NBS TECHNICAL NOTE

472

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

Quarterly Report
July 1 to September 30, 1968

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards[1] 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 three broad program areas and provides central national services in a fourth. 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, and the Center for Radiation Research.

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 the 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 Standard Reference Data and a group of divisions organized by the following areas of science and engineering:

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

THE INSTITUTE FOR MATERIALS RESEARCH conducts materials research leading to methods, standards of measurement, and data needed by industry, commerce, educational institutions, and government. The Institute also provides advisory and research services to other government agencies. The Institute consists of an Office of Standard Reference Materials and a group of divisions organized by the following areas of materials research:

Analytical Chemistry—Polymers—Metallurgy — Inorganic Materials — Physical Chemistry.

THE INSTITUTE FOR APPLIED TECHNOLOGY provides for the creation of appropriate opportunities for the use and application of technology within the Federal Government and within the civilian sector of American industry. The primary functions of the Institute may be broadly classified as programs relating to technological measurements and standards and techniques for the transfer of technology. The Institute consists of a Clearinghouse for Scientific and Technical Information,[3] a Center for Computer Sciences and Technology, and a group of technical divisions and offices organized by the following fields of technology:

Building Research—Electronic Instrumentation — Technical Analysis — Product Evaluation—Invention and Innovation— Weights and Measures — Engineering Standards—Vehicle Systems Research.

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 for Radiation Research consists of the following divisions:

Reactor Radiation—Linac Radiation—Applied Radiation—Nuclear Radiation.

---

[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D. C. 20234.

[2] Located at Boulder, Colorado 80302.

[3] Located at 5285 Port Royal Road, Springfield, Virginia 22151.

UNITED STATES DEPARTMENT OF COMMERCE
C. R. Smith, Secretary
NATIONAL BUREAU OF STANDARDS • A. V. Astin, Director

NBS TECHNICAL NOTE 472

ISSUED DECEMBER 1968

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

Quarterly Report
July 1 to September 30, 1968

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. Naval Ammunition Depot
and
National Aeronautics and Space Administration

For sale by the Superintendent of Documents, Government Printing Office
Washington, D.C., 20402 - Price 50 cents

CONTENTS

---

† Related Projects

ABSTRACT

This quarterly progress report describes NBS activities relating to: measurement of resistivity, carrier lifetime, inhomogeneities, and Hall effect in semiconductor crystals; study of infrared measurement methods, properties of deep-lying impurities (in InSb), and high field effects; establishment of a processing facility; evaluation of wire bonds; review of NASA measurement methods; and measurement of second breakdown in transistors, thermal properties of devices, and noise in microwave diodes. Projects on silicon nuclear radiation detectors and specification of germanium are also described. Supplementary data concerning staff, committee activities, technical and information services, and publications are included as appendixes. A list of ASTM Standards relevant to integrated circuit processing is also included.

Key Words: carrier lifetime; electrical properties; germanium; methods of measurement; microelectronics; resistivity; semiconductor devices; semiconductor materials; semiconductor process control; silicon; thermal properties; wire bonds.

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

Quarterly Report
July 1 to September 30, 1968

1. INTRODUCTION

Through long contact with the semiconductor industry, the Electronic Technology Division of the National Bureau of Standards has gathered substantial evidence of the need for improved measurement methods and standards for semiconductor materials characterization, for process control, and for device characterization. These methods and standards are needed for satisfactory buyer-seller exchanges, for economy in government purchasing, and for improved product uniformity, interchangeability, and reliability. The need is recognized both by industry and by government. NBS as a national center for measurement development, without vendor or user bias, and already competent in the field, has repeatedly been singled out as the uniquely appropriate organization to provide assistance in the resolution of these problems.

NBS has therefore undertaken and partially funded a Joint Program on Methods of Measurement for Semiconductor Materials, Process Control, and Devices. Added financial support has been supplied by concerned government agencies. The Program includes extensive participation in and cooperation from industrial standards organizations. It is cooperative in nature because:

(1) guidance is required in establishing priorities for the work of the Program, and
(2) close contact must be maintained with the prospective users of the methods and standards in both industry and government in order to insure that the methods under development are useful and will be accepted and applied.

Objectives of the Joint Program are enhancement of 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, such as uniformity of response to radiation effects.

These objectives are being approached through:

(1) evaluation of the technical adequacy of relevant measurement methods developed in industrial or other agency organizations and recommendation of modifications to

these methods where such modifications are appropriate,

(2) development of specific methods selected in accordance with the guidance received from participating groups,

(3) assistance to standardizing groups in industry and government agencies as is appropriate to encourage the rapid development, publication, and implementation of improved measurement method standards, and

(4) consultation with sponsors, other government agencies, manufacturers, and users regarding measurement requirements and applications.

The concepts and methodology of the Joint Program have evolved from several projects that have been carried out in the Division during the past several years. In these projects it was clearly established that one of the key elements in the development of realistic, useful standards is cooperation and liaison with both manufacturers and users. This is accomplished through participation in technical committee activities of organizations such as the American Society for Testing and Materials (ASTM) and the Electronic Industries Association (EIA) and through personal contacts with and visits to industrial plants and government laboratories. These contacts also serve to provide Program staff members with a broader view of the problems faced by industry and help to insure that the output of the Program will be useable in the field.

It was also established that there is a critical need for analysis and review of existing measurement methods. Hence, one of the first activities undertaken in connection with the detailed study of a measurement is the collection and collation of available information concerning both commonly used and alternate methods for making this measurement, evidence of the relevance of the measurement to device characteristics, and the level of precision required. In addition to providing a foundation for future experimental work, published bibliographies and summary review papers which result from this activity are often widely distributed and prove to be extremely valuable to both sponsors and industry.

