NBS TECHNICAL NOTE 495

National Bureau of Standards Library, E-01 Admin. B dg OCT 6 1969

A UNITED STATES DEPARTMENT OF COMMERCE PUBLICATION

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

Quarterly Report April 1 to June 30, 1969

U.S. DEPARTMENT OF COMMERCE MATIONAL BUREAU OF STANDARD

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards ' was established by an act of Congress March 3, 1901. Today, in addition to serving as the Nation's central measurement laboratory, the Bureau is a principal focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. To this end the Bureau conducts research and provides central national services in four broad program areas. These are: (1) basic measurements and standards, (2) materials measurements and standards, (3) technological measurements and standards, and (4) transfer of technology.

The Bureau comprises the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, the Center for Radiation Research, the Center for Computer Sciences and Technology, and the Office for Information Programs.

THE INSTITUTE FOR BASIC STANDARDS provides the central basis within the United States of a complete and consistent system of physical measurement; coordinates that system with measurement systems of other nations; and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. The Institute consists of an Office of Measurement Services and the following technical divisions:

Applied Mathematics-Electricity-Metrology-Mechanics-Heat-Atomic and Molecular Physics-Radio Physics 2-Radio Engineering 2-Time and Frequency 2-Astrophysics ²—Cryogenics.²

THE INSTITUTE FOR MATERIALS RESEARCH conducts materials research leading to improved methods of measurement standards, and data on the properties of well-characterized materials needed by industry, commerce, educational institutions, and Government; develops, produces, and distributes standard reference materials; relates the physical and chemical properties of materials to their behavior and their Interaction with their environments; and provides advisory and research services to other Government agencies. The Institute consists of an Office of Standard Reference Materials and the following divisions:

Analytical Chemistry—Polymers—Metallurgy—Inorganic Materials—Physical Chemistry. THE INSTITUTE FOR APPLIED TECHNOLOGY provides technical services to promote the use of available technology and to facilitate technological innovation in industry and Government; cooperates with public and private organizations in the development of technological standards, and test methodologies; and provides advisory and research services for Federal state, and local government agencies. The Institute consists of the following technical divisions and offices:

Engineering Standards-Weights and Measures - Invention and Innovation - Vehicle Systems Research—Product Evaluation—Building Research—Instrument Shops—Measurement Engineering-Electronic Technology-Technical Analysis.

THE CENTER FOR RADIATION RESEARCH engages in research, measurement, and application of radiation to the solution of Bureau mission problems and the problems of other agencies and institutions. The Center consists of the following divisions:

Reactor Radiation-Linac Radiation-Nuclear Radiation-Applied Radiation.

THE CENTER FOR COMPUTER SCIENCES AND TECHNOLOGY conducts research and provides technical services designed to aid Government agencies in the selection, acquisition, and effective use of automatic data processing equipment; and serves as the principal focus for the development of Federal standards for automatic data processing equipment, techniques, and computer languages. The Center consists of the following offices and divisions:

Information Processing Standards-Computer Information - Computer Services - Systems Development-Information Processing Technology.

THE OFFICE FOR INFORMATION PROGRAMS promotes optimum dissemination and accessibility of scientific information generated within NBS and other agencies of the Federal government; promotes the development of the National Standard Reference Data System and a system of information analysis centers dealing with the broader aspects of the National Measurement System, and provides appropriate services to ensure that the NBS staff has optimum accessibility to the scientific information of the world. The Office consists of the following organizational units:

Office of Standard Reference Data-Clearinghouse for Federal Scientific and Technical Information 3-Office of Technical Information and Publications-Library-Office of Public Information-Office of International Relations.

¹ Headquarters and Laboratories at Galthershurg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20284. ² Located at Bouider, Colorado 80302.
³ Located at 5285 Port Royal Road, Springfield, Virginia 22151.

UNITED STATES DEPARTMENT OF COMMERCE Maurice H. Stans, Secretary NATIONAL BUREAU OF STANDARDS • Lewis M. Branscomb, Director



ISSUED SEPTEMBER 1969

Nat. Bur. Stand. (U.S.), Tech. Note 495, 45 pages (Sept. 1969) CODEN: NBTNA

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

Quarterly Report April 1 to June 30, 1969

Edited by W. Murray Bullis Electronic Technology Division Institute for Applied Technology National Bureau of Standards Washington, D.C. 20234

Jointly Supported by National Bureau of Standards Defense Atomic Support Agency U.S. Navy Strategic Systems Project Office and National Aeronautics and Space Administration

NBS Technical Notes are designed to supplement the Bureau's regular publications program. They provide a means for making available scientific data that are of transient or limited interest. Technical Notes may be listed or referred to in the open literature.

CONTENTS

		PAGE
1.	Introduction and Highlights	l
2.	Resistivity	6
3.	Carrier Lifetime	8
4.	Inhomogeneities	12
5.	Infrared Methods	14
6.	Hall Effect	15
7.	Deep-Level Studies	15
8.	Gold-Doped Silicon	16
9.	High Field Effects	16
10.	Specification of Germanium [†]	17
11.	Metallization Evaluation	21
12.	Processing Facility	22
13.	Wire Bond Evaluation	23
14.	Die Attachment Evaluation	31
15.	NASA Measurement Methods	31
16.	Second Breakdown	33
17.	Thermal Properties of Devices	33
18.	Thermographic Measurements	36
19.	Microwave Diode Measurements	36
20.	Silicon Nuclear Radiation Detectorst	37
Appendix A.	Joint Program Staff	40
Appendix B.	Committee Activities	41
Appendix C.	Solid-State Technology and Fabrication Services	42
Appendix D.	Joint Program Publications	43

† Related Projects

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

Quarterly Report April 1 to June 30, 1969

ABSTRACT

This quarterly progress report, fourth of a series, describes NBS activities directed toward the development of methods of measurement for semiconductor materials, process control, and devices. Principal emphasis is placed on measurement of resistivity, carrier lifetime, and electrical inhomogeneities in semiconductor crystals; evaluation of wire bonds; and measurement of thermal properties of semiconductor devices. Other tasks involve study of infrared measurement methods, deep-lying impurities in InSb, gold in silicon, and high field effects; establishment of a processing facility; evaluation of aluminum metallization and wafer die attachment: review of NASA measurement methods; and measurement of Hall effect in semiconductor crystals, second breakdown in transistors, and noise in microwave diodes. Related projects on silicon nuclear radiation detectors and specification of germanium are also described. Supplementary data concerning staff, committee activities, technical services, and publications are included as appendixes.

Key Words: carrier lifetime; die attachment; electrical properties; gamma detectors; germanium; gold-doped silicon; indium antimonide; metallization; methods of measurement; microelectronics; nuclear radiation detectors; resistivity; semiconductor devices; semiconductor materials; semiconductor process control; silicon; thermal resistance; thermographic measurements; wire bonds.

1. INTRODUCTION AND HIGHLIGHTS

This is the fourth quarterly report to the sponsors of the Joint Program on Methods of Measurement for Semiconductor Materials, Process Control, and Devices. The report is subdivided according to tasks which have been identified as parts of the Program. Sections 2 through 10 deal with methods of measurement for materials; sections 11 through 15, with

1. INTRODUCTION AND HIGHLIGHTS

methods of measurement for process control; and sections 16 through 20, with methods of measurement for devices.

Activity is under way on several different methods for measuring resistivity. Following the decision last quarter to redirect the emphasis from studies of the voltage-breakdown method for measuring resistivity of epitaxial layers to studies of the spreading resistance and capacitance-voltage techniques for this measurement, effort on this task has been expanded with the addition of a new staff member. Work on the traditional four-probe method for measuring resistivity is continuing. A round robin being conducted with ASTM Committee F-1 for the purpose of extending the range of applicability of the standard method to higher and lower resistivity values is nearly complete, and the Committee has expressed an interest in further studies to extend the range up to the 10,000 Ω -cm material which is suitable for fabrication of silicon nuclear radiation detectors. The detailed study of this method as applied to measurements on epitaxial and diffused layers is also continuing.

Carrier lifetime is highly sensitive to the presence of impurities which may not affect the resistivity of a semiconductor but which can adversely affect the performance of transistors and other minority carrier devices. Methods for comparing lifetime in bulk crystals and in device structures are essential to an understanding of the influence of both naturally-occurring and neutron-induced recombination and trapping centers in silicon. Several different methods for measuring carrier lifetime in semiconductors are being studied. A revised procedure for making lifetime measurements by the photoconductive decay method is being prepared for the consideration of ASTM Committee F-1. Significant improvements have been made in the hardware associated with the steadystate photomagnetoelectric-photoconductivity equipment. The scope of this task is being extended to include consideration of lifetime measurements in device structures, such as transistor base regions, as well as on bulk crystals.

The work on the deep-lying defect in indium antimonide is nearing completion. The defect has been identified as a lattice vacancy which is thought to occur in the indium sub-lattice. This conclusion is based on the results of resistivity, Hall coefficient, and carrier lifetime measurements on high-resistivity indium antimonide specimens before and after the introduction of lithium. Although this study, which has been conducted as a doctoral thesis topic, is not applicable to characterization of silicon, it does provide new insight into the properties of III-V compounds.

Poor adhesion of metal films is a significant cause of device failures. The first efforts on the metallization evaluation task are being

1. INTRODUCTION AND HIGHLIGHTS

directed toward the development of more reliable test methods for determining film adhesion. The scratch test for determining the adhesion characteristics of aluminum metallization films deposited on silicon and silicon oxide has been selected for detailed study, and apparatus for performing this test has been constructed. At the same time, the literature on thin-film adhesion testing is being methodically searched.

The small fraction of wire bonds which fail constitutes a serious problem in semiconductor device reliability. A significant portion of the Joint Program is directed toward the evaluation and development of methods for testing wire bonds to identify bonds which may fail. A critical review of this field is well under way; field visits to 27 different organizations have been completed, and most of the relevant literature in the field has been obtained. The review is expected to focus on the advantages and limitations of the various practices now employed in the industry and to pinpoint areas where additional information must be obtained. At the same time experimental efforts have been initiated in several areas.

- 1. An improved method for grasping the wire during a pull test was developed last quarter. Since the wire is grasped without deformation and the bond may be pulled in any direction, this convenient method, which involves the use of a quicksetting hot-melt glue, permits increased control of the test conditions. It is also possible to test single bonds.
- 2. Preliminary studies of ultrasonic bonding with aluminum ribbon wire have demonstrated that lower bonding pressures than those required for round wire of the same cross section will produce satisfactory bonds. This suggests that harder and stronger ribbon wire may be used to make bonds without the risk of damage to the semiconductor die. The use of the harder wire is expected to result in stronger bonds. As an added advantage it turns out to be easier to use the ribbon wire than round wire for making ultrasonic bonds.
- 3. Initial tests of a number of ultrasonic bond monitoring techniques have led to the selection of a capacitor microphone technique for future studies. An extensión tip damped with silicone rubber and polytetrafluoroethylene was developed to improve the resolution of the capacitor microphone system.
- 4. Resonance characteristics of a nickel magnetostrictive transducer, tapered horn, and bonding tool were measured under both constant current and constant voltage conditions. These results suggest that a system with a constant current source is less sensitive to variations in the source frequency than is one with a constant voltage source. It was also

found that the maximum tip vibration amplitude occurred at a different frequency from the transducer resonance as indicated by a current meter in series with the transducer coil.

5. It was also established that resonance modes of bonding tools can be observed with the high-resolution capacitor microphone. Thus detailed studies of bonding tool resonance including torsional and other modes of vibration can be undertaken.

