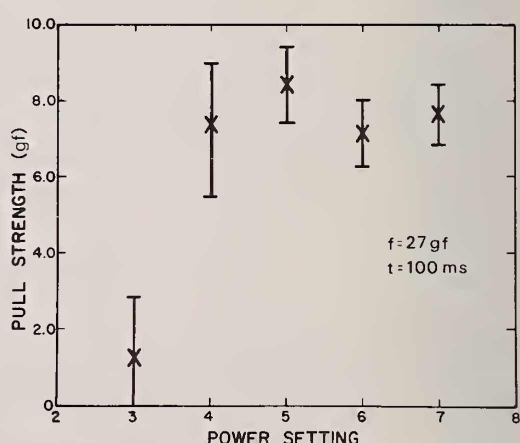


Figure 7. Measured bond pull strength for a second bond as a function of machine power setting.

Figure 8. SEM photomicrograph of a well made ribbon wire bond. (Magnification 530 X)

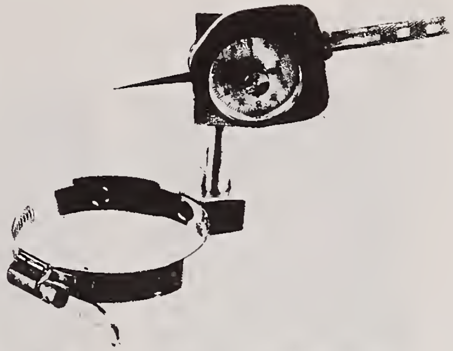


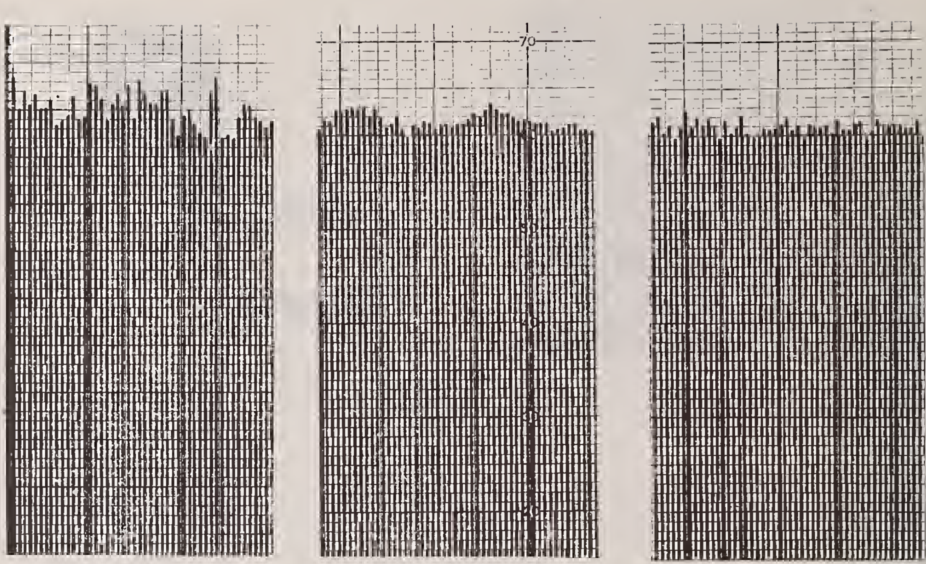
Figure 9. Fixture used in measurement of bonding force.

selected until the single best operating point is obtained. This procedure is expected to yield sample standard deviations small enough (≲ 1 gf) to perform the desired experiments. (K. O. Leedy and C. A. Main)

Ribbon Wire — A new ultrasonic bonding machine designed to bond ribbon wire was received during the quarter. Considerable effort was required to put the machine in working order. This included suppressing electrical transients, polishing the wire clamps, modifying the transducer-motion cam, and designing a new work holder. Since more changes are anticipated, this bonder has not been through the extensive set-up procedure described above. Nevertheless, for a series of single level bonds made with a d/h ratio of 2.5 (tensile force in the wire approximately 0.8 times the measured pull strength) the average measured pull strength was 10.1 gf (99 mN) with a standard deviation of 0.6 gf (5.9 mN). The ribbon wire used for the test had a cross sectional area of approximately 1.5 by 0.5 mil (38 by 13 μm), equivalent to 1-mil (25-μm) diameter round wire, and had a tensile strength of approximately 10 gf (98 mN). Figure 8 shows an SEM photomicrograph of a well made ribbon wire bond. Very little deformation is evident. (H. K. Kessler)

Bonding Force Measurement — The bonding force is the most critical parameter in an ultrasonic bonding schedule. Small changes in the value of the bonding force can markedly affect the nature of the bond. Yet the bonding force is measured on most bonding machines by a hand-held gage, and the force value obtained may depend greatly on the skill of the person making the measurement. Therefore, the special force-gage mounting future shown in figure 9 was designed to clamp onto the bonding machine work-stage. In use, the unit is moved against the transducer until the bonding tool rises 5 mils (125 μm) as shown by comparison with a 5-mil thick shim placed on the work-stage next to the tool, and the indicated force is read. (H. K. Kessler and A. W. Stallings)

Wire Tester — Evaluation of the wire indentation tester described previously (NBS Tech. Note 592, pp. 43-44) has revealed several problems associated with the high sensitivity inherent in its application. These



a. Wire rejected by a device manufacturer.

b. Wire not specified as ultrasonic bonding quality.

c. Wire specified as high-quality for ultrasonic bonding.

Figure 10. Recorder traces of impressions made in three different specimens of 1-mil (25-μm) diameter aluminum (1% silicon) bonding wire.

include slow drift of the tester assembly due to temperature changes, drift of the recorder and other instruments, and insufficient dust shielding. Thus far no method has been devised for calibrating the depth of impression on an absolute basis to within the desired 5 μin (0.13 μm). Therefore, the data must be compared on a relative basis. A series of impressions made on three different grades of 1-mil (25-μm) diameter aluminum (1% silicon) wire is shown in figure 10. Each line represents one impression. Impressions were made each ¼ in. (6.3 mm) for a distance of about 12 in. (0.3 m). The differences in uniformity can be clearly seen by the variations at the top of the repeated traces. The higher the trace, the smaller the impression and the harder the wire. Further experiments must be conducted to establish the optimum depth of impression to produce the maximum resolution. No correlation was found between variability of the wire impressions and the average tensile strength of the wire as measured with the hot-melt-glue puller (NBS Tech. Note 488, pp. 22-24). The wire specified as high-quality for ultrasonic bonding exhibited considerably less variability in the hot-melt-glue-puller measurement than the other two samples which showed about the same variability when measured with the puller. Additional development is necessary before the wire tester can be used to evaluate bonding wire quantitatively. (H. K. Kessler)

Transistor Failure Resulting from Slow-Thermal Cycling — In some applications, such as telemetering, transistors are operated within their power ratings but are subjected to cyclical applications of power. Many transistors have been observed to fail under such conditions of thermal cycling at slow rates (2 or more minutes per cycle). This failure has been traced to aluminum interconnection wire fatigue resulting from repeated thermal-expansion-induced wire flexing [1, 2]. A study was made of the wire bond flexure resulting from such cycling of 500-mW, 50-mA transistors. This low current does not produce significant Joule heating of the lead wires. However, the die may reach 180°C and cause considerable thermal expansion. For the thermal expansion calculation, the die was assumed to be the sole source of heat. The analysis was made specifically for the 1-mil (25-μm) diameter post-to-die aluminum wire interconnection in a particular transistor in a TO-18 can, but it is generally applicable to other devices with similar current and power ratings.