The Joint Program was established formally during the latter part of fiscal year 1968 with support from NBS [1], the National Aeronautics and Space Administration [2], and the Defense Atomic Support Agency [3]. During the present quarter, the U. S. Naval Ammunition Depot at Crane, Indiana, also began support of the Program [4]. The concern of certain sponsors with specific parts of the Program is taken into consideration in program planning. It should be emphasized, however, that all sponsors subscribe to the need for the entire basic program for improvement of measurement methods for semiconductor materials, process control, and devices.

A number of tasks have been identified as parts of the Program. These tasks were selected during an initial program review in April 1968. Some are continuations of projects already in progress; others have been started since the inception of the Program; still others are for future implementation. Because of the cooperative nature of the Program, there is not a one-to-one correspondence between these tasks and the projects by which the Program is supported.

Quarterly progress reports for the Program will be subdivided according to these tasks. Sections 2 through 9 deal with methods of measurement for materials; sections 10 through 12, with methods of measurement for process control; and sections 13 through 16, with methods of measurement for devices. In this first report, which covers the period July through September 1968, objectives and background information are included together with a report of activity during the reporting period and an indication of the direction the work will take during the next reporting period. In addition to tasks sponsored under the Joint Program, this report contains descriptions of activity in 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.

---

1. Through RTS (Research and Technical Services) Projects 4251120, 4251123, 4251126, 4252128, 4254111, 4254112, and 4254115.
2. Through Order ER-11897, Electronics Research Center. (NBS Project 4259523)
3. Through Inter Agency Cost Reimbursement Order 814-68. (NBS Project 4259522)
4. Through Cost Reimbursement Order P09-0016. (NBS Project 4259533)

2. RESISTIVITY

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

Background: Resistivity is the most widely used parameter in the exchange of silicon across the interface between crystal grower and device manufacturer as well as in the design and production control of semiconductor devices. This parameter is important because of its relation to the density of free carriers in semiconducting materials and because of the fact that the electrical characteristics of semiconductor devices depend in a critical way on the free carrier density in the various regions of the device structure.

In 1960, NBS was asked by ASTM Committee F-1 on Materials for Electron Devices and Microelectronics to investigate the problems associated with the measurement of resistivity of bulk silicon wafers. This work [1] led to the formulation of a standard method for measuring resistivity of silicon slices by a four-probe method which was developed in cooperation with Committee F-1 [2]. With the use of this method it is now possible to make the measurement with a precision of ±2 per cent [3] in the resistivity range between 0.005 and 120 Ω-cm.

Although this represented a substantial increase in the quality in the measurement there remains considerable need to extend the range of applicability of the method to both higher and lower resistivities of bulk wafers as well as to resistivity measurements on specialized structures such as epitaxial and diffused layers. The present phase of the project is concerned with these extensions. In addition the expanded scope includes other methods for making resistivity measurements on epitaxial and diffused layers.

Accomplishments during reporting period: A round-robin experiment based on the standard four-probe method is being conducted in cooperation with Committee F-1 to determine the precision which can be expected when measuring bulk silicon wafer resistivity as low as 0.001 Ω-cm and as high as 1000 Ω-cm. Preliminary returns indicate that the resistivity range can be extended with no loss in precision. (F. H. Brewer)

A series of experiments designed to determine the effect of variation in measuring current, probe force, specimen surface condition, and sheet resistance on the measurement of resistivity by the standard four-probe method has been started. These factors are most significant when four-probe measurements are made on epitaxial and diffused layers. Initial measurements, concerned with variations of measuring current, were made on bulk silicon wafers previously used in round-robin experiments.

(F. H. Brewer and W. M. Bullis)

Initial measurements were made on bulk silicon wafers in order to obtain a better understanding of the various parameters which significantly influence the measurement of resistivity of epitaxial layers by the three-probe voltage breakdown method.

A number of observations were noted. A distinct breakdown voltage could be observed on many specimens only after the contact had been electrically formed properly. Usually, forming required that currents in excess of the breakdown current be passed through this contact for a period of time. Once a contact was properly formed, a repeatable breakdown curve could be obtained regardless of the time interval between any successive readings. It did not appear to be neccessary to form n-type specimens to the same extent as p-type specimens.

Preliminary results indicated that surface preparation and cleaning did not significantly affect the value of the breakdown voltage; however, large variations were observed in breakdown current. Studies of the effect of various duty cycles were also initiated. Tentative indications were that less forming time is needed to reach a stable breakdown if the width of the voltage pulse is reduced. (J. R. Ehrstein)

Plans for next quarter: It is hoped that all participants in the round-robin experiment will have completed their measurements and that the analysis will be started. Experimental work on the effects of varying measuring current and probe force will be continued.

The dependence of voltage breakdown on the duty cycle used will be studied in order that the type of generating equipment to be used for further work can be determined. The dependence of voltage breakdown on the forming process will be investigated further in order to determine what forming, if any, gives the most reproducible resistivity measurements.

---

1. The results of this project are summarized in a report prepared for the NASA Electronics Research Center, sponsors of the concluding phase: W. Murray Bullis, "Standard Measurements of the Resistivity of Silicon by the Four-Probe Method," NASA-CR-86032, N68-18067, December 29, 1967.
2. "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.
3. This precision figure is three times the average relative sample standard deviation obtained in a recent round-robin experiment involving 5 laboratories and 8 specimens.

Fig. 1 Test Set-up for Precision Measurements of Resistivity of Silicon Wafers by the Four-Probe Method.

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.

Background: Minority carrier lifetime is a basic parameter for the specification of semiconducting materials for transistors, diodes, and similar devices. Photoconductive decay measurements are made routinely on many crystals used in the fabrication of these devices. Although this measurement suffers both from lack of precision and from limitations in the range of applicability, it is the only method for determination of carrier lifetime which has been accepted as standard [1, 2]. In the initial phase of this project [3] begun in December 1966, preliminary but detailed studies of the photoconductive decay method were accompanied by the establishment of facilities for making measurements by the photomagnetoelectric method, the diode recovery time method, and the surface photovoltage method [4]. In addition, a critical survey of the various methods used to measure carrier lifetime reported in the literature was compiled and an annotated bibliography was prepared [5]. At the present time, work on the four methods for making lifetime measurements is continuing.