An abrupt decrease in d-c current gain in interdigitated mediumpower transistors was observed to occur at the same operating condition as that for which the thermal resistance as measured by the forwardbiased base-emitter voltage abruptly increases. The observation by means of thermographic phosphors of hot-spot formation at the same operating condition suggests that the relatively simple measurement of d-c current gain may be a practical method for identifying the onset of hot-spot formation.

Organizational plans were completed for the Symposium on Silicon Device Processing to be held in June, 1970 under the joint sponsorship of ASTM Committee F-1 and NBS. The Symposium is intended to bring together engineers and applied scientists actively engaged in the silicon device technology field so that they may discuss the most advanced measurement methods for process control and materials characterization in an unhurried and informal atmosphere. The basic theme of the meeting is to stress the interdependence of measurements, techniques, facilities, and materials as they relate to the overall problems of inproving and advancing silicon device sciences and technologies. The opening session will consist of invited papers on silicon crystal growth, surface preparation, diffusion, epitaxy, and silicon oxides and nitrides. Subsequent sessions will deal with the unit processing operations, epitaxy and diffusion, from the standpoint of the required techniques and facilities as well as the properties and characterization of their product. Surface preparation and interdependence of the unit processing operations will also be discussed.

The Joint Program was undertaken last year 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, and which can provide a common basis for the purchase specifications of government agencies. In addition, such methods will provide a basis for controlled improvements in essential device characteristics,

1. INTRODUCTION AND HIGHLIGHTS

such as uniformity of response to radiation effects. The Program is supported by the National Bureau of Standards [1], the National Aeronautics and Space Administration [2], the Defense Atomic Support Agency [3], and the U. S. Navy Strategic Systems Project Office [4]. Because of the cooperative nature of the Program, 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 taken into consideration in planning.

Additional background information on the Program and individual tasks may be found in earlier reports in this series (see Appendix D). Besides the tasks sponsored under the Joint Program, this report contains descriptions of activity on related projects supported by NBS or other agencies. Although the specific objectives of these projects are different from those of the Joint Program, much of the activity undertaken in these projects will be of interest to Joint Program sponsors. The sponsor of each of these related projects is identified in the description of the project.

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

Through Order ER-11897, Electronics Research Center. (NBS Project 4259523)

^{3.} Through Inter-Agency Cost Reimbursement Order 808-69. (NBS Project 4259522)

^{4.} Administered by U. S. Naval Ammunition Depot, Crane, Indiana through Cost Reimbursement Order PO9-0016. (NBS Project 4259533)

METHODS OF MEASUREMENT FOR SEMICONDUCTOR MATERIALS

2. RESISTIVITY

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

<u>Progress</u>: A round robin based on the ASTM four-probe method for measuring resistivity of silicon wafers [1] is being conducted in cooperation with ASTM Committee F-1 on Materials for Electron Devices and Microelectronics to determine the precision which can be expected when measuring bulk silicon wafer resistivity down to 0.001 Ω -cm and up to 2000 Ω -cm. All data have been received from the eight participating laboratories, and all calculations have been checked. Tabulation of the results is nearly complete.

To assist in the extension of the method to epitaxial and diffused layers, studies of the dependence upon current, probe force, and surface condition of the resistivity of silicon wafers as measured by the fourprobe method are continuing. Material in the resistivity range 0.001 to 1000 Ω -cm is being studied at probe forces of 25, 50, 100, and 150 g and current values between 0.1 and 10 times the values recommended for its resistivity level in the ASTM procedure [1]. Three surface conditions, lapped (with 5 μ m alumina), mechanically polished, and chemically polished, are being used.

Measurements are also being made of the contact resistance between the probe points and the wafer surface as a function of current, probe force, and surface condition. The results of these measurements are expected to aid in designing appropriate analog circuits used to test the voltage measuring equipment in these four-probe measurements. Both resistivity and contact resistance measurements have been completed on wafers with lapped surfaces.

Calculations of the results of measurements made as part of a Committee F-l sponsored round robin based on a proposed two-probe method [2] for measuring the resistivity of cylindrical silicon crystals have been completed and sent to the coordinator of the round robin.

- 1. "Method of Test for Resistivity of Silicon Slices Using Four Pointed Probes" (ASTM Designation: F84-68T), 1968 Book of ASTM Standards, Part 8, November, 1968. This method covers the range 0.05 to 120 Ω -cm.
- For a general description of the two-probe method see "Method of Test for Resistivity of Semiconductor Materials" (ASTM Designation: F43-67T), 1968 Book of ASTM Standards, Part 8, November, 1968.

⁽F. H. Brewer)

2. RESISTIVITY

Commercially available components have been selected for use in the measurement of spreading resistance. Design plans are being developed for a probe holder assembly which incorporates features described in the literature [3]. Several materials are being considered for use as probe tips in the spreading resistance apparatus. These include tungsten carbide, stainless steel, osmium, tungsten-ruthenium alloy, and silicon carbide. A source of silicon carbide with controlled doping level has been found, and a procedure for laboratory use, but it may be prohibitively complex for large scale use. No further fabrication refinements are planned until it is determined whether silicon carbide is a satisfactory probe material.

The suitability of an available spectrophotometer for measuring epitaxial layer thickness and bulk resistivity by infrared techniques as suggested by ASTM Committee F-1 [4, 5] is being evaluated. (J. R. Ehrstein)

Instrumentation has been surveyed for the purpose of establishing a capability for measuring doping profiles in epitaxial films by the capacitance-voltage technique [6]. In addition to the knowledge of the diode fabrication procedures and the method of epitaxial layer growth, a good understanding of this measurement method requires study of the effects of the frequency used for the capacitance measurement. Since a large amount of data is required to profile a single epitaxial layer, it is intended to automate the data-taking process. However, instrumentation for capacitance measurement which is capable of being automated has only been found for use at 100 kHz and 1 MHz. Hence it may be necessary to concentrate work at these two frequencies and to sample effects at other frequencies with equipment which is not automated. (J. R. Ehrstein and G. N. Stenbakken)

- J. M. Adley, M. R. Poponiak, C. P. Schneider, P. A. Schumann, Jr., and A. H. Tong, "The Design of a Probe for the Measurement of the Spreading Resistance of Semiconductors," *Semiconductor Silicon*, R. Haberecht and E. Kern, Eds., The Electrochemical Society, New York, 1969, pp. 721-731.
- 4. "Method of Test for Thickness of Epitaxial Layers of Silicon on Substrates of the Same Conductivity Type by Infrared Reflectance" (ASTM Designation: F95-68T), 1968 Book of ASTM Standards, Part 8, November, 1968.
- 5. P. A. Schumann, and R. P. Phillips, "Comparison of Classical Approximations to Free Carrier Absorption in Semiconductors," *Solid-State Electronics* 10, 943-948 (1967).
- 6. G. W. Reuttinger, S. J. Regas, D. J. Sidor, and B. Schwartz, "An Automatic Test Set for Measuring Dopant Concentration Profiles in Epitaxial Films" *Solid-State Electronics* 12, 31-39 (1969).

2. RESISTIVITY

<u>Plans</u>: Results of the four-probe resistivity round robin will be fully tabulated and reported to ASTM Committee F-1. Several 10,000 Ω -cm wafers will be evaluated for uniformity prior to the initiation of a new round robin to extend the method to a still higher resistivity range.

The investigation of the dependence upon current and probe force of resistivity as measured by the four-probe method and of contact resistance between the probe point and the surface of the wafer will proceed. Measurements will be made for the same range of variables previously used, first on mechanically, and then on chemically, polished surfaces.

Preliminary measurements by the spreading resistance method of resistivity of epitaxial layers will be made. Special emphasis will be given to solution of problems related to the low signal levels which occur and the high electrical isolation required by the method. A more versatile spreading resistance probe assembly will be designed and fabrication will begin.

Capability will be established for measuring impurity concentration and epitaxial thickness by infrared methods although available instrumentation may limit the range of concentration or thickness which can be measured.

3. 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 alternate methods for measuring minority carrier lifetime in germanium and silicon which are more precise, more convenient, or more meaningful in the specification of material for device purposes.

<u>Progress</u>: Dispersion of light in the unusually thick lenses of the optical system in the steady-state photomagnetoelectric effect-photoconductivity (PME-PC) equipment made focusing the infrared illumination difficult to approximate by visible light. The problem was eliminated by changing to a front-surface-mirror optical system. In addition, the new system also provides greater illumination intensity with the same light source.

In order to determine whether the unused harmonics of the trapezoidal wave form of the chopped light were producing unwanted effects in the PME-PC measurement, the chopper was modified to produce a sinusoidal variation of light intensity by rotating an array of equi-spaced circular holes past a stationary slit with a width approximately equal to the

diameter of each of the circular holes [1]. A similar arrangement on the same chopper modulated the illumination of a photodiode to generate a reference signal. The nonlinear response of the photodiode generated higher order harmonics. The second harmonic, which had an amplitude a third that of the fundamental, was reduced 27 dB by an electronic lowpass filter. Such harmonic purity is not required for operation of the lock-in amplifier, but it is required for the substitution method of measuring the PME and PC currents because the calibration of these currents is based on the rms voltage input to the precision attenuator and only the fundamental component is utilized; therefore, the indicated current is low by the ratio of the rms value of the fundamental to the rms value of the fundamental plus harmonics. These modifications substantially improved operation of the equipment and are now being used in connection with the deep-level studies (see Section 7). (W. E. Phillips and A. W. Stallings)

Equipment for extending the measurement capability of the photoconductivity decay (PCD) lifetime apparatus to shorter lifetimes was considered. Planned studies on gold-doped silicon specimens will provide an opportunity to evaluate the applicability of the PCD method for measuring lifetimes in the nanoseconds or tens-of-nanoseconds range. The following systems are being evaluated: (1) a short-duration flash tube, (2) a steady light modulated rectangularly by a Pockels cell, and (3) a steady light modulated sinusoidally by a Pockels cell. The study includes an estimate of the performance of each of these systems, an estimate of the cost involved to assemble each system, and a survey of manufacturers from whom the equipment could be purchased.

The study of the contactless PCD lifetime measurement technique of Nishizawa, et al..[2] was continued. In this technique, the specimen is placed on a specimen holder which consists of two D-shaped electrical contacts embedded in a plastic block. The contacts are separated from each other by about 1 mm and are connected in parallel with the tank circuit of an r-f oscillator. The specimen is placed on an insulator, such as a piece of vinyl tape, mica, or one or more glass slides, which is placed on the contacts and serves as the dielectric of the capacitance connecting the specimen to the tank circuit. When the specimen illumination is varied, the change in conductivity of the specimen causes a change in the loading of the oscillator, which changes the Q of the

For an analysis of a similar system, see R. B. McQuistan, "On Radiation Modulation," J. Opt. Soc. Am. 29, 70-74 (1959).

J. Nishizawa, Y. Yamoguchi, N. Shoji, and Y. Tominaga, "Application of Siemens Method to Measure the Resistivity and Lifetime of Small Slices of Silicon," Ultrapurification of Semiconductor Materials, MacMillan Co., New York, 1962, pp. 636-644.

3. CARRIER LIFETIME

circuit, and produces a modulation of the oscillator amplitude. Demodulation of the oscillator output yields an illumination-dependent signal which is observed on an oscilloscope. The time constant of the signal which is observed after the illumination is extinguished is used to calculate the specimen lifetime.