It was found that the changes in loop height and the angle of the wire at the bond heel were approximately proportional to the dissipated power and inversely proportional to the equilibrium loop height. Since wire flexing is reduced as the loop height is increased, failures due to wire flexing are minimized if the largest loop height permitted by device and package geometry is used. For discrete transistors in TO-18 cans, a loop height of 5 to 10 mils (0.13 to 0.25 mm) is recommended when these devices are subjected to thermal cycling during operation.

An alternative solution to using aluminum wire with large loop heights is to use gold bonding wire. The basis for the above flexure calculation is the differential coefficient of expansion between the wire and the iron-nickel-cobalt alloy header. This differential is significantly smaller for gold wire than for aluminum wire. The use of gold wire with the same loop height as aluminum reduces the wire flexing in the device by about one half. In addition, gold wire has a much higher resistance to flexure fatigue. To realize the advantages of gold wire, consideration must be given to the voids that form with intermetallic compounds if the gold wire is attached directly to aluminum films and is subjected to too high a temperature during testing or operation.

In an earlier analysis [3] of lead wire flexing due to heating, the expansion due to self heating by high current pulses was considered in addition to that induced by chip heating. Although the details of this calculation differ from the present one, the conclusion that the greater the loop height the less pronounced is the wire movement was also reached.
(W. E. Phillips)

Pull-Strength Acceptance Criterion — Most wire-bond acceptance criteria do not take account of the degradation of the bond during thermal cycling. Available data [2] suggest that failures due to thermal cycling occur principally near the bond to the semiconductor die. This suggests that a bond pull-strength criterion that is dependent upon the quality of the metallurgical weld, the bond deformation, and strain or cracking in the bond heel is essential. One possible criterion (often termed bond efficiency [4, 5]) that may satisfy the above requirements is based upon the ratio of the actual breaking force in the wire, T_B, to the tensile strength of the wire, T_T, Determination of the minimum value of T_B/T_T for which satisfactory metallurgical welding and bond deformation are indicated and minimum cracking or strain has occurred in the heel region would be valuable in specifying bond quality in terms of actual bond characteristics.

For example, consider a typical 1-mil (25-μm) diameter aluminum wire with a tensile strength of 18 gf (176 mN). If the minimum acceptable value of T_B/T_T be chosen as 0.4, a bond to be acceptable must break with a force not less than 7.2 gf (70 mN) in the wire. To translate this into acceptable measured pull strength it is necessary to consider the geometry (NBS Tech. Note 555, pp. 31-35). For a typical bond pair on a device in a TO-18 can with a chip thickness of 0.125 mm, a post height of 0.25 mm, a bond separation of 1 mm, and a loop height of 0.1 mm pulled vertically halfway between the bonds, a minimum bond pull strength of 3.3 gf (32 mN) would be required. If the wire tensile strength were reduced to 13 gf (127 mN), as might occur as a result of annealing during heat-sealing or testing the device, a minimum bond pull strength of 2.4 gf (24 mN) would be required. If, on the other hand, a value of T_B/T_T of 0.7 be required for acceptance, then bond pull strengths of 5.7 or 4.1 gf (56 or 40 nM) would be required for the wire with tensile strength of 18 or 13 gf, respectively.

Although determination and verification of an appropriate value for T_B/T_T is, at present, beyond the scope of this task, such a criterion based upon actual bond parameters is of sufficient importance to be brought to the attention of persons interested in this field. Ratios of T_B/T_T from 0.8 to 0.9 have been achieved using wires of very different tensile strengths when proper bonding machine parameters were chosen [5]. Bonds made at NBS typically show T_B/T_T ratios of 0.8 or more, and bonds made on certain high-reliability device production lines show T_B/T_T ratios consistently greater than 0.7. Ratios for bonds made on typical commercial lines might be expected to be lower. (G. G. Harman)

Consultations — At the request of a sponsor, a comparison was made between two slow-thermal-cycle test systems, one at a semiconductor manufacturing plant and the other at the sponsor's laboratory. Two informal reports were submitted to the sponsor covering aspects of the comparison. (G. G. Harman)

Four visits were made to semiconductor device producers at the request of sponsors. Calibrated microphones and magnetic pickups were distributed along with preliminary copies of instructions for their use [6]. Measurements were made of the ultrasonic vibration amplitude of the bonding machines at each site. All had identical equipment. Significant variations in tool tip vibration amplitude and bonding time were found; these were more characteristic of the particular producer than of the packages being bonded. In one case, a factor of two variation in power supply power dial settings was observed to produce the same tool tip vibration amplitude from one bonding machine to the next. Twenty-five percent variations were common. This is attributed to variations in transducer conversion efficiency rather than to properties of the power supply. In one case, involving the least efficient transducer, the technician had apparently been unwilling to increase the power dial setting sufficiently to compensate for the transducer inefficiency; instead he increased the bonding time in order to establish good bonding. As it turned out, that machine produced the most reliable bonds on that assembly line so that it would appear that an optimum bonding schedule had been achieved. Although the intuition of a good technician is valuable in setting up a bonding machine, only by actual measurement of the tool vibration amplitude is it possible to set up a known bonding schedule on a number of apparently identical bonding machines.

(G. G. Harman and H. K. Kessler)

In-Process Bond Quality Determination — A system of studying ultrasonic bonding by listening to an audible beat frequency of the vibration bonding tool has been described (NBS Tech. Note 592, pp. 39-41). This early work was performed with two fixed-frequency power supply-transducer combinations. This quarter, initial studies of the ultrasonic bonding process were made with a new self-tuning transducer-power supply system that the experiment was originally designed to use. During normal bonding, the fundamental frequency was observed to shift by several hundred hertz. This frequency shift was very easily distinguished when the operator listened to the audible beat frequency of the second (120 kHz) or third (180 kHz) harmonic. Qualitative experiments were performed under a number of conditions and on different substrates. For instance when the wire was intentionally placed near the edge of the bonding tool the sound was characteristic of the wire being squeezed out from under the tool when the ultrasonic energy was applied. Different sounds were heard when the bonding took place on a normal aluminum bonding pad, to silicon oxide, or only half on the pad. Many variables were encountered. The most sensitive response was obtained when the local oscillator was on the low side of the particular harmonic chosen (generally the second harmonic was preferred), but in some cases the reverse was true. Identification and control of the important parameters of this measurement system are still under investigation. (G. G. Harman)

Bibliography and Critical Review — The draft of the bibliography was completed and work on the final draft of the critical review survey paper was continued. (H. A. Schafft)

Plans: Experimental and statistical analysis of significant factors in the wire bond pull test will continue. Evaluation of ribbon wire for ultrasonic bonding will continue. Work on the wire indentation tester will continue. Further assistance will be given to sponsors in connection with problems encountered on device production lines. Calculations of the wire flexing due to heating of the device during thermal cycling will be refined. Further work on electronic mixing of bonding tool ultrasonic signals with local oscillators will continue in an effort to better understand and control bonding. The bibliography will be submitted for publication as an NBS Technical Note and work on the final draft of the critical review survey paper will be continued.

4.3. PROCESSING FACILITY

Objective: To establish a microelectronic fabrication laboratory with the facilities and procedures necessary for the production of specialized silicon devices and for use in research in measurement methods.

Progress: Considerable effort was spent in attempts to improve the electron beam evaporation system (NBS Tech. Note 592, p. 45). The initial experience with the unit demonstrated that aluminum evaporation rates greater than 5 nm/s could be achieved on silicon substrates. With the use of the planetary substrate holder it was found that the variation of thickness of deposited aluminum was less than 1 percent across the surface of any one substrate and less than 2 percent from substrate to substrate in a given run. All thickness measurements were performed with a multibeam interferometer. Typical deposition thicknesses were in the range of 0.6 µm to 1.0 µm.