Accomplishments during reporting period: Detailed studies of the photoconductive decay method were continued. The principal efforts were concerned with the determination of bulk lifetime from a measured value of filament lifetime in rectangular silicon bars. Measurements were made on a bar sample whose size was successively reduced. Bulk lifetime was calculated from the filament lifetime by means of a formula which assumes an infinite value of surface recombination velocity. In order to obtain a consistent value of bulk lifetime it was necessary to assume a mobility for the minority carrier which was substantially larger than that which is normally obtained. The reason for this discrepancy is not yet understood. A cryostat was set up in order to enable measurements of photoconductive decay lifetime to be made over the temperature range between 77 and 350 K. An initial study of the dependence of lifetime on temperature in the range -10 to 50°C showed that variations from 1 to 5 per cent per degree could be expected. (R. L. Mattis)

Increased emphasis was placed on the measurement of carrier lifetime by diode recovery methods. The instrumentation was modified to permit greater flexibility in measuring current plateau and current decay characteristics. In addition new circuitry to enable voltage decay observations to be made was constructed and is being tested.
(A. J. Baroody)

The vacuum chamber and specimen holder of the steady-state photomagnetoelectric-photoconductive (PME-PC) equipment have been modified to permit lower temperatures to be achieved. Serious difficulties have been encountered in using this equipment on silicon wafers. Since minority carrier diffusion length can be measured directly by the surface photovoltage technique, the emphasis on PME-PC measurements for silicon is no longer necessary. The equipment, however, is being used in connection with the study of deep-lying impurity levels in indium antimonide (see Section 7). (W. E. Phillips and A. W. Stallings)

Plans for next quarter: The discrepancy in the bulk lifetime calculations from photoconductive decay filament lifetime measurements will be further investigated. Work on lifetime interpretation for various recombination models will be emphasized. PME-PC measurements on indium antimonide will be completed. Diode recovery lifetime measurements will be made on several types of diodes including lithium-drifted p-i-n devices.

---

1. "Method for Measuring the Minority-Carrier Lifetime in Bulk Germanium and Silicon" (ASTM Designation: F28-66), *1968 Book of ASTM Standards*, Part 8, November 1968.
2. "Measurement of Minority-Carrier Lifetime in Germanium and Silicon by the Method of Photoconductive Decay" (IEEE Standard 225), *Proc. IRE*, 49, 1292-1299 (1961).
3. Supported by U. S. Air Force Materials Laboratory under Delivery Order F 33615-67-M-5007.
4. R. L. Mattis, W. E. Phillips, and W. M. Bullis, "Measurement and Interpretation of Carrier Lifetime in Silicon and Germanium," AFML-TR-68-81, July 1968.
5. W. M. Bullis, "Measurement of Carrier Lifetime in Semiconductors--An Annotated Bibliography Covering the Period 1949-1967," NBS Tech. Note 465, June 1968.

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.

Background: Variations in the resistivity of a silicon wafer can cause significant variations in device characteristics. This problem is aggravated in large scale integrated circuits and large area power devices where the resulting nonuniform current distributions can be unnoticed until they cause device failure during operation. Although it is possible to measure resistivity variations by means of a point-to-point measurement with conventional four-probe or spreading resistance techniques, a scanning technique is more suitable in a large number of applications because it does not require the placement of probes on the region of interest. Hence a photovoltaic method for measuring resistivity variations which was introduced by Oroshnik and Many [1] was selected for detailed study.

In the first phase of this project [2] which was begun in November 1966 it was demonstrated that it was feasible to use this method to obtain not only precise measurements of resistivity profiles along germanium and silicon bar-shaped specimens but also qualitative profiles on curcular wafers [3]. As another part of this phase of the project a comprehensive bibliography on methods for the measurement of resistivity variations and other inhomogeneities in semiconductors was assembled and published [4]. The present phase of the project is directed toward further refinement in the procedures for making the photovoltaic measurement, extension of the method to include quantitive measurements on circular wafers, and improvement in the means of recording experimental data and computing the resistivity profile.

Accomplishments during reporting period: The causes for occasional nonreproducibility observed in earlier photovoltaic resistivity measurements on rectangular bars of germanium and silicon were investigated in detail. It was found necessary to: (1) replace the tungsten lamp used as the light source for the light probe prior to the time when significant but sporadic variations in the light output occur; (2) improve the light and air shields to reduce exposure of the specimen to scattered light and air currents; (3) use a thermal shunt across the specimen to minimize thermal gradients while, at the same time, keep the electrical capacitance in parallel with the specimen small so the time required for measurement is not increased; and (4) make measurements on specimens with non-specular surfaces. Tests were also made to insure that the light probe did not introduce any significant thermal gradients within the specimen and that the range of light intensities used in the experiment injected a density of carriers which was much less than the equilibrium concentration of majority carriers in the specimen. The latter condition must be met if small-signal theory is to be used in the analysis of the measurements. (H. A. Schafft and D. L. Blackburn)

Plans for next quarter: Further improvements of the measurement techniques will be investigated. The information obtained will be applied in measurements on p-type germanium and n- and p-type silicon bar specimens. In preparation for quantitative measurements on circular wafers, preliminary measurements will be started on rectangular-shaped specimens where the light probe will not extend completely across the width of the specimen.

---

1. J. Oroshnik and A. Many, "Quantitative Photovoltaic Evaluation of the Resistivity Homogeneity of Germanium Single Crystals," *Solid-State Electronics*, 1, 46-53, (1960).
2. Supported by Rome Air Development Center under Contract F 30602-67-C-0105.
3. H. A. Schafft, "Photovoltaic Method for the Measurement of Resistivity [Profiles] in Germanium and Silicon," RADC-TR-68-64, June 1968.
4. H. A. Schafft and S. G. Needham, "A Bibliography on Methods for the Measurement of Inhomogeneities in Semiconductors (1953-1967)," NBS Tech. Note 445, May 1968.