The main difficulty with this technique is that the time constant of decay of excess carriers in the specimen is not always equal to the time constant of the demodulated oscillation envelope signal. This occurs because the equivalent resistance of the specimen is not in parallel with the tank circuit but is part of a network composed of the two coupling capacitors, specimen holder capacitances, and a resistor representing the effective specimen resistance. When a transformation is applied to this network it is found that changes in the effective specimen resistance and changes in the equivalent parallel resistance are not always proportional. Experimentally this leads to erroneously long lifetime measurements. It was found, however, that by centering the specimen on the specimen holder so that its longitudinal axis is perpendicular to the specimen holder slot, and by reducing the coupling capacitance by elevating the specimen as far above the specimen holder as possible consistent with obtaining a measurable signal, the maximum difference between contactless and standard PCD lifetime measurements on a group of twelve specimens under conditions of identical illumination was eight percent.

An apparatus to measure PCD lifetime by a second contactless method [3] is being constructed so that it may be compared with the method described above. In this method one measures the power absorbed by a specimen placed in a slotted cage inside the coil of an r-f oscillator tank circuit.

A draft of a revised standard procedure for measuring carrier lifetime by the PCD method has been prepared and is being reviewed prior to submission to ASTM Committee F-1. (R. L. Mattis)

Efforts on the study of the correlation of carrier lifetime in silicon with characteristics of nuclear radiation detectors made from the same material (see Section 20) were concentrated in the area of diode recovery measurements of carrier lifetime. Voltage decay measurements on point-contact diodes were continued. Point-contact diodes were selected for study because their fabrication leaves the bulk semiconductor material unaltered by heat treatment. Although the initial region of linear voltage decay seen with junction devices was not observed in point-

R. M. Lichtenstein and H. J. Willard, Jr., "Simple Contactless Method for Measuring Decay Time of Photoconductivity in Silicon," *Rev. Sci. Instrum.* 38, 133-134 (1967).

contact diodes (NBS Tech. Note 488, p. 6) it has been suggested [4] that the tail of the decay curve should be an exponential function of the lifetime. Measurements on diodes fabricated from wafers of several values of resistivity so far failed to reveal an exponential tail. Since the early portion of the voltage decay depends on the effective junction radius of the point-contact rather than the lifetime [5, 6], efforts were shifted to the reverse recovery technique. However, the recovery curves did not exhibit the flat region of constant reverse current such as was seen with p-n junction diodes so it was not possible to interpret them in terms of carrier lifetime. It can therefore be concluded that neither the voltage decay technique nor the reverse recovery technique is readily adaptable to making lifetime measurements with point-contact diodes.

Consideration of alternate types of diodes for this study led next to the surface barrier diode. Diodes for initial studies are being fabricated on *p*-type silicon with resistivity of 1500 Ω -cm. Ohmic contact is made to the base by an evaporated layer of gold on a lapped surface. The barrier is formed on the wafer by evaporating gold on the face of the wafer after etching.

An experiment to study the reproducibility of the reverse recovery technique for measuring carrier lifetime in p-n junction diodes is being conducted. Lifetimes of a group of seven commercial diodes have been measured monthly for a period of six months. (A. J. Baroody)

Plans: Study of the surface photovoltage method will be resumed when PME-PC measurements on indium antimonide are completed (see Section 7).

A decision will be made regarding the extension of the PCD measurement capability to shorter lifetimes. Comparisons of the two contactless methods for measuring PCD lifetime will be undertaken when the apparatus for the second method is completed. The draft of the revised standard method for PCD measurements will be reviewed and submitted to ASTM Committee F-l for its consideration.

Voltage decay measurements of diode recovery lifetimes will be made on surface barrier diodes. The voltage decay technique will also be applied to p-i-n, alloyed, and diffused diodes which will be fabricated. Mathematical and experimental investigation of the dependence of the

6. B. R. Gossick, "On the Transient Behavior of Semiconductor Rectifiers," J. Appl. Phys. 27, 905-911 (1956).

H. L. Armstrong, "On the Tail in the Transient Behavior of Point-Contact Diodes," Proc. IRE 45, 696-697 (1957).

^{5.} H. L. Armstrong, "On Open Circuit Transient Effects in Point-Contact Rectifiers," J. Appl. Phys., 27, 420-421 (1956).

3. CARRIER LIFETIME

carrier lifetime as measured by diode recovery techniques upon injection levels in the various diode structures will be continued.

Results of the experiment to study the reproducibility of the reverse recovery technique will be analyzed. When this is completed, the dependence of the measured reverse recovery time on the frequency and amplitude of the input square wave will be studied. Efforts to modify the circuitry to permit forward to reverse current ratios between 0.01 and 100 will continue.

A study of methods for measuring minority carrier lifetime in transistor structures will be undertaken. The problems encountered and the contradictions between methods and with bulk measurements will be investigated in regard to making more meaningful carrier lifetime measurements for device purposes. On the basis of such findings, further experimental carrier lifetime measurements under various geometries will be planned.

4. INHOMOGENEITIES

Objective: To develop improved methods for measuring inhomogeneities responsible for reduced performance and reliability of silicon devices and, in particular, to evaluate a photovoltaic method as a means to accomplish this.

Progress: It was decided to concentrate on the problems encountered in the surface treatment of *p*-type silicon. A number of surface treatments for reducing the surface recombination velocity, s, in *p*-type silicon were tried but their use continued to result in the observation of anomalous photovoltages as described previously (NBS Tech. Note 475, p. 8). Further investigation of these treatments was suspended, however, when success was achieved by modifying a treatment used earlier which had not consistently reduced s.

This treatment involves an etch (10 HNO_3 + 1 HF) to remove the work damage induced in shaping the specimen and an oxidizing agent (hot sodium dichromate) to create a surface accumulation layer which inhibits the diffusion of minority carriers to the surface. Consistent reductions in s were achieved with this treatment by increasing the etching time in the treatment from about 1.5 min to about 4.5 min which increased the thickness of the material removed from about 15 μ m to about 50 μ m. Apparently the shorter etching time used previously did not always remove enough of the work-damaged silicon.

Reproducible results have been obtained, using this preparation, with four *p*-type bar-shaped silicon specimens (two with resistivity of

4. INHOMOGENEITIES

v40 Ω -cm and two of v10 Ω -cm). Two of these specimens were ones for which the surface treatment using the shorter etching time had been unsuccessful. Resistivity profiles obtained from two-probe measurements were available for two of the specimens. They agreed with the respective photovoltaic resistivity profiles to within 1 percent. Such agreement is typical of previous measurements on silicon and germanium when the surface treatment had been successful.

A new airtight enclosure for the specimen holder with a transparent top for the light probe has been incorporated in the measurement apparatus. The use of the enclosure has much improved shielding from stray light and air currents.

Originally, the enclosure was constructed as part of the equipment to be used to maintain a controlled ambient atmosphere for the specimen during the surface treatment and the photovoltaic resistivity measurements if this proved to be necessary. Use of a controlled atmosphere was suspended at least until work on *n*-type silicon indicates that such measures will be required.

A preliminary attempt was made to reduce s by placing the specimen between transparent capacitor plates and applying a voltage to induce an accumulation layer in the specimen surface. No measurable effect was observed for applied voltage as high as 550 V. Additional study of this method was suspended in view of the success of the surface treatment on p-type silicon.

To increase the generally smaller values of photovoltage and photoconductivity measured on circular specimens, a 500-W incandescent lamp was tried. Because of the less efficient focusing element in the lamp, the intensity of light that reached the specimen was not significantly greater than that obtained from the 150-W lamp used previously. Another alternative which has been considered but not yet tried is the use of a helium-neon laser with an output power of 50 mW at 1.15 µm and of 5 mW at 1.084 µm. The output power at 1.15 µm is estimated to be greater than the power incident on the specimen from the incandescent lamp because of the highly inefficient optical means of forming the light probe from the lamp.

Work on circular specimens was deferred during this quarter to concentrate on improving the surface treatment of p-type silicon. (D. L. Blackburn and H. A. Schafft)

Plans: Photovoltaic resistivity measurements will be made on p-type silicon bars with a resistivity of about 1 Ω -cm to investigate problems in making measurements at lower resistivity levels.

Photovoltaic resistivity measurements will be made on *n*-type germanium and *p*-type silicon circular specimens to substantiate the

4. INHOMOGENEITIES

usefulness of the equation which relates resistivity gradient to photovoltage (NBS Tech. Note 488, p. 9).

Efforts will be made to improve the surface treatment for *n*-type silicon. The first treatment to be examined is similar to the one used successfully for *p*-type silicon except that a reducing agent is substituted for the oxidizing agent.

A preliminary trial of a laser source will be made to determine its utility as a light probe for circular specimens.

5. INFRARED METHODS

Objective: To evaluate impurity photoconductivity as a method for detecting low concentrations of deep-lying impurities such as copper, gold, iron, and nickel in silicon and germanium, and to assist ASTM Committee F-l in extending the applicability of infrared absorption as a method for detecting impurities such as oxygen and carbon in silicon and germanium.

Progress: Specimens of copper-doped germanium were prepared for impurity photoconductivity measurements by diffusing copper, which had been electroplated on the surfaces, into the germanium for a time sufficient to saturate the specimen. A specimen with 1014 copper atoms/cm3 was prepared by heating at 570°C for 288 hours, and a specimen with 10^{15} copper atoms/cm³, by heating at 650°C for 48 hours. The specimens were quenched in water. So that the results will be useful for the germanium program (see Section 10), the starting material was 20 Ω -cm p-type germanium, rather than n-type which is normally used for infrared detectors. Measurements are made with the specimen mounted on the cold finger of a liquid helium cryostat. The electrical circuit consists of a d-c power supply and load resistor in series with the specimen. The response as a function of wavelength is determined across either the load resistor or specimen with a lock-in detector. So far photoconductivity has been observed only to 20 µm, but improvements are under way so that response can be detected at longer wavelengths. The long wavelength limit for copper in germanium is 31 µm, which corresponds to the copper impurity level at 0.04 eV above the valence band. (W. R. Thurber)

Plans: Photoconductivity measurements will continue on the germanium specimens saturated with copper.

A summary report will be prepared describing the work completed on the determination of oxygen in silicon and germanium by infrared absorption methods.

6. HALL EFFECT

Objective: To establish a facility for making measurements of Hall coefficient as a function of temperature between 4 and 350 K and to improve methods for collecting and interpreting Hall effect data.

Progress: The Hall effect apparatus is being modified to permit automatic measurements on high-resistivity materials. The design work is essentially completed, operational amplifiers have been purchased, and a new specimen holder is being fabricated.

The report concerning the use of a time-shared computer to control the Hall experiment is nearly complete. Work on the report on Hall measurements and their interpretation is continuing.

(W. R. Thurber and W. M. Bullis)

Plans: Work will continue on the new Hall apparatus which will be used for measurements of high-purity germanium (see Section 10) and golddoped silicon (see Section 8).

The two reports now being prepared will be completed and published.

7. DEEP-LEVEL STUDIES

Objective: To determine the nature and origin of the deep-lying centers in high-resistivity indium antimonide.

Progress: From the results of resistivity, Hall coefficient, and carrier lifetime measurements on high-resistivity indium antimonide before and after the introduction of lithium, it was concluded that the residual deep-lying energy level in indium antimonide is associated with a lattice vacancy, probably on the indium sublattice. Lithium added to the material fills the vacancies and removes the residual level [1].