However, during the initial testing period, excessive pressure increases were noted during the time the electron beam current was energized. The pressures further increased as the system was run. Typically the pressure within the chamber increased from 5×10^{-7} to 5×10^{-5} Torr when the beam was established. The latter pressure is too high for the production of high quality films because residual gasses are entrapped in the film. The result is oxidized aluminum having a milky appearance, poor electrical conductivity, and poor etching characteristics. At this time the reason for the pressure increase in uncertain, but it is suspected that secondary electrons, released from the aluminum charge as the primary beam strikes it, outgas the surface of the chamber providing the pump with an excessive gas load. In an attempt to provide a greater throughput of gas, all vacuum components were disassembled, cleaned, and re-installed. The diffusion pump was charged with a lighter molecular weight oil than used previously.

Installation of a new three-tube diffusion furnace was begun. The necessary electrical power was made available for its operation and some minor defects in the temperature controllers and overheat systems were corrected. Delays in the fabrication of the quartzware and procurement of the flow meters made it impossible to complete the furnace installation. (T. F. Leedy and J. Krawczyk)

Plans: Work will continue on both the completion of the diffusion furnace installation and the improvement of the electron beam evaporation system. The rebuilt vacuum system will be tested to determine if the gas pressure can be reduced during evaporation. If necessary, other measures, such as the installation of a larger diffusion pump or cryogenic pumping of the chamber during evaporation, will be considered. Either of the measures would involve extensive modification of the system.

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5. METHODS OF MEASUREMENT FOR SEMICONDUCTOR DEVICES

5.1. THERMAL PROPERTIES OF DEVICES

Objective: To evaluate and, if necessary, improve electrical measurement techniques for determining the thermal characteristics of semiconductor devices.

Progress: Work on the literature search and compilation of articles for the bibliography on methods for measurement of thermal resistance and transient thermal response of semiconductor devices were postponed. After reviewing a portion of the first draft of the survey paper on electrical methods for these measurements it was decided to utilize the relevant portions as an introduction in an appropriate report on the results of the semiconductor device thermal studies now in progress rather than publish an independent survey.

Work on the implementation of a preliminary thermal resistance round robin being conducted by JEDEC Committee JC-25 on Power Transistors was continued. Completed questionnaires on the capabilities of the available on-line thermal resistance measuring equipment have been received from 7 of the 10 members of Committee JC-25. (F. F. Oettinger)

Both the d-c and dynamic procedures for calibrating base-emitter voltage, V_{BE}, as a function of case temperature have been reported previously (NBS Tech Note 571, pp. 35-37). It was noted that at low power levels the d-c calibration procedure could give erroneous values of computed thermal resistance due to the presence of non-thermal switching transients, but that a reasonably good correction for such low level distortions could be effected by using the appropriate dynamic calibration procedure.

Measurements were made on a variety of transistors rated at 20 to 35 W to compare the dynamic calibration procedure with the d-c procedure using various measuring or calibration currents. In the dynamic procedure, V_{BE} was measured 10, 20, 50 and 100 μs after the transistor under test was switched from an operating condition of collector-emitter voltage of 1 V and collector current of 1 A to the test condition. In both procedures, the base current was controlled at 6.5 mA during measurement of V_{BE}. It was found that for the transistors tested, the measuring base current was small enough to avoid significant heating of the base-emitter junction and large enough to reduce the non-thermal switching transients to manageable proportions. Typical plots of V_{BE} as a function of case temperature for the various conditions, shown in figure 11, are essentially parallel straight lines. Although the lines are separated as a result of both a cooling of the junction and the decay of the electrical component of the base switching transient, the slopes are essentially the same. Therefore, if the thermal resistance is calculated from a difference in temperature and power between two different power levels, the

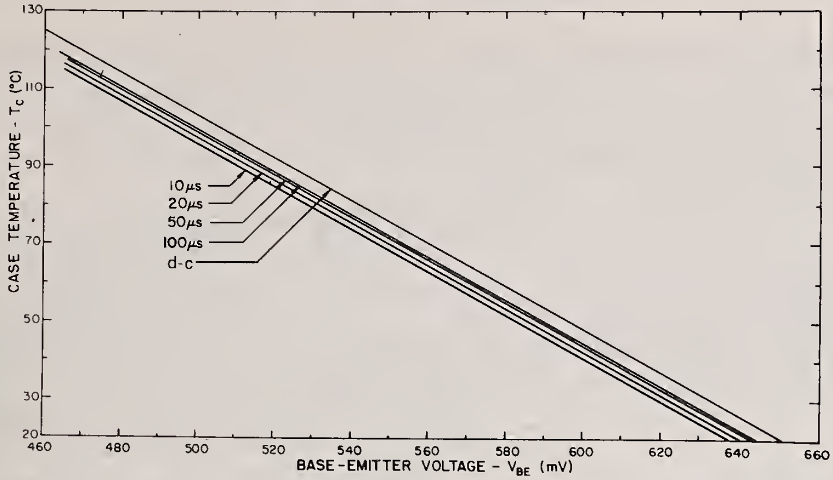

Figure 11. Comparison of dynamic and d-c calibration of a 25-W, silicon *n-p-n* transistor.

dynamic calibration does not afford any advantage provided that the measurement is not made at very low power levels where V_{BE} is not a linear function of power (NBS Tech. Note 592, pp. 52-53).

Long-term, single-operator measurements were undertaken to check the reproducibility of the equipment for measuring thermal resistance of power transistors. Ten measurements of V_{BE} were made both 10 and 20 μs after the termination of the heating power pulse over a period of three weeks on three transistors of the same type under conditions that would not stress the transistors. From these data the junction temperature was determined with the use of appropriate calibration curves. It was assumed that any significant change in the parameters of the transistors would not occur simultaneously in more than one unit at the same time and in the same way. During this period, the electrical characteristics of one of the transistors changed so it was dropped from the experiment. If the data for the remaining transistors are assumed to be normally distributed, it can be asserted with 95 percent confidence that the standard deviation of the junction temperature is less than 0.51°C. Other measurements suggest that over a workable range this standard deviation does not depend significantly on the magnitude of the junction temperature. Therefore, the relative standard deviation can be reduced by increasing the heating power.

In a continuation of the study in which it was found that for the three transistor types tested, for collector currents of 100 and 200 mA and for collector-emitter voltage levels below those at which current constriction occurred the thermal resistance, R_θ, was essentially constant (NBS Tech. Note 571, p. 37), R_θ was measured as a function of power for collector current levels up to 1 A. The measurements at both the higher and lower current levels showed similar results although as the collector current was increased the devices with low gain or relatively high saturation voltage levels showed increased non-thermal switching