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 germanium and silicon.

Background: An increase in the conductivity of a specimen occurs when radiation incident on the specimen is of sufficient energy to ionize impurity atoms with energy states located in the forbidden gap. From measurements of impurity photoconductivity as a function of wavelength in the appropriate region it is possible to identify energy levels associated with the impurities in the specimen. This project was established this quarter in order to determine whether the photo-ionization of impurities at densities as low as 10^{13} atoms/cm^3 can be detected and whether the density of impurity atoms can be determined.

Accomplishments this reporting period: A monochrometer system which covers the wavelength range between 2 and 6.4 μm by means of interchangeable gratings was set up. The lock-in amplifier and x-y recorder which will be used to detect and record the signal were also set up.
(W. R. Thurber)

Plans for next quarter: A liquid helium cryostat will be designed and fabricated. In addition the wavelength drive of the monochrometer will be modified so that it also drives the x axis of the x-y recorder. Additional gratings to extend the wavelength range to 40 μm will be obtained. Preliminary measurements on germanium specimens will be initiated when the equipment is assembled.

6. HALL EFFECT

Objective: To establish a facility for making measurements of Hall coefficient as a function of temperature in the range between 4.2 and 350 K and to improve methods for collecting and interpreting Hall effect data with automatic data acquisition systems.

Background: Measurements of the Hall effect are widely used in the the study of semiconducting materials. During the past year, a cooperative program carried out with the NBS Computer Center demonstrated the feasibility of using a time-shared computer system in conjunction with the Hall effect experiment [1]. Recently a new commercial data acquisition system was purchased and is being installed.

Assistance has been given to ASTM Committee F-1 in the formulation of a standard method for making Hall effect measurements on semiconductor crystals [2]. Additional assistance is being given the committee in conjunction with the interpretation of Hall effect measurements.

Accomplishments during reporting period: Circuitry to reverse and turn on and off both the magnetic field and the specimen current at the required time during the data-taking cycle was developed for the new Hall effect data acquisition system. A test run with a previously measured specimen confirmed that the circuits functioned properly. Programs were developed to process the punched card output on the NBS computer. A circuit with 48 potentiometers and a 48-position stepping switch was designed to permit automatic control of specimen temperature by this system.

Information concerning the relationship between Hall coefficient and carrier concentration in extrinsic semiconductors is being assembled for publication in conjunction with the activities of the Optoelectronics Task Force of ASTM Committee F-1. The materials of interest are germanium, silicon, gallium arsenide, and indium antimonide,

(W. R. Thurber, J. L. Scales, and W. M. Bullis)

Plans for Quarter: The temperature control system for the Hall effect experiment will be built and tested. Programs will be written to enable the data to be plotted by computer. Work on the relationship between Hall coefficient and carrier concentration will be continued with emphasis on gallium arsenide. The report describing the coupling between the Hall experiment and the time-shared computer system will be written in cooperation with Computer Center personnel.

---

1. W. M. Bullis, A. L. Koenig, T. N. Pyke, Jr., W. R. Thurber, and F. H. Ulmer, "Use of a Time-Shared Computer System to Control a Hall Effect Experiment," NBS Tech. Note (in preparation).
2. "Method for Measuring Hall Mobility in Extrinsic Semiconductor Single Crystals" (ASTM Designation: F76-68), *1968 Book of ASTM Standards*, Part 8, November 1968.

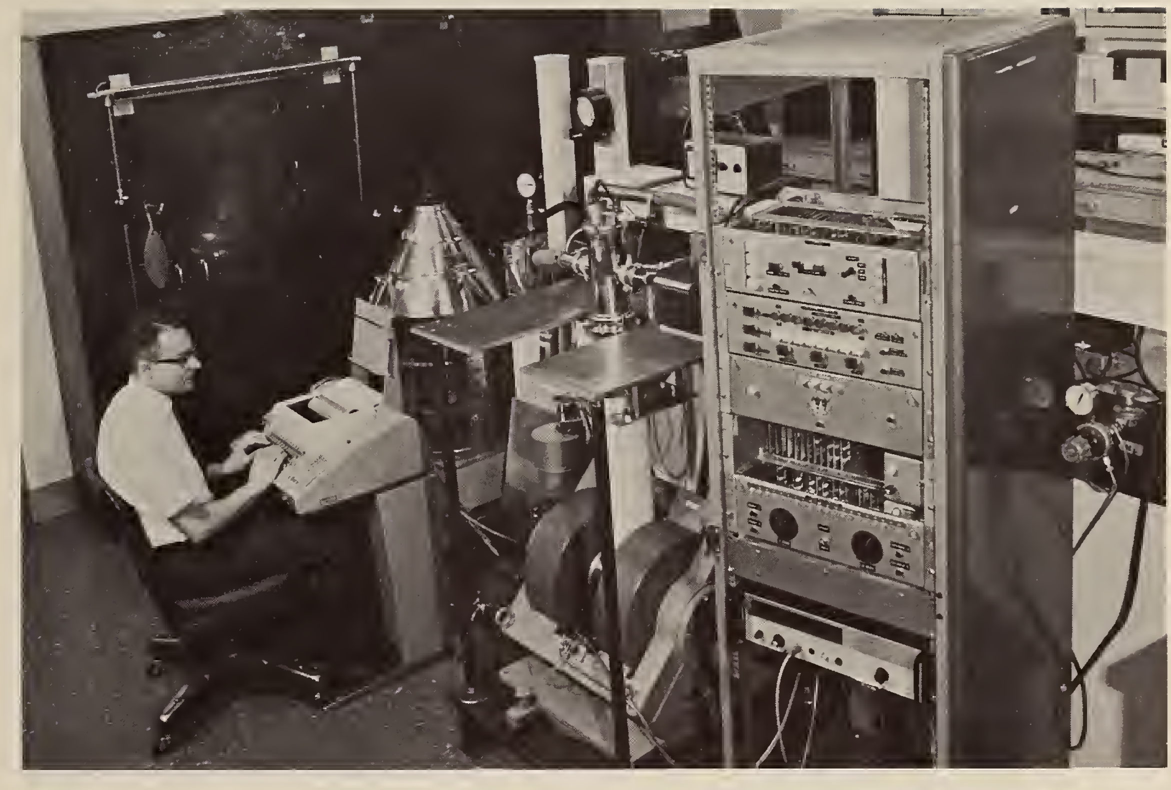

Fig. 2. Computer-controlled System for Hall Measurements in the Temperature Range Between 4.2 and 350 K.