The change caused by the addition of lithium was clearly evident in the lifetime measurements: at liquid nitrogen temperature, the electron lifetime was four orders of magnitude greater in the specimens treated with lithium. The interpretation of lifetime data was not completely unambiguous, however, because the variation of lifetime with temperature was not of the form required by elementary theory. In particular, the lifetimes calculated from photoconductivity and photomagnetoelectric

J. L. Scales, "Investigation of the Residual Defect in Indium Antimonide," Ph. D. thesis, Physics Department, University of Maryland (March, 1969).

effect measurements are expected to be equal at some warmer temperature, according to the theory, but this was not observed.

Consequently attempts have been made to improve the lifetime measurements. The optical system was changed, and a new light chopper was made (see Section 3). In addition a null method was adopted for measuring the induced photo-voltages. In the new arrangement, the specimen is driven with a sinusoidal voltage that has the same frequency as, but can be adjusted to be 180 deg out of phase with, the induced signal. By varying the amplitude of the driving voltage, one can null out the induced photo-voltage without turning off the light or changing any experimental condition, as was previously necessary. (J. L. Scales)

Plans: Some lifetime measurements on Li-treated and untreated specimens will be repeated, using the improved apparatus and the null techniques for making measurements. It is hoped that the discrepancy between theory and experiment at the warmer temperatures can be resolved. A final report will be prepared on completion of the measurements.

8. 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.

<u>Progress</u>: Work on establishment of procedures for diffusing gold into silicon wafers was initiated. A gold film, 40-nm thick, was vacuum evaporated onto the polished surface of a number of $10-\Omega$ -cm boron-doped silicon wafers preparatory to carrying out diffusions. No difficulty was experienced with the adhesion of the gold to the silicon surface. (J. Krawczyk)

Plans: Gold diffusions will be carried out at 850°C, 950°C, 1050°C, 1150°C, and 1250°C for one-half hour to obtain specimens containing different amounts of gold. Diffusions will be done in an oxygen atmosphere.

Electrical measurements on specimens before and after gold diffusion will begin.

9. HIGH FIELD EFFECTS

Objective: To study the physical characteristics of hot carrier semiconductor structures and relate these to performance of devices.

9. HIGH FIELD EFFECTS

Progress: The priority assigned to this task has been reduced to permit acceleration of the wire bond evaluation task (see Section 13). (G. G. Harman)

Plans: As time becomes available, further measurements will be made. The effect of strong magnetic fields will be investigated and the temperature dependence of the oscillation will be studied.

10. 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.

<u>Progress</u>: Pulse height distributions (gamma-ray peaks) produced by monoenergetic gamma-rays in Ge(Li) detectors are being studied to deduce information on crystal quality. Spectral.data obtained from collimatedbeam scanning of Ge(Li) detectors have been analyzed by a specially written computer program, PEAKFIT-III, written in OMNITAB programming language [1]. Each data set, consisting of the 662-keV 137 Cs gamma-ray peak and a pulser-generated peak at somewhat higher equivalent energy, is analyzed to obtain the positions of the peak centroids, resolution at l/2 and l/10 maximum peak height, the detector contribution to peak width, ΔE_D , and the ratio of the variance in the number of ion pairs produced to the number of ion pairs produced, σ^2/\bar{N} . The background distribution was fitted by a polynomial of degree 2 and subtracted from the raw data prior to peak analysis [2]. Using the method of least squares a function of the following form is fitted to the spectral peaks:

$$Y(x_i) = \exp(a + bx_i + cx_i^2)$$
 (1)

where $Y(x_i)$ is the number of counts in channel x_i and a, b, and c are adjustable constants.

2. The origin of the distribution of points between the full energy peak and the Compton "knee" is the subject of continuing study.

Supported by the Division of Biology and Medicine, U. S. Atomic Energy Commission. (NBS Project 4259425)

^{1.} J. Hilsenrath, G. G. Ziegler, C. G. Messina, P. J. Walsh, and R. J. Herbold, *OMNITAB*, *A Computer Program for Statistical and Numerical Analysis*, NBS Handbook 101, rev. ed., January, 1968.

10. SPECIFICATION OF GERMANIUM

Reports of measurements of an effective Fano factor which have previously been made using uncollimated gamma-ray sources included attempts to dissociate the effects of carrier trapping and noise from the result [3-5]. These studies yielded a value of about 0.13 for the Fano factor in germanium. In the present work, using a collimated source of gamma-rays, it has been possible to determine explicitly the region of a Ge(Li) detector where carrier trapping is minimized and thus obtain a better measure of the Fano factor.

Measurements of the detector resolution (ΔE_D) of a 2-cm³ Ge(Li) detector as a function of both electric field and beam scanning position between the n^+ -i and i-p junctions were made by recording pulse height spectra of the 662-keV ¹³⁷Cs gamma-ray peak along with a pulser-generated peak. Analyses of ΔE_D as a function of scanning position showed that at low electric fields electrons were preferentially trapped in the detector; at high fields the trapping was greatly reduced. However, irradiation of the region 0.5 to 1.5 mm from the n^+ -contact, where the effect of electron trapping is minimized because the signal is due mainly to holes traversing the *i*-region, yielded the greatest improvement in detector resolution.

The quantity σ^2/\bar{N} plotted against distance from the n^+ -contact of the detector is shown in Fig. 1. At the top of the figure, the boundaries of the *i*-region are indicated at 0.5 and 5.5 mm from the contact surface. The depth of the n^+ -region (500 µm) is in good agreement with measurements made after the initial diffusion of lithium into the crystal [6]. The data for a field of 1200 V/cm (single measurements) show that σ^2/\bar{N} increases (from increased ΔE_p) as the beam moves toward the *i*-*p* junction. Spectra are obtained when the beam is centered at the n^+ contact (0 mm) due both to the finite width of the beam and to the larger width of the secondary ionization sheath produced in the detector. These points are shown only for reference purposes. Data points at an electric field of 2400 V/cm show values of σ^2/\bar{N} which are significantly lower than 0.13 by one-sided 99.5 percent confidence interval t-test [7] in the

- 3. H. R. Bilger, "Fano Factor in Germanium at 77°K," Phys. Rev. <u>193</u>, 283-253 (1967).
- 4. J. M. Palms, P. Venugopalo Rao, and R. E. Wood, "The Characteristics of an Ultra-High Resolution Ge(Li) Spectrometer for Singles and Coincidence X-Ray Gamma-ray Spectra," *IEEE Trans. Nucl. Sci.* <u>NS-16</u>, 36-46 (January, 1969).
- 5. A. H. Sher and B. D. Pate, "Determination of the Fano Factor in Germanium at 77°K," *Nucl. Instr. and Meth.*, to be published.
- A. H. Sher, "Lithium Ion Drift Mobility in Germanium," J. Appl. Phys. 40, 2600-2607 (1969).
- 7. M. G. Natrella, *Experimental Statistics*, NBS Handbook 91, rev. ed., 1966, p. 3-20.

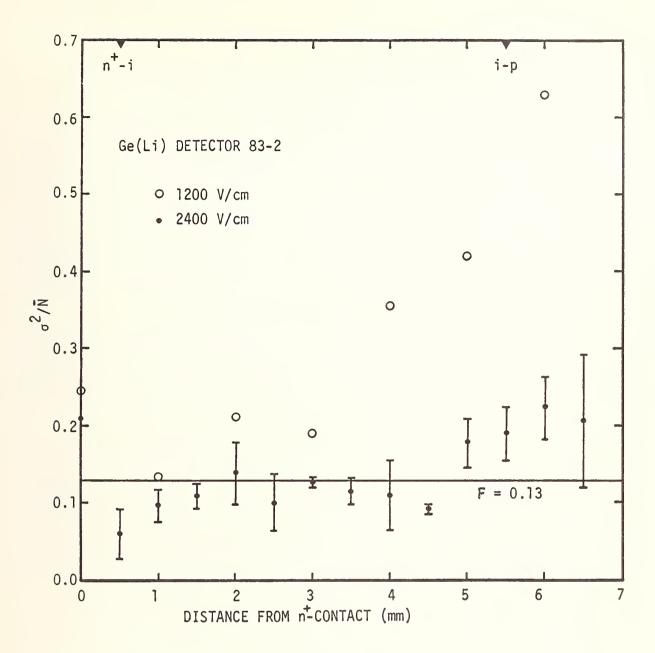


Fig. 1: σ^2/\bar{N} as a function of distance from the n^+ -contact of Ge(Li) detector 83-2. Error bars on the data points for the field of 2400 V/cm are the 95 percent confidence interval estimates of the mean [7]. Estimated standard deviations are smaller.

region from 0.5 to 1.5 mm. The error bars represent the two-sided 95 percent confidence interval [7] of the mean of at least four measurements. The mean value of σ^2/\bar{N} for data points from 2.0 to 4.5 mm is 0.114 but is not significantly different from 0.13 by the above *t*-test. Analysis as in earlier reports [3-5] of the response of the detector operated at 2400 V/cm when irradiated by an uncollimated 137 Cs source yielded $\sigma^2/\bar{N} = 0.133$. If the upper limit of F is given by σ^2/\bar{N} , the present results show that for the region of this detector near the n^+ -contact, where electron trapping is negligible, F < 0.13. It is difficult to assign a specific value for the Fano factor, however, as secondary ionization effects make detailed knowledge of the beam width difficult to obtain, but it is probable that an upper limit for the Fano factor is $\sigma^2/\bar{N} \approx 0.10$, the average value in the region 0.5 to 1.5 mm from the contact. These results have been submitted for presentation at the 1969 Nuclear Science Symposium. (A. H. Sher and W. J. Keery)

Specimens of germanium doped with copper were prepared for lowtemperature impurity photoconductivity measurements so that data could be obtained on crystals with known impurity concentrations before examining crystals included in the germanium study (see Section 5). A liquid nitrogen cryostat with a germanium window has been constructed to study the infrared response of germanium p-i-n diodes in order to determine if correlations exist between the two infrared analytical techniques. A suitable preamplifier for use with the cryostat is being constructed. (W. R. Thurber and A. H. Sher)

Plans: Lithium mobility studies (including effects of surface type) and detector performance measurements with emphasis on carrier trapping utilizing a collimated gamma-ray beam and subsequent analysis of the peak shape will be continued. Impurity photoconductivity measurements (see Section 5) will be made on germanium crystals which show high degrees of charge-trapping. Infrared response measurements at 77 K will be made on Ge(Li) detector structures fabricated from the same material.

METHODS OF MEASUREMENT FOR SEMICONDUCTOR PROCESS CONTROL

11. METALLIZATION EVALUATION

Objective: To improve methods for measuring the properties of thin metal films with initial emphasis on adhesion of aluminum metallization deposited on various substrates.

Progress: The literature search on thin film adhesion has indicated that the scratch test appears to be the only adhesion test which is presently capable of yielding quantitative and consistent data. The scratch test basically consists of mechanically loading a spherically tipped stylus, and then drawing it smoothly across a thin metal film. The film adhesion force is proportional to the minimum loading on the stylus at which the film under the stylus is removed.

A simple scratch test apparatus has been assembled to perform preliminary studies on this test method. Except that a dynamometer is used both as the loading force generator and as the measuring device, this apparatus is similar to the apparatus outlined in the draft method for performing the scratch test which is now being developed by ASTM Committee F-1.

In order to calculate the shear force required to remove a film from its substrate when making a scratch test, it is necessary to know the radius of curvature of the stylus tip. The shear stress is a sensitive function of the tip radius, thereby making an accurate determination of this quantity quite necessary. The tip radii of the diamond styli now being used are less than 25 μ m. Shadowgraph techniques for determining the radius of curvature were found to be inadequate. Instead, techniques are being developed to determine the geometry of the tip from a series of photomicrographs.