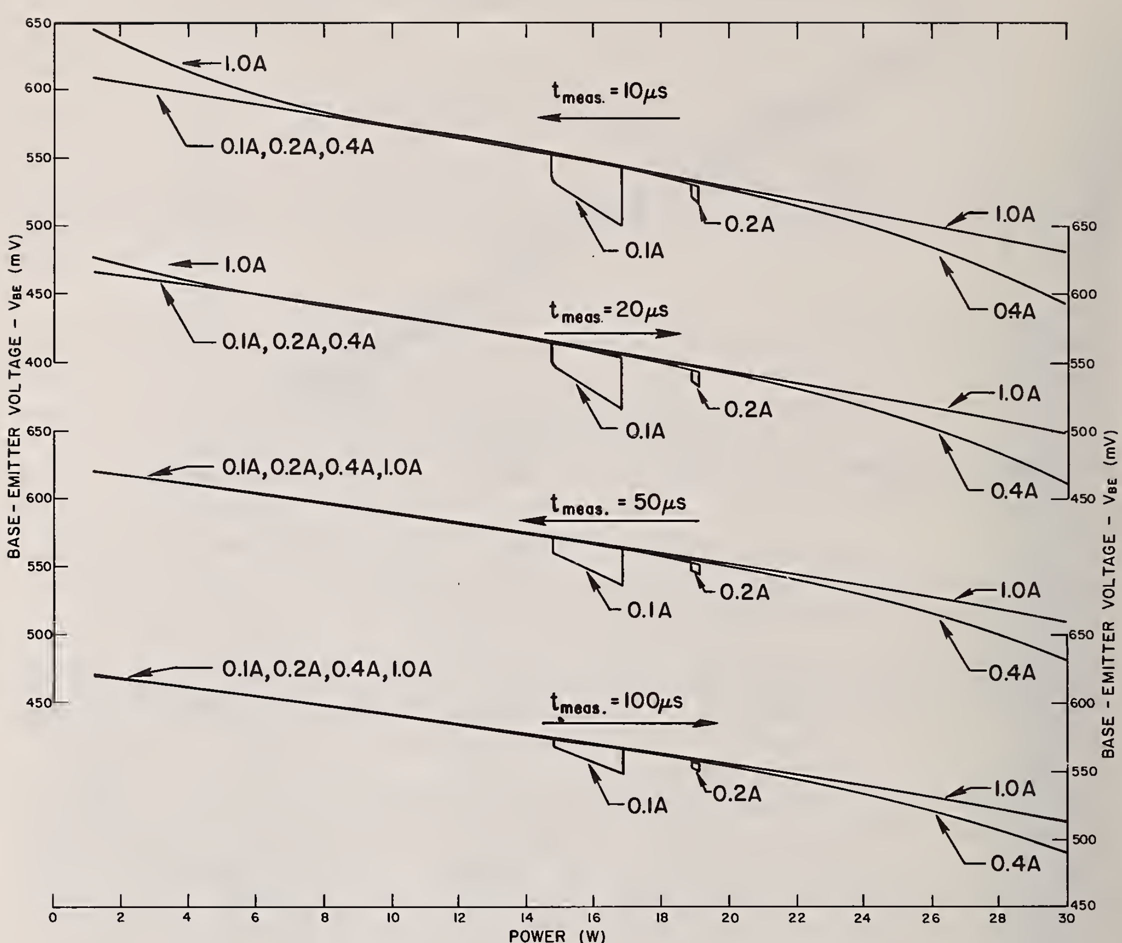

Figure 12. Base-emitter voltage of a 35-W, triple-diffused, silicon transistor measured as a function of applied power for collector currents of 0.1, 0.2, 0.4, and 1.0 A, at times 10, 20, 50, and 100 μs after removal of power.

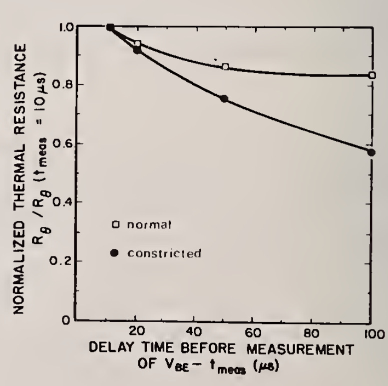

Figure 13. Normalized thermal resistance of a 35-W, triple-diffused, silicon transistor measured as a function of delay time for both current constricted and normal (non-constricted) operating conditions.

transient effects at low and, in some cases, intermediate power levels when V_{BE} was measured 10 μs after the termination of power pulse. Increasing the time between the termination of power pulse and the measurement of V_{BE} decreased the effects of these non-thermal transients.

The curves depicted in figure 12 are plots of V_{BE} measured as a function of power dissipation for various collector currents and measurement delay times on a 35-W, triple-diffused, silicon transistor. The measuring base current was 6.5 mA and the case was held at 25°C. The thermal resistance was calculated from the raw data by dividing the slope of the linear portion of the $(V_{BE})_{meas}$ — power curve by the slope of the d-c calibration curve:

$$R_\Theta = \left(\frac{\Delta V_{BE}}{\Delta W}\right)_{meas} \left(\frac{\Delta V_{BE}}{\Delta T_C}\right)^{-1}_{d\text{-}c\ calib}.$$

As previously reported (NBS Tech. Note 571, p. 37), if thermal hysteresis occurs when a hot spot is formed, the hot spot remains during the measurement of R_θ even though the power to the device under test is interrupted for periods of time up to 120 μs. The data in figure 12 for a collector current of 0.1 A also indicate that while the current is constricted, the measured thermal resistance is apparently constant. Further studies are needed to determine how closely junction temperatures calculated from the slope of the $(V_{BE})_{meas}$ — power curve during the current-constricted operating mode approximate the actual hot-spot temperature. This depends in part on the distribution of the measuring current when the device is operating in the current-constricted mode. Figure 13 shows the effect of delaying the measurement of V_{BE} in both the normal (non-constricted) and constricted operating conditions based on the 0.1-A curves of figure 12. As expected, the curves indicate that the larger the temperature difference involved, the greater is the error in delaying the measuring of V_{BE}.

To determine the general applicability of various techniques for use as industrial standards, a study was begun to compare other widely used methods for measuring thermal resistance to the common-emitter, base-and-collector-switching technique. This technique, in which both the base and collector of the transistor under test are momentarily opened and a constant current of a given magnitude is applied to the base, was compared to a collector-only-switching method, in which the constant base drive that is applied during the heating cycle is continued when the collector is momentarily opened for test purposes. The measurement of thermal resistance by the collector-only-switching method is usually made by applying a given collector voltage and collector current during the heating cycle and computing the dissipation. The value of V_{BE} measured during the collector-off period is then used to determine junction temperature from a calibration curve of V_{BE} as a function of case temperature (assumed equal to the junction temperature during calibration) for the same magnitude of base drive used during the transistor heating period. Measurements indicated that erroneous values of thermal resistance could be obtained if measurements were made only at zero power and at a specified higher power level. Error can be minimized by using a procedure similar to that developed for the base-and-collector-switching method. Measurements were made using the same collector current at two different levels of collector voltage, V_{CE}, in the linear portion of the V_{BE} — power curve. The thermal resistance was calculated by taking the difference in junction temperature, as determined from the appropriate d-c calibration curve relating V_{BE} to case temperature, and dividing it by the difference in power between the two voltage levels used. It should be noted that since the collector current was kept constant for the two different collector voltage measurements, two different base current drives and therefore two different calibration curves had to be used. Measurements of R_θ by this technique are valid only if the two calibration curves have essentially the same slope. Measurements of thermal resistance determined by both methods on a 35-W, triple-diffused, silicon transistor are given in table 3. Measurements of V_{BE} were made 10, 20, 50, and 100 μs after the termination of the heating power pulse. Power levels of 10 and 20 W at a collector current of 1 A were employed. The difference between the measured thermal resistance using the two methods in all cases is less than 3 percent. The data also show the cooling effect due to increased delay times when measuring V_{BE}.