7. DEEP-LEVEL STUDIES

Objective: To attempt to determine the origin of the deep-lying impurity centers in high-resistivity indium antimonide.

Background: A number of investigators have found that high resistivity indium antimonide contains approximately 10^{14} unidentified deep-lying impurity centers per cubic centimeter. Several models have been proposed to explain the properties of high-resistivity indium antimonide and hence considerable confusion exists in the literature concerning the nature of these centers. Prior to this quarter a number of models had been analyzed, some new Hall effect data had been taken, and initial lifetime measurements had been made.

Progress during reporting period: Experiments were undertaken to determine the interaction of lithium with the deep-lying impurity centers. Lithium was vacuum-deposited on the wafer surface after which the wafer was sealed in an argon-filled ampoule and heated for 5 hours at 250°C, in order to diffuse the lithium in the wafer. Preliminary results have shown that p-type wafers convert to n-type immediately after the lithium diffusion. However precipitation occurs and continues for several days at room temperature so eventually the wafers recover their p-type character. Hall measurements indicate that the wafer was more p-type after precipitation than it was initially. Hence the lithium seemed to act in two ways: in large quantities it was a simple donor impurity (probably an interstitial) but in small quantities it neutralized residual donors that were already present in the material. This neutralization made the diffused material more p-type than the original material.
(J. L. Scales)

Plans for next quarter: Lithium will be diffused into other p-type wafers. Photomagnetoelectric effect measurements will be resumed on completion of the modifications of the cryostat (see Section 3) so that additional lifetime data can be obtained. Further work will be done on the analysis of various models. In addition, various phases of the investigation will be written up.

8. HIGH FIELD EFFECTS

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

Background: Anisotropic tracking effects have been observed in silicon structures under high field and high current conditions as part of a general investigation into the hot minority carrier transport problem. In addition oscillations have been observed during portions of the current and voltage pulses.

Progress during reporting period: The high voltage pulser and cryostat mounts were significantly improved to clean up the pulse shapes so that the current and voltage waveforms could be observed unambiguously. Conductivity data were taken on 8 specimens at 300 and 77 K. Conduction anisotropy was exhibited clearly by a very low carrier transport in junction specimens having an orientation of 20° from a <100> direction on a $\{100\}$ surface.

Oscillations were observed in the current pulse at 77 K in samples with the current direction oriented 20 to 25° from a <100> direction on a $\{100\}$ surface. These oscillations reached a maximum on 22° specimens (approximately 10 per cent efficiency) with a frequency of about 2 MHz. Specimens oriented 20 and 25° from a <100> direction also oscillate but with higher frequency (up to 30 MHz) and lower efficiency (1 to 2 per cent). A magnetic flux density of about 15 mT (150 gauss) lowers the current threshold for oscillation. The reasons for this are not yet understood. (G. G. Harman and K. O. Leedy)

Plans for next quarter: Investigations will be made on additional silicon specimens with various orientations, length, and conductivity type.

9. 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.

Background: For a number of years, uncontrolled variations in the quality of germanium intended for fabrication into gamma-ray detectors have occurred for reasons which, even now, are not understood. Crystals are presently selected on the basis of room temperature resistivity, photoconductive decay lifetime, and etch-pit density. Use of these parameters does not always enable one to discriminate between crystals suitable for detector fabrication and crystals which are not. This project, which was begun early in 1967, involves material evaluation, device fabrication, and device characterization utilizing "good" and "bad" crystals [1]. To protect the sources of bad crystals, a code number has been assigned to each crystal used in the study.

Progress during reporting period: Values of lithium mobility were obtained for samples NBS-13, -617, and -83 at 23.8, 36.2, and 46.2°C. Additional samples of NBS-13 were also run at 61.2°C. The mobility values obtained for these samples were consistently higher than predicted by the extrapolation of the curve of diffusion constant against reciprocal absolute temperature derived by Fuller and Severiens [2]. A description of this aspect of the mobility results is being prepared for publication in the immediate future.

A preliminary effort was made to observe the effect of surface condition of the drifting diode on the capacitance against time measurements used in determining lithium drift mobility. An apparatus was assembled which permitted enveloping the drifting diode in various gaseous mixtures, e.g., nitrogen and water vapor, nitrogen and alcohol vapor, etc. Preliminary analysis of results indicated that such treatment does affect the capacitance measurement; however, the surface effects noted were not identical to the effects observed in diodes after long lithium drift times.

Additionally, the method of Pell [3] for determining lithium mobility in silicon at very short drift times (<2 hours) from the slope of the $1/C^3$ (where C is the capacitance) against time curve was studied for use on germanium. Preliminary results indicate that this method is in agreement with the method which uses the slope of the $1/C^2$ against time curve for long drift times on certain types of samples. It also appears to be easier to obtain a unique value of mobility from the slope of $1/C^3$ against time curves than from the slope of $1/C^2$ plots.