An extensive listing of unclassified reports and abstracts related to thin metallic films and adhesion, supplied by the Defense Documentation Center, is being searched for work relating to thin film adhesion testing. (J. Oroshnik)

To permit operation of the substrate at higher temperatures during evaporations carried out to prepare film samples, a platinum-bonded silicon sandwich temperature detector was constructed to replace the goldbonded unit previously described. During assembly, the unit cracked, most probably due to the thermal expansion mismatch between the silicon and platinum. It is believed that this can be remedied by using a considerably thinner platinum foil or by sputtering about 800 nm of platinum on the silicon surfaces to be alloy-bonded. (W. K. Croll)

11. METALLIZATION EVALUATION

Plans: The literature search on thin film adhesion will continue. Suitable optical systems will be considered for observing the scratches produced with the adhesion test device. Since transmitted light is most convenient for observing a scratch at the failure point, the initial system which will use visible light will be suitable for aluminum films deposited on transparent substrates. Later, a second system which will use infrared light will be built for use with films deposited on silicon dioxide substrates over silicon. Work will also continue on developing a satisfactory method for determining the radius of curvature of the stylus tip.

Consideration of the pull test as an alternative method for measuring adhesion strength will continue. A search will be made for a cement suitable for bonding to aluminum dots. Modifications to the design of the platinum-bonded silicon sandwich temperature sensor will be made, and a new sensor will be constructed.

12. PROCESSING FACILITY

<u>Objective</u>: To establish a microelectronics fabrication laboratory consisting of the facilities necessary for the production of specialized silicon devices for use in research on measurement methods.

Progress: The photomasking facility was developed to the point that 12.5-µm (0.5-mil) lines can be photoetched in either aluminum or silicon dioxide. This resolution is sufficient for the devices currently needed for wire bonding and other research studies. (J. Krawczyk)

The assembly of a pinhole camera was undertaken for the purpose of making photomasks. Using very simple optical techniques, photomask sets suitable for present requirements can be produced at relatively low cost and with a very modest equipment investment.

A groove grinding device was designed and built which produces a polished cylindrical groove in the surface of the silicon wafer for measuring junction depths [1]. The grooving technique was found to be more convenient and faster than the conventional angle lapping method [1]. Preliminary studies of various staining techniques for delineating junctions have been initiated. (T. Leedy)

A new heat-treating furnace and its associated control equipment was assembled and put into operation. This furnace, which has a working bore

B. McDonald and A. Goetzberger, "Measurement of the Depth of Diffused Layers in Silicon by the Grooving Method," J. Electrochem. Soc. 109, 141-144 (1962).

of about 7.5 cm, will accommodate fifteen 2.5-cm diameter wafers within its flat zone. It has a maximum working temperature of 1010°C, provision for atmospheres of dry hydrogen, helium, or nitrogen, and allows considerable flexibility in heat treating evaporated aluminum films. (W. K. Croll and J. Krawczyk)

Several circuit analysis computer programs are being investigated to determine their usefulness in establishing fabrication conditions for devices which may be required for research on measurement methods. Since the effects of variations of diffusion concentrations on gain and frequency response may be predicted, it is possible to test various fabrication parameters prior to actual construction of devices. Each of these computer programs has its own advantages. SNAP-II [2] has the capability of solving circuit problems in the frequency domain, and includes provisions for sensitivity testing and Monte Carlo analysis. ECAP [3] is characterized by its ease of computer programming and its ability to solve problems in either the frequency or time domain. CIRCUS [4] simulates the time domain response of an electronic circuit to an arbitrary forcing function. (T. F. Leedy)

Plans: Mechanical work on the photomasking camera should be completed by the end of the next quarter. Further studies of staining techniques for junction delineation will continue. The computer programs will become operational on the NBS computer and further studies of their applicability to the needs of the Program will be made.

13. WIRE BOND EVALUATION

Objective: To survey and evaluate methods for characterizing wire bond systems in semiconductor devices and where necessary to improve

- 2. T. D. Price, C. M. Kimme, and J. R. Gliessman, "User's Manual for SNAP-II Computer Program," Prepared for U. S. Naval Weapons Center, China Lake, California, by ARINC Research Corporation, Western Division, Santa Ana, California. Publication 474-01-1-909. (Available from Naval Weapons Center.)
- 3. IBM Application Program, "1620 Electronic Circuit Analysis Program (ECAP) (1620-EE-02X) User's Manual," (Available from either IBM, Technical Publications Department, 112 East Post Road, White Plains, N. Y. 10601 or UNIVAC Division of Sperry Rand Corporation, P. O. Box 8100, Philadelphia, Pa. 19101.)
- 4. L. D. Milliman, W. A. Massena, and R. H. Dickhaut, "CIRCUS--A Digital Computer Program for Transient Analysis of Electronic Circuits--User's Guide," Prepared for U. S. Army Material Command, Harry Diamond Laboratories, Washington, D. C. 20438, by The Boeing Company, P. O. Box 3999, Seattle, Washington, 98124, Publication 346-1.

existing methods or develop new methods in order to detect more reliably those bonds which eventually will fail.

<u>Progress</u>: A new request, accompanied by a more detailed description of project requirements, was sent to the Defense Documentation Center (DDC) for a search of reports and work units for possible inclusion in the bibliography. DDC used a two-level search strategy: only those reports and work units were selected which were indexed in at least one category in each of the two levels. Level one contained the categories: semiconductor devices, integrated circuits, and microelectronics. Level two contained the categories: circuit interconnections, bonding, bonded joints, and ultrasonic welding. A total of 41 work units and a report bibliography containing 357 entries were received from DDC. About onefourth of these work units and entries were found to be of interest. In consultation with a DDC official it was concluded that the search conducted had collected all reports of interest within the limits imposed by the indexing.

The survey of the journal literature continued. A patent search was conducted which found 44 patents of interest. A request for a bibliographic search was sent to the Reliability Analysis Center [1]. A list of 93 references (DDC reports, company reports, and journal papers) was received shortly before the end of the quarter.

(H. A. Schafft and E. C. Cohen)

The preparation of a detailed outline of the critical review survey paper is in progress. (H. A. Schafft)

Work on the evaluation of aluminum ribbon wire has continued during the guarter. New 1.5 by 0.5-mil wire which has the same cross section as 1-mil round wire was received from the vendor. This wire contained 1 percent silicon and had a nominal tensile strength of 14 g. It was obtained from the same source as the round wire used in comparisons and both had the same specifications except for dimensions. Experiments were made using various bonding tools, ultrasonic power levels, and bonding pressures. The bonds were made to aluminum metallization evaporated on both silicon and silicon oxide. It was found that the present ribbon wire with 14-g tensile strength produces bonds with statistically similar pull strengths to equivalent round wire. Under constant ultrasonic energy conditions ribbon wire requires lower absolute bonding force than round wire. In addition, this is distributed over a larger area. Hence, hard wire (>24-g tensile strength), which would result in considerably greater bond strengths, can be used without greater damage to the semiconductor chip. Hard ribbon wire for such tests has been ordered.

Administered for the Department of Defense by the Rome Air Development Center, U. S. Air Force, and operated for that Center by the IIT Research Institute, 10 West 35th Street, Chicago, Illinois 60610.

13. WIRE BOND EVALUATION

In addition the bonding operator finds ribbon wire more convenient to use than round wire. Ribbon wire is easier to grasp with tweezers, and there is less chance of damaging it in the process. The wire stays centered under the bonding tool and accurate centering on a small pad is easier and quicker. (K. O. Leedy)

Near the end of the quarter, work was initiated on the effect of metallization sintering temperature on bond strength. Sintering at temperatures ranging from 425 to 575°C is being investigated. (W. K. Croll, K. O. Leedy, and L. R. Williams)

Several methods of monitoring ultrasonic parameters during the bonding operation were investigated. The first method used a non-contacting magnetic pickup placed near the tungsten carbide bonding tool. The tool is weakly ferromagnetic since the tungsten carbide particles are cemented with cobalt. The loading of the bonding tool results in decreasing tool velocity or vibrational amplitude and hence an output signal that decreases in amplitude during bond formation. Therefore, this method affords some indication of bond quality. There was some inconvenience in using this system while actually making a bond because the available shielded polarized magnetic pickup coil was larger than desired. Further work on this system has been postponed until a smaller coil can be obtained.

Another form of ultrasonic parameter monitoring was studied by observing the 60-kHz voltage applied across the ultrasonic transducer from a relatively high impedance source. The transducer which drives the bonding tool was of the nickel magnetostrictive type having an acoustical Q of about 100. The tool loading during bond formation results in lowered impedance of the transducer and a corresponding decrease in voltage across the transducer. Thus the information on bond quality obtained by this method is presumably similar to that which would be obtained with the magnetic pickup system.

As a third approach, ceramic transducers were placed at various points on the ultrasonic horn and on the tungsten carbide bonding tool [2]. The bond monitoring information obtained from this system was similar to the other methods. The high-impedance transducer required electrostatic shielding to avoid stray pickup.

The final parameter monitoring experiment involved the use of a capacitor microphone to detect the ultrasonic pressure radiated from the

S. Bonis, "Poseidon Semiconductor Captive Line Process Development," Progress Report, 16 September 1968 to 15 November 1968, Report R68-4566, Raytheon Company, Space and Information Systems Division, Sudbury, Mass. 01776 (AD 844820L).

13. WIRE BOND EVALUATION

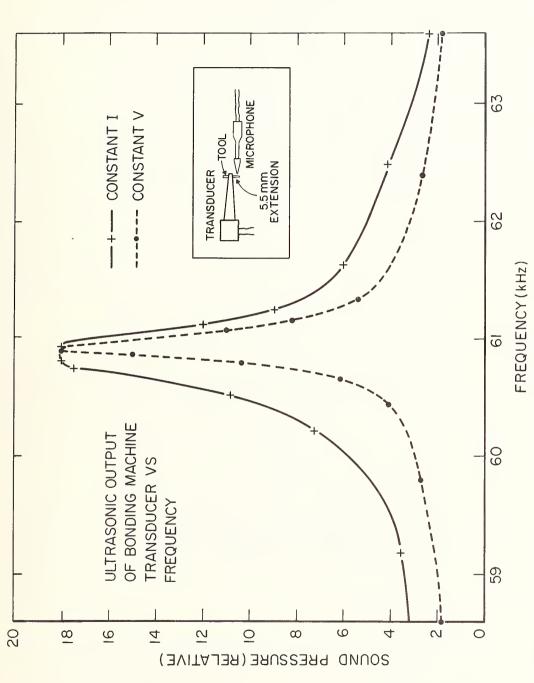
bonding tool tip during bond formation [3]. Addition of a simple directional extension cone damped with silicon rubber and polytetrafluoroethylene to the microphone allowed sufficiently high resolution to detect the sound pressure from a 0.5-mm diameter area located within 1 mm of the bonding tool tip. A related experiment was performed by measuring the ultrasonic vibration transmitted (from tool through wire into the chip) from the substrate. Both procedures gave results that may be useful in bond monitoring, but were experimentally awkward because of the physical size of the available ultrasonic microphone. However, this is the only simple way that one can measure a known parameter, sound pressure, which is directly related to the bonding tip motion. The other methods measure an indirect parameter or a vibration at some point remote from the actual bonding operation. Therefore, despite its physical awk-wardness, the capacitor microphone has been selected as the primary means for studying the bonding process and for understanding the significance of various other ultrasonic bond monitoring methods.