The 35-W, triple-diffused, silicon transistor was also used in a study to compare the electrically measured thermal resistance with measurements by an infrared microradiometer. The electrical measurements were made at V_{CE} values of 10 and 20 V with a collector current of 500 mA. Infrared measurements of the chip surface temperature made at a V_{CE} of 20 V indicated that a significant temperature gradient existed with a low temperature around the edges of the chip, a relatively constant intermediate temperature over the greater portion of the interior of the chip, and a higher temperature on the chip in the vicinity of the emitter lead. The computed thermal resistance was 1.88°C/W in the interior area of the chip and 2.68°C/W in the hottest portion of the chip near the emitter

Table 3 — Comparison of Thermal Resistance Measured using Collector-and-Base Switching and Collector-Only Switching

t_{meas}	Collector-and-Base Switching	Collector-Only Switching
10μs	2.16°C/W	2.10°C/W
20	2.01	2.00
50	1.92	1.88
100	1.85	1.83

lead. Thermal resistance measured electrically 10 μs after the termination of the heating power pulse was 2.08°C/W, higher than the value measured with the infrared microradiometer in the major portion of the chip interior, but less than that in the elevated temperature region. It should be pointed out that these measurements were made under operating conditions well below those that cause severe current constriction or hot-spot formation. Further studies are needed to determine to what extent the electrically measured thermal resistance is increased by properly extrapolating V_{BE} to zero time delay after the termination of the heating power pulse. (S. Rubin and F. F. Oettinger)

Plans: Data collection for the Committee JC-25 preliminary thermal resistance round robin will be started. A common-base circuit for measuring R_{θ}, wherein both the collector and emitter of the transistor under test can be switched, will be designed and fabricated. Measurements of thermal resistance and d-c current gain of power transistors under various operating conditions will continue. Infrared microradiometer measurements will be made on the same devices to continue the investigation of relationships which may exist between the peak temperature and the electrically measured temperature.

5.2. THERMOGRAPHIC MEASUREMENTS

Objective: To evaluate the utility of thermographic techniques for detection of hot spots and measurement of temperature distribution in semiconductor devices.

Progress: Although the work on the characterization of the phosphors is far from complete, it is possible to estimate some of the capabilities of the phosphors and equipment. By illuminating the specimen with minature ultraviolet lamps and detecting the radiation with the photometric microscope, temperature sensitivities (change in brightness) between 5 and 7 %/°C have been obtained on the four phosphors that cover the range between room temperature and 400°C. In two of the phosphors this is less than the sensitivity specified by the phosphor manufacturer, but it is still sufficient to permit measurement of temperature changes of 2°C. The low sensitivity may be due to the thinness of the phosphor coating, approximately one particle thick, obtained by the flotation technique (NBS Tech. Note 488, p. 31).

Spatial resolution is limited by the capabilities of the photometric microscope and 50-μm fiber optic probe to about 35 μm. For comparison, the spatial resolution was estimated to be 20 μm when the light output of the phosphor was recorded photographically [1], and the spatial resolution using the infrared microscope at a comparable working distance was found to be 35-μm. Since maximum phosphor particle size is about 25-μm, particle size does not limit the spatial resolution at present.
(G. J. Rogers)

Plans: Fabrication of a new heat sink with better temperature control for testing silicon wafers will begin. Test specimens with surfaces of silicon dioxide, polished silicon, aluminum, chromium, and gold will be fabricated. These surfaces will be coated with phosphor by the slurry-settling technique and characterized.

5.3. MICROWAVE DEVICE MEASUREMENTS

Objective: To study the problems and uncertainties associated with the measurements of microwave devices, and to improve the techniques of these measurements.

Progress: The r-f portion of the X-band mixer measurement system was extensively rebuilt to permit active stabilization (leveling) of the local oscillator power and to improve certain other aspects of the circuit. The new circuit, shown schematically in figure 14, incorporates the following changes from the previously reported circuit (NBS Tech. Note 560, p. 43):

1. A heat sink has been added next to the klystron. This is a 10-in. (25-cm) long waveguide section, machined from a 4-in. (10-cm) diameter copper bar. The bar was sliced in half lengthwise, the waveguide opening and cooling fins milled, and the halves silver-soldered together using plugs to maintain the correct waveguide width. This design avoids the thermal resistance that would have been added by the use of separately attached waveguide flanges or cooling fins. With heat-conducting silicone grease between the klystron and the heat sink and with the blower off, the temperature rise of the klystron at a point opposite the waveguide flange was only about half as much as it was before the heat sink was installed. The heat sink is expected to improve the life and stability of the klystron without the complexity of water cooling. It also adds considerable mechanical stability to the klystron and adjacent components.

2. A 20 dB directional coupler was substituted for the 10 dB coupler, previously used to extract power to frequency stabilize (phase-lock) the klystron in order to reduce the power loss. To obtain sufficient phase-lock power, the hybrid tee previously used for initial klystron adjustments was replaced by another 20 dB directional coupler. A diode detector is now used for these adjustments in place of the bolometer because of the much lower power level.

3. The PIN modulator is now permanently in the local-oscillator (l-o) line. This eliminates loss and leakage of the coaxial cable previously used to connect the modulator. Leveling is achieved by a negative-feedback loop using a 10 dB directional coupler and a diode detector to sample the l-o power. The detector output is fed to an

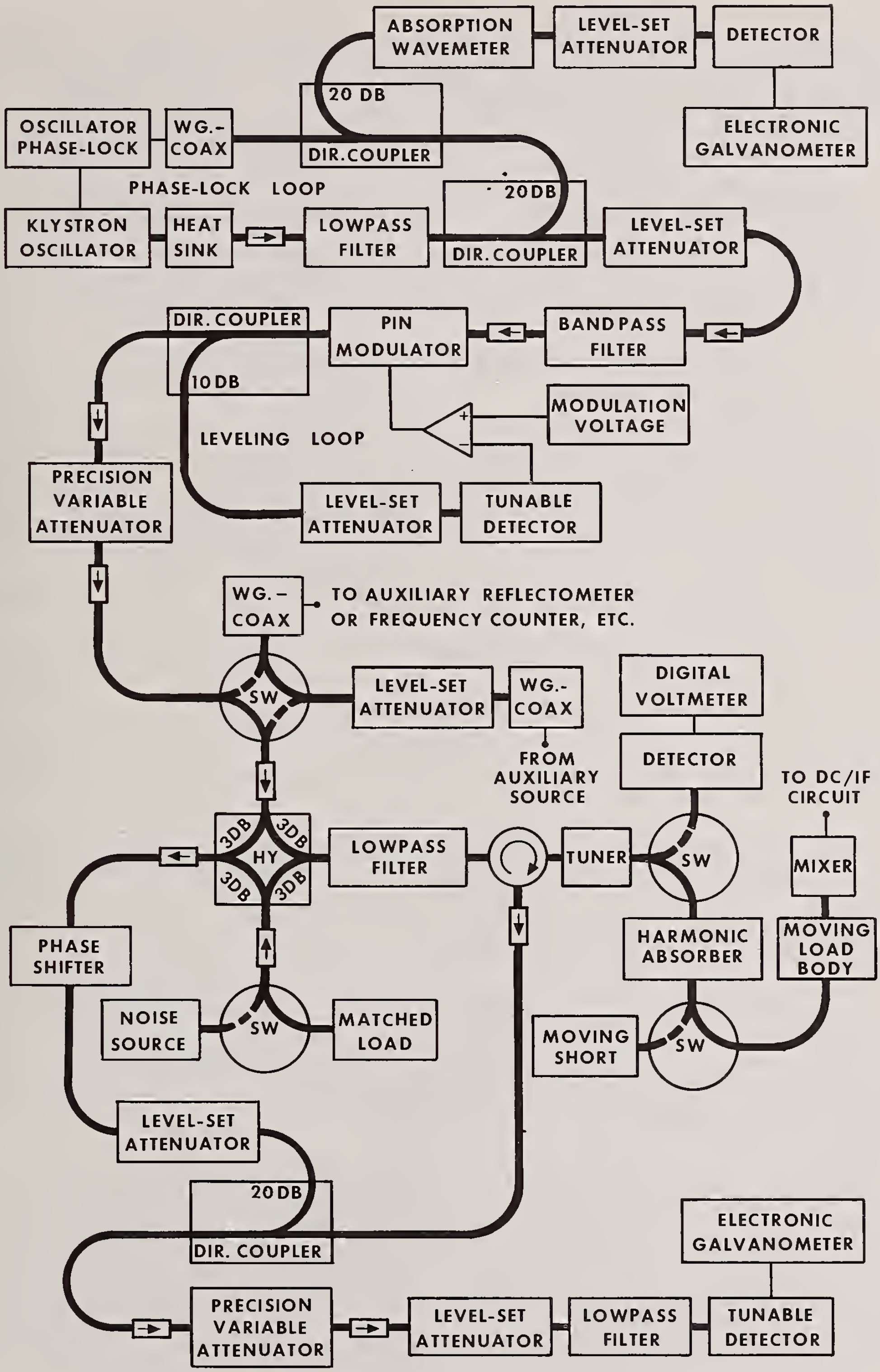

Figure 14. Modified r-f section of microwave mixer measurement system.