(R. D. Clayton, W. J. Keery, and A. H. Sher)

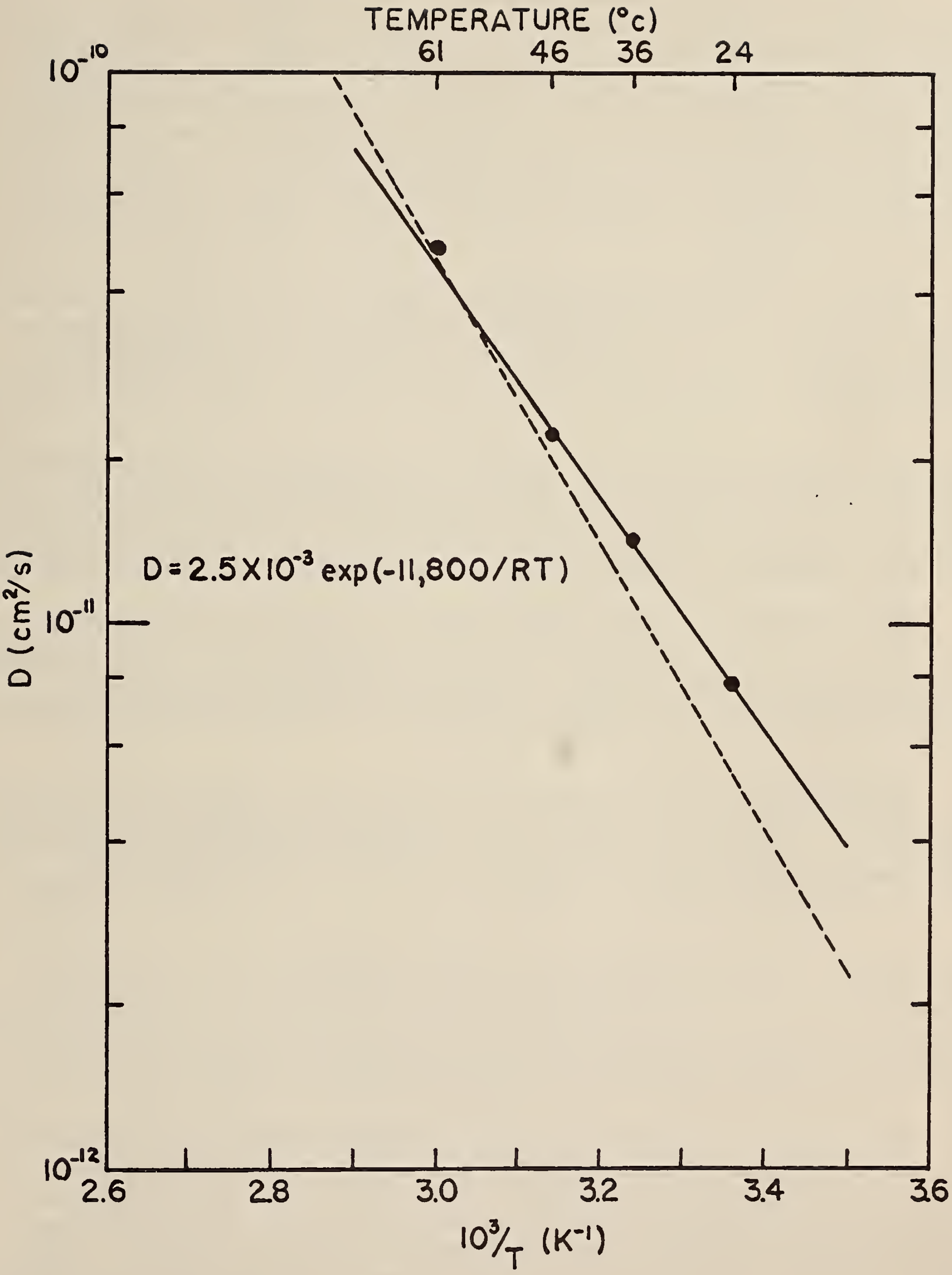

Fig. 3 Diffusion Constant of Lithium in Germanium Derived from Measurements of Lithium Mobility. (Dashed curve is taken from Reference 2).

A study was undertaken to determine the possibility of using a copper staining technique on germanium to develop "coring" patterns as had been observed when copper was electroplated on silicon. The germanium crystal was immersed in a specially constructed ice bath and staining with copper sulfate solutions of various concentrations containing various amounts of hydrofluoric acid. Infrared illumination was used (with and without extra filters of etched germanium slices) at varying times of exposure. However, no reproducible results regarding the copper stain patterns on a lapped germanium slice were noted. This project has been temporarily suspended. (H. E. Dyson and A. H. Sher)

Lithium precipitation measurements have been completed for samples NBS-01, -11, -61, -64, -65, -66, -610, -612, and -91. Oxygen concentrations of 9.5 x 10^{14} and 8 x 10^{14} atoms/cm^3 were determined in samples NBS-01 and -91, respectively. The data on the other samples are being evaluated.

An improved procedure for evaporating lithium metal was developed. The main innovation was in the use of a thermocouple to monitor the source boat to yield a more precise indication of the beginning and end of the evaporation. Thicker, more uniform, and more reproducible deposits of lithium have resulted from this improvement. (W. K. Croll)

Electron drift mobility measurements were made on samples NBS-01, -13, and -63 at room temperature and 77 K. Samples NBS-01 which contains approximately 1.5 x 10^{15} oxygen atoms/cm^3 as determined by infrared absorption and lithium precipitation showed a normal electron drift mobility at 77 K but low lithium drift mobility.

Absorption of infrared radiation in the 11.7 μm region was measured on sample NBS-91 and an oxygen concentration of 9.9 x 10^{14} atoms/cm^3 was determined. Lithium precipitation and lithium mobility measurements on this sample yielded oxygen concentrations of 8 x 10^{14} and 9 x 10^{14} atoms/cm^3, respectively. (W. R. Thurber)

A 1.4 cm^3 Ge(Li) detector was fabricated from a test slice of crystal NBS-83. The characteristics of this detector at 77 K were determined and the results were compared with test criteria selected previously (see Table 9.1). The measured characteristics of this detector will serve as a reference against which the performance of detectors fabricated from other material under evaluation may be measured.