An additional method of studying the ultrasonic bonding tool motion was considered. This consists of reflecting light, possibly a laser beam, off the bonding tool or horn and observing the amplitude modulation. Commercial equipment exists that is capable of such measurements. However, since the microphone method has sufficient resolution for the purpose, the investment required to set up the optical system does not appear to be warranted at the present time.

The objective of these ultrasonic bond monitoring studies was to assemble equipment and survey various possible monitoring methods. No statistical study of the resulting bonds has yet been made and no single method has been chosen as being practical for in-process bond monitoring. All of these methods gave the same qualitative results, although only the microphone gave results in terms of known parameters. All methods give some information on the bonding tool loading as manifested by a change in tip vibration amplitude due to deformation of the wire. Only a much more intensive investigation can determine the relative sensitivity of each method to subtle changes in bonding conditions. (H. K. Kessler)

In studying any physical system, in this case the ultrasonic bonding system, it is necessary to characterize the important components in a way that is meaningful to the end use. There is no consensus on such subjects as the optimum impedance of the ultrasonic power supply (constant current or constant voltage) or the effect of sweeping the ultrasonic drive frequency. To begin characterization of the power supply, resonance curves were made using a nickel magnetostrictive transducer including the tapered horn and bonding tool. The ultrasonic sound pressure was measured directly in front of the bonding tool tip using the high-resolution

Neil Scott, Hughes Aircraft Company, Newport Beach, California 92663, private communication.



The d-c bias and Sound pressure from an ultrasonic bonding tool and transducer versus frequency for constant current and constant voltage. The inset gives the experimental arrangement. ultrasonic power were the same as used for typical bonding operations. 2 Fig.

13. WIRE BOND EVALUATION

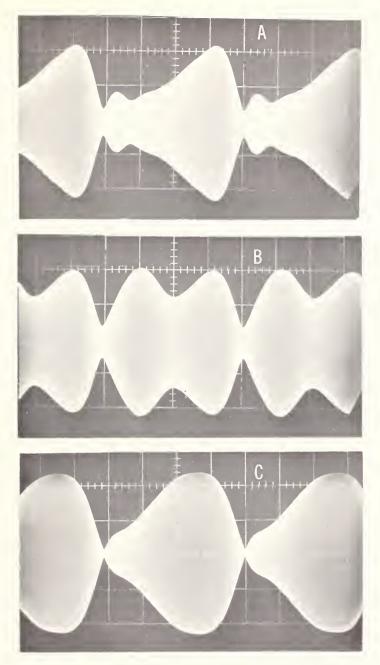
extension cone on the microphone. This gave the entire system response in the direction of the longitudinal axis of the horn. Measurements were made at both constant voltage and constant current as a function of frequency. The frequency was measured to within several hertz with a digital frequency meter. Sound pressure data were taken directly from the tool tip with various bonding tool extensions as well as directly off the end of the horn.

Typical resonance curves are shown in Fig. 2. The electrical Q of the system as measured in the external circuit was about 130. It is obvious from Fig. 2 that the acoustical Q is strongly dependent on the impedance of the external driving circuit. Even a small-value resistor inserted in series with the power supply will affect the acoustical Q appreciably. The constant voltage (low impedance source) curve is very sharp: $Q \approx 270$. For this case a driving source with extremely stable frequency is required. In addition heavy loading of the bonding tool tip could throw this system off resonance. The constant current curve (higher impedance source) has a lower Q (\approx 140) with a broad, almost flat, top. Values of Q as low as 100 have been observed with slightly different power supply characteristics. It is apparent that constant current drive would result in relatively constant ultrasonic tool motion even if the source frequency drifted or a large load were applied to the bonding tool.

The ultrasonic microphone was used to examine the displacement amplitude of the bonding tool tip while the transducer was driven in the swept-frequency mode with a relatively low Q system. The envelope of the displacement amplitude for three values of the center frequency, f₀, is displayed in Fig. 3. The shape of the envelope can be changed significantly by relatively small changes in the center frequency. Thus, to maintain reproducible bonding conditions, it is necessary both to have a stable oscillator and to exercise care in tuning the ultrasonic system even when the swept-frequency mode is used.

While making the above measurements it was found that the maximum tip vibration amplitude occurred at a different frequency from the transducer resonance as indicated by a current meter in series with the transducer coil. Thus, it is apparent from the swept-frequency oscillograms and the acoustical resonance curves that regardless of the power supply characteristics an ultrasonic microphone is almost essential for tuning the ultrasonic system of a bonding machine.

In addition to the detailed resonance curve around the normal 60-kHz operating frequency, the broad frequency response of the system was measured at the vibrating tool tip. There were from 13 to 18 other resonances between 7 and 90 kHz. Different bonding tool extensions do not appreciably effect the 60-kHz resonant frequency but many of the other resonances are dependent on specific extension lengths.



- Fig. 3 Three oscillograms of the sound pressure from the tip of a bonding tool. These patterns result from sweeping the frequency of the power supply by approximately 1200 Hz. The vertical axis is proportional to bonding tool displacement. The horizontal axis is time (1 ms/division). The sweep period is about 4 ms.
 - A: $f_0 = 60.50$ kHz (manufacturer's specification).
 - B: f₀ = 61.00 kHz (system was tuned to give maximum ultrasonic output without sweeping).
 - C: f₀ = 61.38 kHz (pattern least affected by slight detuning of the center frequency).

It has been reported that the resonant nodes of bonding tools can be seen by sprinkling lycopodium dust over the tool [4]. Other powders, that may be more readily available, have been found to perform the same function [5]. The powder-delineated nodes have been clearly observed by using the ultrasonic microphone with its high-resolution extension tip. With this system the resonant maxima can also be clearly observed. By studying the amplitude of the sound pressure around the perimeter of the bonding tool it is expected that torsional and other modes of vibration can be observed. One common opinion is that varying the drive frequency around the resonance (sweeping or wobbling) may excite such different vibrational modes. (G. G. Harman)

There are many advantages to ball bonding as opposed to wedge bonding. The ball is commonly used with gold wire in thermocompression bonding and occasionally for ultrasonic stitch bonding. However, gold does not easily bond ultrasonically. If an aluminum ball could be easily and reproducibly formed on 1-mil wire, then it should be possible to make strong ultrasonic ball bonds. Because of oxide formation, aluminum does not ball when flamed. In a feasibility experiment performed to make such a ball a capacitor discharge across a spark gap supplied the necessary heat to melt the aluminum wire, and dry nitrogen prevented oxide formation. A number of good aluminum balls were made. There was more variability in their diameter than would be desired for production use, but the results were sufficiently good to warrant continued investigation. (H. K. Kessler)

Plans: The collection of papers for the bibliography, the assignment of key words for each entry, and the indexes will be completed and the typing of the manuscript will be under way by the end of the next quarter. Work on the critical review paper will continue.

Investigation of the tensile strength of wire and wire bond systems after burn-in at various temperatures will continue. In some cases devices will be operated for greatly extended periods in order to simulate normal system operation. These tests will include wire obtained from several different sources. Cross correlations will be made between the optimum-angle pull strength, the loop height, and the length of normal stitch bonds with the use of the hot-melt-glue bond puller. The work on ribbon wire will resume when new, harder wire is received. The effect of metallization sintering time and temperature on ultimate wire bond strength will continue.

^{4.} S. Bonis, "Poseidon Semiconductor Captive Line Process Development," Progress Report, 16 November 1968 to 15 March 1969, Report R69-4198, Raytheon Company, Space and Information Systems Division, Sudbury, Mass. 01776.

^{5.} Such as Shell VPI #250, Shell Oil Company, New York, New York 10020.

13. WIRE BOND EVALUATION

Several ultrasonic bond monitoring systems will be investigated in greater detail with emphasis on the use of an ultrasonic microphone in an effort to obtain a fundamental understanding of such systems. Ultimately the results will be compared with wire pull and "push" tests. A highcurrent pulse method for nondestructive wire bond testing will be investigated and the feasibility study of aluminum wire ball formation will be concluded.

14. DIE ATTACHMENT EVALUATION

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

<u>Progress</u>: The literature search on the techniques utilized in evaluating the uniformity and quality of semiconductor device die attachment was continued. This search is being carried out in conjunction with the Wire Bond Evaluation search (see Section 13). An industrial organization concerned with both the manufacturing and utilization of semiconductor devices was visited to discuss the techniques used to detect poor die attachment. The information gathered thus far indicates that not only is a precise die bond evaluation technique needed but also a more repeatable technique for making die bonds themselves.

X-ray equipment capable of conveniently detecting voids in the die attachment was installed and is operational. Radiographs can be made on a variety of film types and sizes. (F. F. Oettinger)

Plans: The literature search on the techniques utilized in evaluating the uniformity and quality of semiconductor device die attachment will continue. Radiographic studies of various chip and packaging configurations will be made to determine practical sizes and locations for voids. Following these studies fabrication of test diodes with controlled voids will begin.

15. NASA MEASUREMENT METHODS

Objective: To review existing semiconductor test method standards for materials and process control measurements and to prepare interim test methods in a standard format as may be appropriate.

Progress: A draft of an interim method for measuring oxide and metallization thickness has been completed and has been sent to the NASA Electronics Research Center for review.

15. NABA MEASUREMENT METHODS

Responses were tabulated for the ASTM Committee F-l questionnaire on use and adequacy of ASTM standards for microcircuit processing. The tabulation has been sent to the ASTM Committee F-l Chairman and is consistent with the preliminary conclusions reported previously (NBS Tech. Note 475, p. 24). (W. E. Phillips)

A review of NASA test methods [1] was completed and sent to the NASA Electronics Research Center.

Information on various leak testing methods currently in use or proposed by NASA, the military and other organizations was assembled and presented to ASTM Committee F-1. The Committee will modify existing tentative and draft methods as a result of this activity. (W. M. Bullis)

Plans: In connection with other tasks, procedures for determining junction depth (see Section 12) and conductivity of thin metallic films will be reviewed. Additional assistance will be given ASTM Committee F-1 in the development of standard procedures for leak testing.

 [&]quot;Test Standards for Microcircuits," Draft of NASA-STD-XX-3, December 1, 1968.

METHODS OF MEASUREMENT FOR SEMICONDUCTOR DEVICES

16. SECOND BREAKDOWN

Objective: To maintain an awareness of progress in the field of second breakdown and to assist both manufacturers and users of semiconductor junction devices in the development and use of meaningful specifications for maximum operating conditions free from second breakdown.

Progress: The first draft of the manuscript on "Failure Modes" was completed and submitted to the task group of the JEDEC Committee on Power Transistors, JS-6, responsible for writing a chapter titled "Users Guide for Power Transistors." This chapter is to be part of a JEDEC suggested standard to be titled "Recommended Standards for Power Transistors."

A definition for second breakdown was proposed to Committee JS-6 for possible use in "Recommended Standards for Power Transistors" and to the technical adviser to the U. S. National Committee of IEC Technical Committee No. 47 on Semiconductor Devices. (H. A. Schafft)

Plans: Assistance will be given to the JS-6 group in the revision of the initial drafts of the chapter "Users Guide for Power Transistors".

The review of the status of the second breakdown field with particular emphasis on radiation-induced effects will begin after work on the wire bond bibliography and survey is complete (see Section 13).