operational amplifier which provides a large open-loop gain for precise leveling and whose output is capable of directly driving the PIN modulator. A stable r-f attenuator controlling the sampled power is used in setting the l-o power available to the mixer. This attenuator setting and the tuning adjustment on the detector can be locked. Stable modulation of the l-o power, used to produce a test signal, is easily achieved by applying the modulation voltage to the otherwise unused input terminal of the operational amplifier. The peak-to-trough ratio of this modulation, from which the equivalent single-frequency signal is calculated, is measured by using a precision variable attenuator following the leveling loop in a substitution method (NBS Tech. Note 571, p. 44).

4. An auxiliary l-o source for braodband tuning with a 30 MHz i-f can be introduced through the transfer switch that is normally set so that it powers the auxiliary reflectometer that is used in obtaining a line-matched source for the mixer, in setting and checking the l-o frequency on a counter, and for experimental purposes. The level-set attenuator at the auxiliary source port is primarily intended to reduce the reflection from this port of power leading through the isolator and circulator when the auxiliary reflectometer is is use. This minimizes the change in mixer-source match as the transfer switch is thrown.

5. The low-pass filter used to remove out-of-band power from the noise source has been moved into the l-o line where it will also remove harmonics that may possibly be generated in the PIN modulator. The new location also affords better mechanical stability by shortening the noise-source arm, and it properly spaces the circulator from the hybrid tee isolator so that these magnetic components do not interact.

6. The waveguide loop used for the reflectometer was rearranged completely to make it more compact and rigid, with shorter paths and fewer bends. The 10 dB directional coupler formerly used in the reflectometer nulling circuit was replaced by a 20 dB coupler.

7. The hybrid tee formerly used to separate reflected from incident l-o power, for the reflectometer, has been replaced by a circulator, thereby saving 2 to 2 1/2 dB of loss each for both the incident power and the reflected power. This change reduces the l-o power requirement and increases the reflectometer senstivity.

8. Power monitoring, that had previously utilized the half of the l-o power lost in the hybrid tee now replaced by the circulator, is now accomplished with a switch following the circulator and the tuner. This switch allows the relative amplitude of the l-o power to be ascertained by measuring the output voltage of either a diode or a film thermocouple with a high-precision digital voltmeter. The relative stability of these two types of detectors has yet to be determined.

9. Since it may be desirable, for reproducible measurements, to avoid reflecting harmonics of the l-o frequency generated by the mixer diode back to the mixer, a well-matched pad has been constructed to absorb a moderate amount of power at all frequencies (including, unfortunately, the l-o and signal frequencies). This pad consists of an 11-in. (28-cm) long section of waveguide containing a dielectric element with 5-in. (13-cm) long tapers on both ends. At the center, the dielectric completely fills the waveguide, thereby providing attenuation for all modes of propagation. Two types of pads were made: one with a nylon element which exhibited a loss of only 2 dB, the other with an unfired steatite element with a loss of 7 dB. The higher loss can be tolerated for the low i-f measurements, but may be too high for the 30 MHz i-f measurements using the gas-discharge noise source.

10. The guillotine-type waveguide gate formerly used to reflect the incident power has been replaced with a transfer switch and a movable non-contacting, choke-type short. The switch is more reproducible in its action than the gate, and the movable short is less lossy and thereby provides a more nearly perfect reflection. (J. M. Kenney)

Plans: Methods for improving the precision of measurement of mixer output voltage will be sought by reducing non-random interference, modulation drift, and power drift.

5.4. CARRIER TRANSPORT IN JUNCTION DEVICES

Objective: To improve methods of measurement for charge carrrier transport and related properties of junction semiconductor devices.

Progress: Most of the laboratory effort this quarter was concentrated on the Sandia-type delay-time bridge [1]. Prior to the insertion of the transistor bias networks within the attenuator and phase-splitter box and prior to connecting a transistor socket at the bridge-summing (null) point, interchannel (emitter and collector delay line) isolation, constancy of delay-time zero setting with frequency, and incremental delay ratio were measured for signal frequencies in the operating range of the bridge between 3 and 30 MHz. The interchannel isolation was found to be greater than 50 dB at all frequencies. This means that the maximum amplitude error introduced into the test channel from the reference channel will be less than 0.5 percent. The variation in the "zero set" delay-time reference value was found to be less than 2.5×10^{-10} s, which is felt to be an acceptably small value: large values of shift in the reference value of delay time could be a nuisance if one wished to make rapid measurements at a variety of frequencies. Incremental delay ratio measurements were made by measuring the delay required to be added to the test channel, for null, when known time delays were inserted in the reference channel. Ideally the ratio of these two should be unity, independent of frequency. The desired 1:1 incremental delay time ratio was approached closely for frequencies between 3 and 30 MHz, and for delay time increments between a few tens of picoseconds and 10 ms. A log-log plot showed a straight-line behavior over this delay-time range with deviations of only a few picoseconds at the lower end.

Following these measurements, bias networks were inserted into the emitter and collector (ground) returns within the attenuator and phase-splitter box, and a TO-18 transistor socket and collector-voltage-monitoring circuit and jack were connected to the radio receiver end of the emitter and collector delay lines. Preliminary delay-time measurements were made as a function of emitter current on a silicon *n-p-n* transistor with f_α of about 350 MHz as a functional check of the operation of the bridge. (D. E. Sawyer and G. J. Rogers)

The delay-time error that might be introduced by moving the primary loop in the attenuator and phase-splitter box relative to the two secondary loops was analyzed theoretically. Such relative motion could be the result of rebalancing the bridge, during a measurement sequence, to accomodate a change in the common-base current gain due to changes in biasing conditions such as a change in emitter current. (C. Hoer*)

A survey of noise in semiconductor devices was completed this quarter [2]. One of the conclusions reached is that with noise-spectrum measurements one should be able to measure separately device phenomena which otherwise are almost always measured by their sum effect. One example is the prediction that one might be able to separate the true base delay time in a transistor from the delay time associated with the carriers interacting with the collector junction electric field following their passage across the base.

Additional references have been compiled on the utilization of various probing techniques for the determination of transistor device parameters while the devices are in the wafer stage of preparation.
(D. E. Sawyer)

Plans: The equivalent circuit of a transistor in the Sandia-type bridge will be analyzed to relate the measured delay time to the various times within the device, such as the emitter RC charging time and the base-collector delay time. A second delay-time measurement circuit will be constructed after the arrival of the vector voltmeter. A third system, based on the measurement of S-parameters will be assembled following the ordering and arrival of necessary additional parts. Intensive device measurements will be begun following the construction of the system based on the vector voltmeter, since two different systems will then be available for intercomparison measurements. The analysis of delay-time error will be evaluated within the context of the measurement procedure.

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*NBS Electromagnetics Division.