The analysis of gamma-ray peak shapes from Ge(Li) detectors to obtain information on carrier trapping has begun. The approach taken is to treat the non-gaussian peak shapes, those which indicate trapping, as complex peaks. Initially, this treatment was applied to gamma-ray peak shapes obtained from the total irradiation of a Ge(Li) detector. Eventually, spectra obtained from collimated-beam irradiation will be treated. (A. H. Sher and W. J. Keery)

Table 9.1 Detector Characteristics at 77 K

Parameter	Criterion	NBS-83
Breakdown voltage	$\geq$ 500 V	> 1000 V
Leakage current at breakdown	$\leq$ 5nA	$\leq$ 3nA @ 1000 V(a)
Gamma-ray resolution		
Total system, @ 122 keV	$\leq$ 3 keV FWHM	2.7 keV FWHM
Detector, @ 1333 keV	$\leq$ 3 keV FWHM	1.8 keV FWHM
Peak shape:		
(FW.1M/FWHM) system	$\leq$ 2.12(b)	2.0 @ 122 keV
		2.1 @ 1333 keV
Capacitance vs Voltage:		
$\frac{dC}{dV}$ for V > 100 V	$\leq$ 5%	3%

(a) Actual breakdown voltage not measured

(b) (FW.1M/FWHM) for a gaussian peak + 16%

Plans for next quarter: Hall effect measurements, infrared absorption studies, impurity photoconductivity measurements, 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. It is also anticipated that a report on the use of lithium mobility, lithium precipitation, and infrared absorption for measuring oxygen concentration in germanium will be prepared for publication.

[†] Supported by the Division of Biology and Medicine, Atomic Energy Commission. (NBS Project 4259425)

1. W. M. Bullis and J. A. Coleman, "Characterization of Ge and Si for Nuclear Radiation Detectors," *Nucleonics in Aerospace*, P. Polishuk, Ed. (Plenum Press, New York, 1968) pp. 166-175.
2. C. S. Fuller and J. C. Severiens, "Mobility of Impurity Ions in Germanium and Silicon," *Phys. Rev.* 96, 21-24 (1954).
3. E. M. Pell, Jr., "Study of Li-O Interaction in Si by Ion Drift," *J. Appl. Phys.* 32, 1048-1051 (1961).

10. PROCESSING FACILITY

Objective: To establish a microelectronics fabrication laboratory consisting of oxidation, diffusion, photomasking, and contacting facilities capable of producing specialized silicon devices for use in research on measurement methods.

Progress during reporting period: All major steps in the installation of the diffusion furnaces were completed and the initial evaluation of boron diffusions from a gas-phase source using an open-tube technique was begun. An open-tube silicon oxidation system has been placed into operation. Oxides in the range of 5,000 to 6,000 Å have been grown on silicon wafers. The thicknesses were confirmed by infrared spectrophotometric absorption measurements.

A metallograph, a Watson-type interferometer for thin film measurement, and associated photographic accessories have been assembled into a single microscopy area. (T. F. Leedy)

Facilities have been established for the deposition of metallic and non-metallic thin films by vacuum evaporation and by sputtering. The vacuum evaporation facility has the capability of evaporating two source materials simultaneously. Film thickness is measured by a quartz crystal deposition monitor. The sputtering facility has the capability of employing rf, dc, or a combination of these techniques. Conditions for the rf sputtering of silicon dioxide are now being established. Cathodes for depositing silicon dioxide, gold, aluminum, silver, titanium, and tantalum are available.

Other facilities include those for the production of distilled and deionized water, preparation and examination of metallographic cross-sections, alloying at temperatures up to 1000°C in clean, dry hydrogen or inert gas atmospheres for chip bonding or contacts, and evaporation of lithium or other high vapor-pressure evaporants. A vertical furnace capable of heat treating quartz ampoules at temperatures up to 1000°C is also available. (W. K. Croll)

Plans for next quarter: Detailed evaluation of the oxidation process will begin. This will include the establishment of a firm procedure, the measurement of oxide thickness by interferometric and infrared absorption techniques, and the measurement of index of refraction by ellipsometry. In addition, the second diffusion furnace will be put into operation.

A substrate heater for the vacuum evaporation facility will be designed and installed. Sputtering techniques for available cathodes will be developed. A system for distributing distilled-deionized water to a number of chemical hoods will be designed and installed.

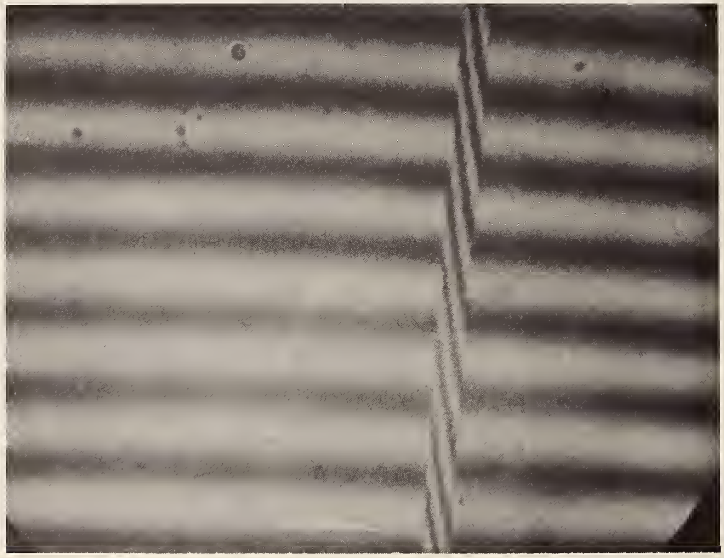

Fig. 4 Interference Pattern of a 0.54 μm Oxide Step on a Silicon Wafer.

11. WIRE BOND EVALUATION

Objective: To improve existing methods or develop new methods for evaluating the characteristics of wire bond contacts to semiconductor devices in order to detect more reliably those bonds which eventually will fail.