17. THERMAL PROPERTIES OF DEVICES

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

Progress: The literature search and the review of the methods of measurement of thermal resistance and transient thermal response of semiconductor devices were continued. Since thermal resistance alone, either steady-state or transient, is inadequate for fully characterizing thermal performance of semiconductor devices, the scope of the literature search has been expanded to include articles on other semiconductor properties which contain information of value in understanding thermal behavior. (M. Sigman and F. F. Oettinger)

The circuit modifications that were made to reduce the switching transients caused by the termination of the power pulse were only partially successful, but measurements can be made within 15 µs after the

17. THERMAL PROPERTIES OF DEVICES

termination of the power pulse, which is sufficient for present studies. It was also found that for temperatures above 50°C increasing the lowlevel metering current (from 0.5 to 1.0 mA for 25- and 35-W transistors) did not appreciably change the slope of the calibration curve (forwardbiased base-emitter voltage, $V_{\rm RE}$, versus temperature).

Measured values of thermal resistance, R_{Θ} , and of d-c current gain, h_{FE} , were compared when they were plotted as functions of power dissipation for a given device. Abrupt changes in the curves for both types of measurements were observed at corresponding operating points. An example of such curves is shown in Fig. 4 for a power transistor rated at 35-W maximum dissipation with the case held at 25°C. In making these measurements the transistor under test was operated in a common-emitter configuration with a variable constant-current source in the base. The collector current was held constant by varying the base current as the collector-emitter voltage was varied. The h_{FE} was determined from the ratio of collector to base current. Thermal resistance was measured using V_{BE} as the temperature indicator by interrupting the collector voltage supply and simultaneously reducing the base drive to the required low-level metering current.

In a further measurement made using thermographic phosphors to detect hot-spot formation it was observed that hot spots developed at operating conditions corresponding to those at the appearance of the abrupt changes in R_{Θ} and $h_{\rm FE}$.

The curves of h_{FE} versus power illustrate the fact that until the point of abrupt change is reached the increase in h_{FE} is almost entirely due to temperature effects for the type of interdigitated transistor structure studied. Below the abrupt change, within the experimental error of the procedures, all the curves are approximately coincident. At a given abscissa the power dissipation, and hence the indicated temperature, is the same for all four curves although the voltages and currents vary widely from curve to curve. The change in h_{FE} with increasing power is therefore primarily a function of the temperature rather than of collector voltage or current. In the case of the 100- and 200-mA curves, the increase in the slope of h_{FE} versus power which appears somewhat before the abrupt change could be evidence of charge carrier multiplication. The plotted data also indicate the fact that the device is more susceptible to lateral thermal instabilities at the high voltage-low current dition [1].

If the observed correlation between $h_{\rm FE}$ and R_Θ exists for other transistor types, the use of $h_{\rm FE}$ measurements may provide both the user

D. Navon and E. A. Miller, "Thermal Instability in Power Transistor Structures," Solid-State Electronics 12, 69-78 (1969).

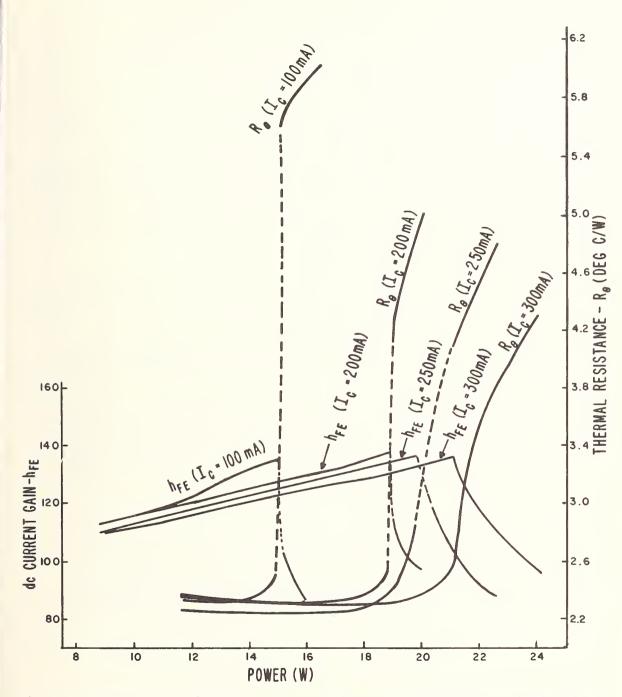


Fig. 4 D-c current gain and thermal resistance versus power dissipation for a 35-W silicon n-p-n transistor. All data points were within the specified maximum operating areas for the device.

and supplier of devices with a more convenient screen for thermal instabilities than the difficult and lengthy measurement of R_{Θ} . (S. Rubin, R. L. Gladhill, and F. F. Oettinger)

Plans: The literature search will continue. Writing of the review paper on the methods for measurement of thermal resistance and transient thermal response of semiconductor devices will begin.

Work will continue to determine the extent of the correlation between h_{FE} and R_{Θ} as measured by the V_{BE} technique on transistors with different geometry and structure. The results obtained will be correlated with thermographic studies of the uncased transistors (see Section 18). The h_{FE} test apparatus will be modified to provide automatic control of the collector current by providing a servo-loop to control the base drive so that the collector current will remain constant during the voltage sweep. This will allow an investigation of the repeatibility of the data for the same, and for differing, sweep speeds.

18. THERMOGRAPHIC MEASUREMENTS

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

Progress: A photometric microscope has been substituted for the camera for recording the light output of the thermographic phosphors. The equipment has been used to obtain qualitative results which indicate that temperature differences on transistor chips can be recorded. Quantitative results must await modification of the equipment to eliminate all extraneous visible light from the microscope system. (G. J. Rogers and F. F. Oettinger)

<u>Plans</u>: After modification of the photometric microscope system is completed, the equipment will be used to record temperature distributions on the surface of transistor chips. These results will be compared with the results obtained by electrical measurements using h_{FE} and V_{BE} as temperature-sensitive parameters. Studies of the spatial resolution and temperature resolution of the phosphors will be resumed.

19. MICROWAVE DIODE MEASUREMENTS

Objective: To study the problems and uncertainties associated with measurement of microwave mixer diode properties and to improve the methods of measurement for selected characteristics.

19. MICROWAVE DIODE MEASUREMENTS

Progress: Technical activity on this task has been deferred until the negotiations to determine the priority of specific problems to be studied are complete. Discussions have been held with representatives of the Electronic Industries Association, the Defense Electronic Supply Center, the IEEE Microwave Theory and Technique Group, the Defense Atomic Support Agency, the Air Force Weapons Laboratory, the Naval Electronic Systems Command, the Navy Applied Science Laboratory, the Air Force Material Laboratory, the Air Force Avionics Laboratory, and others to determine further the nature of the work which is to be undertaken in this area. (R. C. Powell)

Plans: Negotiations will be continued. Detailed studies are expected to begin late next quarter.

20. 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: The chamber for the thermal cycling and life-testing of detectors under space-simulated conditions has been assembled, baked out, and evacuated to a pressure of approximately 10⁻⁹ torr. Electrical wiring and installation of the unit, which will provide thermal cycling for the detectors between -20 and +50°C, remain to be completed. Equipment is being obtained for the system which will automatically monitor the leakage current and noise of detectors while in storage under bias. The principal problem anticipated with this system is the excessive noise from the elaborate switching network during the measurement of detector noise. Circular, finger-type spring contacts were designed and made for mounting Si(Li) detectors in the IMP-I radiation detector telescopes. Assistance was provided to the sponsor with the testing and evaluation of the detectors for the IMP-I telescopes.

The sensitive faces of two different types of 2-mm thick Si(Li) detectors, each with double concentric grooves which define an annular guard-ring detector surrounding the central circular detector, were scanned with a 1-mm diameter beam of light from a pulsed GaAs lightemitting diode. The objective was to determine the amount of cross-talk between the central detector and the annular detector. The response of

Supported by Goddard Space Flight Center, National Aeronautics and Space Administration. (NBS Project 4254429) Irradiations were carried out at Goddard Space Flight Center.

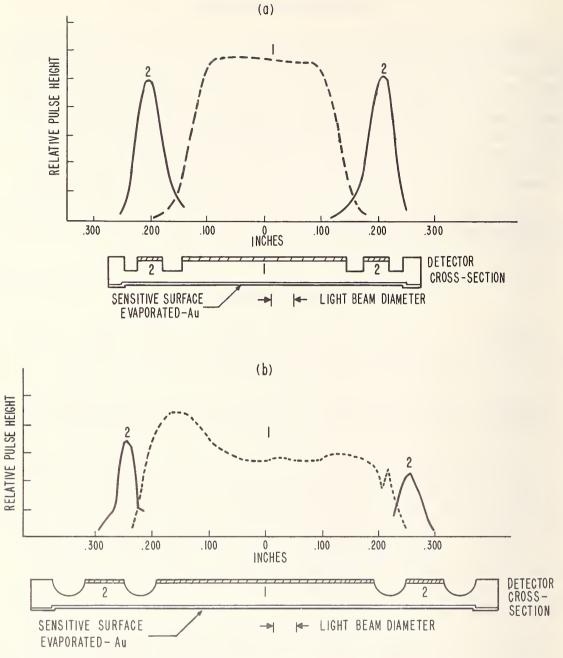


Fig. 5 Relative pulse height response of two Si(Li) detectors to a collimated beam from a pulsed GaAs light-emitting diode scanned along the diameter of the sensitive surface. Each detector has a central region (1) which is used to detect radiation incident normal to the sensitive surface and an annular region (2) which is used as an anti-coincidence detector for radiation entering from the side of the device. A large uniform response throughout region (1) and a small response from each region at the transition point is sought in these detectors.

20. SILICON NUCLEAR RADIATION DETECTORS

each region in terms of pulse height as a function of beam position along a diameter is shown in Fig. 6. A cross-sectional view of each detector (drawn to scale) is shown below each scan. The irregular response shown in Fig. 6 (b) could be caused by a nonuniformity in the layer of metallization applied on the sensitive surface, incomplete depletion of the silicon near the surface, or poor charge collection in the depletion region due to inhomogeneities in the crystal.

Efforts on the study of the correlation of carrier lifetime in silicon with the characteristics of detectors made from the same material have been concentrated in the area of diode recovery measurements of carrier lifetime (see Section 3). (B. H. Audet)

As part of the continuing investigation of the effects of radiation damage in silicon detectors by protons and electrons, a study has been made of the response of two 1-mm thick silicon, surface-barrier, totally depleted detectors to radiation damage by 1-MeV electrons, which have a range of 1.5 mm in silicon. A 20-mm² spot on the barrier contact of one detector and on the ohmic contact of the other detector were irradiated with fluences up to 10^{15} electrons/cm².

After irradiation of the barrier contact with 3×10^{13} electrons/cm² the detector leakage current, capacitance, and noise increased sharply at the normal operating voltage. Decreasing collection efficiency for alpha particles incident on the barrier contact was observed with increasing fluence. However, counting with alpha particles through the ohmic contact of this device was degraded only slightly for fluences up to 10^{14} electrons/cm².