5.5. SILICON NUCLEAR RADIATION DETECTORS

Objective: To conduct a program of research, development, and device evaluation in the field of silicon nuclear radiation detectors with emphasis on the improvement of detector technology, and to provide consultation and specialized device fabrication services to the sponsor.

Progress: Pre-flight bench-testing of lithium-drifted silicon radiation detectors continued. A test chamber for carrying out measurements of the effects of ambients that may affect the performance characteristics of semiconductor detectors was constructed and evaluated. Radiation damage measurements on lithium-drifted silicon detectors using 1.5-MeV electrons were begun.

Testing and Evaluation — Commercial lithium-drifted silicon detectors were given extensive bench-tests as part of a continuing program of detector evaluation and acceptance for the sponsor. These measurements, for the IMP-H and Pioneer-F programs, were performed at the NASA-GSFC site. (A. J. Baroody)

Radiation Damage — A commercial, 2-mm thick, lithium-drifted silicon detector was irradiated with 1.5-MeV electrons at fluences between 10^{12} and 10^{14} cm^{-2} on a 20-mm^2 spot on the particle-entrance window (p-contact). The irradiation was carried out at room temperature with the detector reverse biased at 400 V. The detector reverse leakage current, noise, and capacitance were observed to increase with increasing fluence with a sharp increase in noise above a fluence of approximately 3×10^{13} cm^{-2}. After the irradiation at 10^{14} electrons per square centimeter, the current and noise had increased by more than an order of magnitude over the initial values, and the charge collection efficiency for charged particles incident on the irradiated contact was much degraded.

The effect of room temperature annealing on the detector characteristics was monitored continuously with the detector kept under 400-V reverse bias. After approximately four days, the current and noise had almost completely returned to their pre-irradiation levels, while the capacitance and charge collection efficiency exhibited only a small percentage recovery. The collection efficiency and energy resolution remained relatively unchanged from pre-irradiation levels, however, for particles incident on the n-contact following irradiation and annealing. This observation might be explained by the fact that radiation-induced defect centers act as traps only for electrons. However, it is also possible that the irradiation produced a dead layer near the i-p junction due to precipitation of lithium on the radiation-induced defects. The characteristics of the irradiated detector continued to deteriorate when the applied bias was removed.

A second detector was irradiated under the same conditions as above, except that the detector was under a reverse bias of 200 V. The increase of current and noise with increasing fluence was alower than in the 400-V case, whereas the increase of capacitance and reduction in charge collection efficiency was accelerated. (Y. M. Liu)

Ambient Exposure Tests — The construction of the new all-metal vacuum system was completed. Preliminary tests were completed to check out the performance of the system. The ultimate pressure attained was approximately 3×10^{-5} Torr, and the system was found capable of returning to its ultimate pressure after exposure to dry ammonia gas at pressure up to 1 Torr.

Electronic equipment in accordance with standard procedures [1] was assembled and tested for the measurement of noise of detectors exposed to ambients such as ammonia, methanol, and a commercial leak-detection fluid. (E. I. Klein, W. K. Croll, and B. H. Audet)

Plans: Pre-flight bench-testing of detectors will continue as required by the sponsor. The radiation damage study of lithium-drifted silicon detectors will continue with irradiation with 600-keV electrons and fast neutrons. The pressure of gas at which measurable effects on detector noise and leakage current are observed will be determined as well as the duration of such effects once the gas is removed from the system.

5.6. REFERENCES

5.2. Thermographic Measurements

1. Brenner, D. J., A Technique for Measuring the Surface Temperature of Transistors by Means of Fluorescent Phosphor, NBS Tech. Note 591 (July, 1971).

5.4. Carrier Transport in Junction Devices

1. Sullivan, W. H., A New Technique for the Direct Measurement of Minority Carrier Base Transit Time in the Junction Transistor, Sandia Laboratories Report SC-DR-69-419, September, 1969. Available from the National Technical Information Service, Springfield, Va. 22151

2. Sawyer, D. E., Noise in Semiconductor Devices, Its Determination and Significance, NBS Electron Devices Section Report 921, January, 1971. This unpublished internal report, written under the guidance of Prof. H. P. D. Lanyon of the Worcester Polytechnic Institute, is intended to provide preliminary information for discussion purposes.

5.5. Silicon Nuclear Radiation Detectors

1. USA Standard and IEEE Test Procedure for Semiconductor Radiation Detectors (for Ionizing Radiation), USAS N42.1/IEEE No. 300/(1969), pp. 5-6.

Appendix A

JOINT PROGRAM STAFF

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Consultant: C. P. Marsden

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× Dr. J. A. Coleman is on temporary assignment to Office of Associate Director for Programs.

Appendix B

COMMITTEE ACTIVITIES

ASTM Committee F-1 on Electronics

- A. J. Baroody, Lifetime Section
- C. F. Bolton, Committee Assistant Secretary
- W. M. Bullis, Editor, Subcommittee 4, Semiconductor Crystals, and Subcommittee 7, Hybrid Microelectronics; Leaks, Resistivity, Mobility, Dielectrics, and Compound Semiconductors Sections
- J. A. Coleman, Secretary, Subcommittee 5, Semiconductor Processing Materials
- J. R. Ehrstein, Resistivity, Epitaxial Resistivity, and Epitaxial Thickness Sections
- J. C. French, Committee Editor
- T. F. Leedy, Photoresist Section
- C. P. Marsden, Committee Secretary
- R. L. Mattis, Lifetime Section
- W. E. Phillips, Chairman, Lifetime Section; Secretary, Subcommittee 4, Semiconductor Crystals; Crystal Perfection, Encapsulation, Thin Films, and Thick Films Sections.
- A. H. Sher, Germanium Section
- W. R. Thurber, Mobility, Germanium, Compound Semiconductors, and Impurities in Semiconductors Sections

ASTM Committee E-10 on Radioisotopes and Radiation Effects

- W. M. Bullis, Subcommittee 7, Radiation Effects on Electronic Materials
- J. C. French, Subcommittee 7, Radiation Effects on Electronic Materials

Electronic Industries Association, Solid State Products Division, Joint Electron Device Engineering Council (JEDEC):

- J. M. Kenney, Microwave Diode Measurements, Committee JC-21 on UHF and Microwave Diodes
- F. F. Oettinger, Chairman, Task Group JC-11.3-1 on Thermal Considerations for Microelectronic Devices, Committee JC-11 on Mechanical Standardization; Technical Advisor, Thermal Resistance Measurements, Committees JC-22 on Thyristors, JC-20 on Signal Diodes, JC-25 on Power Transistors, and JC-30 on Hybrid Integrated Circuits
- S. Rubin, Chairman, Council Task Group on Galvanomagnetic Devices
- H. A. Schafft, Technical Advisor, Second Breakdown and Related Specifications, Committee JC-25 on Power Transistors

IEEE Electron Devices Group:

- J. C. French, Standards Committee
- J. M. Kenney, Chairman, Standards Committee Task Force on Micorwave Solid State Devices II (Mixer and Video Detector Diodes)

H. A. Schafft, Chairman, Standards Committee Task Force on Second Breakdown Measurement Standards

IEEE Nuclear Science Group:
J. A. Coleman, Administrative Committee, Nuclear Instruments and Detectors Committee; Editorial Board, *Transactions on Nuclear Science*

IEEE Magnetics Group
S. Rubin, Chairman, Galvanomagnetic Standards Subcommittee

IEEE Parts, Hybrids, and Packaging Group
W. M. Bullis, New Technology Subcommittee, Technical Committee on Hybrid Microelectronics

Society of Automotive Engineers
J. C. French, Subcommittee A-2N on Radiation Hardness and Nuclear Survivability

IEC TC47, Semiconductor Devices and Integrated Circuits:
S. Rubin, Technical Expert, Galvanomagnetic Devices; U. S. Specialist for Working Group 5 on Hall Devices and Magnetoresistive Devices

NMAB ad hoc Committee on Materials and Processes for Electron Devices:
W. M. Bullis

NMAB ad hoc Committee on Materials for Radiation Detection Devices:
D. E. Sawyer

Appendix C

SOLID-STATE TECHNOLOGY & FABRICATION SERVICES

Technical services in areas of competence are provided to other NBS activities and other government agencies as they are requested. Usually these are short-term, specialized services that cannot be obtained through normal commercial channels. Such services provided during the last quarter are listed below and indicate the kinds of technology available to the program.