When the ohmic contact of the other device was irradiated with electrons, a slow increase in detector leakage current, capacitance, and noise was observed with increasing fluence. The collection efficiency for alpha particles incident on either contact was changed very little for fluences to 10^{15} electrons/cm². These results are similar to previous observations in which the electrons did not penetrate through the entire detector; radiation is more detrimental to surface-barrier detectors when it is incident on the barrier contact. (Y. M. Liu and J. A. Coleman)

Plans: Establishment of the detector storage and monitoring system and the high-vacuum test chamber facilities will continue. A study of the noise contribution of the switching network will be emphasized. Additional detectors for the IMP-I telescopes will be tested. Evaluation of the resistivity, carrier lifetime, oxygen content, and lithium ion mobility in a 50-mm diameter, dislocation-free silicon crystal will be undertaken. Radiation damage by 400-keV electrons in surface-barrier detectors will be studied. The sensitivity of avalanche detectors to lowenergy (<10-keV) electrons will be evaluated.

Appendix A

JOINT PROGRAM STAFF Coordinator: J. C. French Consultant: C. P. Marsden

Semiconductor Characterization Section

Dr. W. M. Bullis, Chief

A. J. Baroody, Jr.	R. L. Mattis
D. L. Blackburn	Dr. W. E. Phillips
F. H. Brewer	Miss T. A. Poole ⁺
Mrs. E. C. Cohen*	Miss D. R. Ricks*
M. Cosman	Dr. J. L. Scales†
Dr. J. R. Ehrstein	H. A. Schafft
R. L. Gladhill	A. W. Stallings
G. G. Harman	G. N. Stenbakken
F. R. Kelly§	W. R. Thurber
H. K. Kessler	Miss R. E. Young ⁺
Mrs. K. O. Leedy	

Semiconductor Processing Section

Dr. J. A. Coleman, Chief

Miss D. A. Adamson§	E. I. Klein
B. H. Audet	J. Krawczyk
H. A. Briscoe	T. F. Leedy
R. D. Clayton§	Y. M. Liu
W. K. Croll	J. Oroshnik
R. T. Daltont	R. C. Schaevitz†
Mrs. S. A. Davis ⁺	Dr. A. H. Sher
H. E. Dyson*	L. M. Smith
H. A. Gitelson§	G. P. Spurlock
W. J. Keery	Miss E. D. Willis§

Electron Devices Section

J. C. French, Chief

Mrs. C. F. Bolton ⁺	R.	C. Powell*
Mrs. R. Y. Cowan	G.	J. Rogers
Miss B. S. Hope ⁺	S.	Rubin
F. F. Oettinger	М.	Sigman
M. K. Phillips	L.	R. Williams

^{*} Part Time

[†] Guest Worker

[§] Summer

⁺ Secretary

Appendix B

COMMITTEE ACTIVITIES

ASTM Committee F-1; Materials for Electron Devices and Microelectronics

- F. H. Brewer, Task Force on Resistivity
- W. M. Bullis, Editor, Subcommittee IV, Semiconductor Crystals
- J. A. Coleman, Secretary, Subcommittee V, Semiconductor Processing Materials
- J. R. Ehrstein, Task Forces on Epitaxial Resistivity and Epitaxial Thickness
- J. C. French, Chairman, Subcommittee VIII, Editorial
- T. F. Leedy, Task Force on Photomasking
- J. Oroshnik, Task Forces on Thin Films, Thick Films, and Photomasking
- W. E. Phillips, Task Forces on Crystal Perfection, Encapsulation, Thin Films, and Thick Films
- A. H. Sher, Task Force on Germanium
- M. Sigman, Editor, Subcommittee V, Semiconductor Processing Materials
- W. R. Thurber, Task Forces on Impurities in Semiconductors and Germanium

Electronic Industries Association:

- MED 32, Active Digital Circuits: F. F. Oettinger, TG 32.5, Thermal Resistance and Test Methods
- MED 41, Physical Characterization Requirements: F. F. Oettinger, TG 41.6, Thermal Considerations

Joint Electron Device Engineering Council (EIA-NEMA):

- JS-3, UHF and Microwave Diodes: R. C. Powell, Microwave Diode Specification Problems
- JS-6, Power Transistors: H. A. Schafft, Consultant on Second Breakdown Specifications
- JS-9, Low Power Transistors: F. F. Oettinger, Thermal Resistance Measurements
- IS-14, Thyristors: F. F. Oettinger, Thermal Resistance of SCR's

IEEE:

- Nuclear Science Group: J. A. Coleman; Administrative Committee; Nuclear Instruments and Detectors Committee; Editorial Board, *Transactions on Nuclear Science*; Chairman, 1970 Nuclear Science Symposium
- Magnetics Group: S. Rubin; Chairman, Galvanomagnetic Standards Subcommittee
- IEC TC47, Semiconductor Devices and Integrated Circuits: F. F. Oettinger, U. S. Experts Advisory Committee S. Rubin, Technical Expert, Galvanomagnetic Devices

NAS-NRC Semiconductor Detector Panel: J. A. Coleman

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. <u>Hall effect samples</u> (H. K. Kessler) Ohmic contact dots were applied to epitaxial GaAs Hall effect samples for the Harry Diamond Laboratories.
- 2. <u>Radiation detectors</u> (B. H. Audet and A. H. Sher) A 3-mm thick Al-p-Si surface-barrier silicon detector which operated successfully at liquid helium temperature was made for the Nuclear Spectroscopy Section. Assistance was provided to the Center for Radiation Research in developing and testing special Ge(Li) gamma-ray detectors.
- 3. <u>Quartz and glass fabrication</u> (E. I. Klein) Various glass-to-metal seals were prepared for several groups. A programmed annealing of special quartz specimens was carried out for the Metrology Division.

Appendix D

JOINT PROGRAM PUBLICATIONS

Prior Reports:

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, July 1 to September 30, 1968," NBS Tech. Note 472, December, 1968.

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, October 1 to December 31, 1968," NBS Tech. Note 475, February, 1969.

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, January 1 to March 31, 1969," NBS Tech. Note 488, July, 1969.

Current Publications:

A. H. Sher, "Lithium Ion Drift Mobility in Germanium" J. Appl. Phys. <u>40</u>, 2600-2607 (May, 1969).

G. G. Harman, "Topological Features of Hot-Carrier Induced Anisotropic Breakdown on Silicon Diode Surfaces," J. Res. Natl. Bur. Standards, 73A, 321-331, (May-June, 1969).

W. M. Bullis and R. I. Scace,[†] "Measurement Standards for Integrated Circuit Processing," to appear in *Proc. IEEE*, Special Issue on Materials and Materials Problems in Microelectronics, September, 1969.

A. H. Sher and W. J. Keery, "Variation of the Effective Fano Factor in a Ge(Li) Detector," accepted for presentation at the Nuclear Science Symposium, San Francisco, October, 1969.

General Electric Company, Auburn, N. Y. 13021 (Chairman, ASTM Committee F-1.)

THE NATIONAL ECONOMIC GOAL Sustained maximum growth in a free market economy, without inflation, under conditions of full employment and equal opportunity

THE DEPARTMENT OF COMMERCE

The historic mission of the Department is "to foster, promote and develop the foreign and domestic commerce'' of the United States. This has evolved, as a result of legislative and administrative additions, to encompass broadly the responsibility to foster, serve and promote the nation's economic development and technological advancement. The Department seeks to fulfill this mission through these activities:



MISSION AND **FUNCTIONS OF THE** DEPARTMENT OF COMMERCE

"to foster, serve and promote the nation's economic development and technological advancement"

Participating with other government agencies in the creation of national policy, through the President's Cabinet and its subdivisions.

- Cabinet Committee
 Office of Field on Economic Policy
- Urban Affairs Council
- Environmental **Quality Council**

Promoting progressive Assisting states, business policies and communities and growth.

- Business and **Defense Services** Administration
- Services

individuals toward economic progress.

- Economic Development Administration
- Regional Planning Commissions
- Office of Minority **Business Enterprise**

Strengthening the international economic position of the United States.

- Bureau of International Commerce
- Office of Foreign Commercial Services
- Office of Foreign Direct Investments
- United States Travel Service
- Maritime Administration

Assuring effective use and growth of the and disseminating nation's scientific and technical resources.

- Environmental Science Services Administration
- Patent Office
- National Bureau of **Standards**
- Office of Telecommunications
- Office of State **Technical Services**

Acquiring, analyzing information concern ing the nation and the economy to help achieve increased social and economic benefit.

- Bureau of the Census
- Office of Business Economics

NOTE: This schematic is neither an organization chart nor a program outline for budget purposes. It is a general statement of the Department's mission in relation to the national goal of economic development.

NBS TECHNICAL PUBLICATIONS

PERIODICALS

JOURNAL OF RESEARCH reports National Bureau of Standards research and development in physics, mathematics, chemistry, and engineering. Comprehensive scientific papers give complete details of the work, including laboratory data, experimental procedures, and theoretical and mathematical analyses. Illustrated with photographs, drawings, and charts.

Published in three sections, available separately:

Physics and Chemistry

Papers of interest primarily to scientists working in these fields. This section covers a broad range of physical and chemical research, with major emphasis on standards of physical measurement, fundamental constants, and properties of matter. Issued six times a year. Annual subscription: Domestic, \$9.50; foreign, \$11.75*.

• Mathematical Sciences

Studies and compilations designed mainly for the mathematician and theoretical physicist. Topics in mathematical statistics, theory of experiment design, numerical analysis, theoretical physics and chemistry, logical design and programming of computers and computer systems. Short numerical tables. Issued quarterly. Annual subscription: Domestic, \$5.00; foreign, \$6.25*.

• Engineering and Instrumentation

Reporting results of interest chiefly to the engineer and the applied scientist. This section includes many of the new developments in instrumentation resulting from the Bureau's work in physical measurement, data processing, and development of test methods. It will also cover some of the work in acoustics, applied mechanics, building research, and cryogenic engineering. Issued quarterly. Annual subscription: Domestic, \$5.00; foreign, \$6.25*.

TECHNICAL NEWS BULLETIN

The best single source of information concerning the Bureau's research, developmental, cooperative and publication activities, this monthly publication is designed for the industry-oriented individual whose daily work involves intimate contact with science and technology—for engineers, chemists, physicists, research managers, product-development managers, and company executives. Annual subscription: Domestic, \$3.00; foreign, \$4.00*.

* Difference in price is due to extra cost of foreign meiling.

Order NBS publications from:

NONPERIODICALS

Applied Mathematics Series. Mathematical tables, manuals, and studies.

Building Science Series. Research results, test methods, and performance criteria of building materials, components, systems, and structures.

Handbooks. Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications. Proceedings of NBS conferences, bibliographies, annual reports, wall charts, pamphlets, etc.

Monographs. Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

National Standard Reference Data Series. NSRDS provides quantitive data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated.

Product Standards. Provide requirements for sizes, types, quality and methods for testing various industrial products. These standards are developed cooperatively with interested Government and industry groups and provide the basis for common understanding of product characteristics for both buyers and sellers. Their use is voluntary.

Technical Notes. This series consists of communications and reports (covering both other agency and NBS-sponsored work) of limited or transitory interest.

Federal Information Processing Standards Publications. This series is the official publication within the Federal Government for information on standards adopted and promulgated under the Public Law 89–306, and Bureau of the Budget Circular A–86 entitled, Standardization of Data Elements and Codes in Data Systems.

CLEARINGHOUSE

The Clearinghouse for Federal Scientific and Technical Information, operated by NBS, supplies unclassified information related to Government-generated science and technology in defense, space, atomic energy, and other national programs. For further information on Clearinghouse services, write:

> Clearinghouse U.S. Department of Commerce Springfield, Virginia 22151

Superintendent of Documents Government Printing Office Washington, D.C. 20402

U.S. DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20230

OFFICIAL BUSINESS



POSTAGE AND FEES PAID U.S. DEPARTMENT OF COMMERCE