1. Carrier Lifetime Measurements (W. E. Phillips)
   Carrier lifetime was measured in three silicon solar cell blanks by the surface photovoltage method for NASA Lewis Research Center.

2. Infrared Absorption Measurements (W. R. Thurber)
   Thirteen silicon solar cell blanks were examined for oxygen content by the infrared absorption method for NASA Lewis Research Center to discriminate between Czochralski-grown and float-zoned crystals.

3. Thin-Film Deposition (T. F. Leedy)
   Reticles were fabricated for the Microwave and Mechanical Instrumentation Section using vacuum evaporation of chromium onto glass plates.

4. Radiation Detectors (B. H. Audet)
   Four silicon nuclear radiation detectors were reprocessed for the Photonuclear Physics Section.

5. Sectioning and Plating (H. A. Briscoe)
   Transistors were sectioned, polished, and stained to reveal cross-sectional geometries, small piece parts were gold or nickel plated, and silver films were vacuum evaporated on several types of plastics for other groups in the Electronic Technology Division.

6. Thermographic Studies of Semiconductor Devices (F. F. Oettinger)
   Thermal studies were performed on transistors and integrated circuits using an infrared microradiometer for the Naval Ammunition Depot, Crane, Indiana.

Appendix D

JOINT PROGRAM PUBLICATIONS

Prior Reports:

A review of the early work leading to this Program is given in Bullis, W. M., Measurement Methods for the Semiconductor Device Industry—A Review of NBS Activity, NBS Tech. Note 511, December, 1969.

Quarterly reports covering the period since July 1, 1968, have been issued under the title Methods of Measurement for Semiconductor Materials, Process Control, and Devices. These reports may be obtained from the Superintendent of Documents (Catalog Number C.13.46:XXX) where XXX is the appropriate technical note number. Microfiche copies are available from the National Technical Information Service (NTIS), Springfield, Va. 22151.

Quarter Ending	NBS Tech. Note	Date Issued	NTIS Accession No.
September 30, 1968	472	December, 1968	AD 681330
December 31, 1968	475	February, 1969	AD 683808
March 31, 1969	488	July, 1969	AD 692232
June 30, 1969	495	September, 1969	AD 695820
September 30, 1969	520	March, 1970	AD 702833
December 31, 1969	527	May, 1970	AD 710906
March 31, 1970	555	September, 1970	AD 718534
June 30, 1970	560	November, 1970	AD 719976
September 30, 1970	571	April, 1971	AD 723671
December 31, 1970	592	August, 1971	

Current Publications:

Sher, A. H., Carrier Trapping in Ge(Li) Detectors, *IEEE Trans. Nucl. Sci.* NS-18, No. 1, 175-183 (February, 1971).

Liu, Y. M., and Coleman, J. A., Electron Radiation Damage Effects in Silicon Surface-Barrier Detectors, *IEEE Trans. Nucl. Sci.* NS-18, No. 1, 192-199 (February, 1971).

Rubin, S., and Oettinger, F. F., Thermal Hysteresis and its Possible Effect on Restriction of the Hot-Spot Free Operating Range of Some Power Transistors, *IEEE Trans. Electron Devices* ED-18, 393-394 (June, 1971).

Thurber, W. R., Determination of Deep Impurities in Silicon and Germanium by Infrared Photoconductivity, NBS Tech. Note 570 (March, 1971).

Harman, G. G., and Kessler, H. K., Application of Capacitor Microphones and Magnetic Pickups to the Tuning and Trouble Shooting of Microelectronic Ultrasonic Bonding Equipment, NBS Tech. Note 573 (April, 1971).

APPENDIX D

Sher, A. H., and Thurber, W. R., Minority Carrier and Lithium-Ion Drift Mobilities and Oxygen Concentration in p-Type Germanium, submitted to *J. Appl. Phys.*

Sher, A. H., Croll, W. K., and Thurber, W. R., Determination of Oxygen in Germanium Below 20 ppb by Measurements of Lithium Mobility and Precipitation, submitted to *Anal. Chem.*

Blackburn, D. L., Nondestructive Photovoltaic Resistivity Gradient Measurements of Circular Semiconductor Wafers, Recent News Paper, Electrochemical Society Meeting, Washington, D. C. (May, 1971).

FORM NBS-114A (1-71)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET	1. PUBLICATION OR REPORT NO. NBS-TN-598	2. Gov't Accession No.	3. Recipient's Accession No.

4. TITLE AND SUBTITLE Methods of Measurement for Semiconductor Materials, Process Control, and Devices. Quarterly Report, January 1 to March 31, 1971.	5. Publication Date October 1971
	6. Performing Organization Code
7. AUTHOR(S) W. Murray Bullis, Editor	8. Performing Organization
9. PERFORMING ORGANIZATION NAME AND ADDRESS NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234	10. Project/Task/Work Unit No. See Item 15
	11. Contract/Grant No. See Item 12
12. Sponsoring Organization Name and Address NBS, DASA (EA071-801), Navy SSPO, NAD (Crane) (PO-1-0030, PO-1-0041, PO-1-0067) and NAFI (WR-1-1038), NESC (PO-1-1057), AFWL (F29601-71-F-0002), AEC, and NASA (S-70003-G and H-76553A).	13. Type of Report & Period Covered Interim Jan. 1 to March 31, 1971
	14. Sponsoring Agency Code

15. SUPPLEMENTARY NOTES 4251120, 4251123, 4251126, 4252114, 4252119, 4252128, 4254111, 4254112, 4254115, 4259522, 4259533, 4254432, 4252534, 4252535, 4259425, 4254429, 4251449.

16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

This quarterly progress report, eleventh of a series, describes NBS activities directed toward the development of methods of measurement for semiconductor materials, process control, and devices. Significant accomplishments during this reporting period include application of tuning and other related procedures for ultrasonic wire bonders in an industrial environment with a reported reduction of rejection rate at visual inspection by more than a factor of two, more complete evaluation of the photovoltaic method for determining radial resistivity profiles of circular semiconductor wafers, and identification of test conditions in which a d-c calibration curve may properly be used in measurements of thermal resistance. Work is continuing on measurement of resistivity, carrier lifetime, and electrical inhomogeneities in semiconductor crystals; specification of germanium for gamma-ray detectors; evaluation of wire bonds and die attachment; measurement of thermal properties of semiconductor devices, transit-time and related carrier transport properties in junction devices, and electrical properties of microwave devices; and characterization of silicon nuclear radiation detectors. Supplementary data concerning staff, standards committee activities, technical services, and publications are included as appendixes.

Key Words (cont.): microelectronics; microwave devices; nuclear radiation detectors; probe techniques (a-c); resistivity; semiconductor devices; semiconductor materials; semiconductor process control; silicon; thermal resistance; thermographic measurements; ultrasonic bonder; wire bonds.

17. KEY WORDS (Alphabetical order, separated by semicolons) Alpha-particle detectors; aluminum wire; base transit time; carrier lifetime; die attachment; electrical properties; epitaxial silicon; gamma-ray detectors; germanium; gold-doped silicon; methods of measurement;

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