Background: A significant fraction of device failures under both radiation and non-radiation environments can be traced to failure of wire bonds. Since most wire bonds made are of good quality and do not fail, the problem is the early identification of these which will eventually fail in the field. In this project, which was established during the last quarter of fiscal year 1968, efforts are being made to define the problems more completely both through searches of the literature and through visits to orgainzations concerned with wire bonds from a variety of aspects. In addition facilities are being set up so that additional experimental work may be begun.

Progress during reporting period: Ten different organizations (government installations, universities, semiconductor device manufacturers, and bonding machine manufacturers) were visited to discuss the wire bonding problem. Particular emphasis was placed on bond strength tests employed and the bonding parameters and material characteristics which were considered important. Many important contacts were made during these visits. As was expected, the visits revealed numerous problems, questions, and a number of contradictory opinions. The results of these visits are still being studied and evaluated.

Compilation of a bibliography on wire bonding and related subjects was begun and more than 170 reports and papers have been collected.
(H. A. Schafft and K. O. Leedy)

A number of experimental approaches are actively being considered. A systematic study of the conditions that will produce poor bonds has been initiated. Equipment is being assembled to study, under various conditions of vibration and shock, the optimum curvature and slack to be specified for an attached wire bond. Methods for incorporating a 100 per cent wire pull test directly in a bonding machine are being considered. Two experimental bonders have been set up to facilitate this investigation.
(G. G. Harman and H. K. Kessler)

Plans for next quarter: Evaluation of the information gained on the visits already made will be continued and new visits will be scheduled during the next two quarters. Work will continue on assembly and review of material for the bibliography. Additional facilities for making wire bonds will be established and detailed experiments will be begun in the areas described above. Other methods for testing wire bonds will be considered.

12. 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.

Background: The line certification program which is now being considered by NASA required for its implementation standard forms of a large variety of test methods at all phases in the fabrication of microcircuits. Preliminary forms of typical test methods used in microcircuit fabrication have been compiled by NASA [1]. A project was begun earlier this year to assist in the review and improvement of these test methods. In some cases, ASTM standard tests were available to make the measurements suggested. In other cases ASTM task forces are actively working on the measurement methods proposed. These standards were identified and listed during the first phase of this project. A revised summary of ASTM activity appears as Appendix F of this quarterly report.

Progress during reporting period: The comparison of the NASA methods with ASTM standards was updated to include recent ASTM work. A questionnaire has been mailed by the chairman of ASTM Committee F-1 in order to poll committee members on the use of the various tests and on the applicability and use of the ASTM equivalents in production environments. The results of this poll will assist in the determination of priorities for future work. (W. E. Phillips)

Plans for next quarter: Some test methods, probably those not under active consideration by ASTM Committee F-1 at the present time, will be selected for editorial revision into a standard format based on ASTM style.

---

1. "Test Standards for Microcircuits," Draft of NASA-STD-XX-3, January 16, 1968.

13. 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.

Background: This project is a low-level continuation of a study of second breakdown which began about 1960. The study led to the development of a new type of specification for maximum operating conditions free from second breakdown which was based on the concept of a triggering energy for second breakdown dependent on the operating condition of a transistor [1]. In addition a series of review articles on the field was published [2] and a comprehensive bibliography of work in the field of second breakdown was compiled [3]. At the present time no experimental work is under way in this project.

Progress during reporting period: Three manuscripts dealing with second breakdown and thermal runaway in semiconductor devices were reviewed at the request of journal editors. An invited paper on second breakdown in transistors was prepared for delivery at the Second Symposium on Reliability in Electronics which will be held in Budapest in October. (H. A. Schafft)

Plans for next quarter: The invited paper will be presented and a round table discussion will be chaired at the Second Symposium on Reliability in Electronics in Budapest in October.

---

1. H. A. Schafft and J. C. French, "Second Breakdown in Transistors," *IRE Trans. Electron Devices*, ED-9, 129-136 (1962)
2. The most recent of these is H. A. Schafft, "Second Breakdown--A Comprehensive Review," *Proc. IEEE*, 55, 1272-1288 (1967)
3. H. A. Schafft, "Second Breakdwon in Semiconductor Devices--A Bibliography," NBS Tech. Note 431, October 1967.

14. THERMAL PROPERTIES OF DEVICES

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

Background: The thermal impedance rating of a semiconductor device is one of the most important ratings specified by a device manufacturer. It is measured electrically or, in some cases, calculated from heat transfer considerations by the device manufacturer to determine the maximum allowable power dissipation of a device family. The electrical measurement of thermal impedance is also used as a screening tool by some manufacturers to cull out defects such as poor solder wetting due to faulty processing. Designers use thermal impedance as a tool to determine the junction temperature of a semiconductor device under pulse as well as d-c operating conditions.

Present practices for measuring thermal impedance yield results of questionable accuracy and poor reproducibility. It is anticipated that detailed study of the measurement of thermal impedance will show that many of the problems involved with this reputed inaccuracy and poor reproducibility are due in part to inherent current distribution non-uniformities and their dependence on electrical operating conditions. These current distribution non-uniformities will cause localized variations in the junction temperature.

Temperature sensitive parameters customarily used to determine the junction temperature may not indicate the highest and therefore most critical local temperature [1]. Rather, a lower average temperature may be indicated which will result in calculating a value for thermal impedance which will be too low, leading to inadequate protection of the device. The calculated thermal impedance generally depends on the electrical operating conditions of the device during the measurement. Thus, without careful specification and control of operating conditions during measurement, the value of thermal impedance obtained may apparently show poor reproducibility. Because of the dependence on these operating conditions, a single value for the thermal impedance appears to be inadequate for characterizing device performance in general. It should also be recognized that other problems in the measurement of thermal impedance exist because of difficulties in defining and obtaining suitable test fixtures and measuring equipment.

JEDEC Committee JS-9 (Industrial Signal Transistors) and EIA Committee MED-32 (Active Digital Circuits) have shown an increasing interest in the thermal measurements problem. Both committees are in the process of establishing task groups concerned with thermal measurements.

Accomplishments during reporting period: Approximately one hundred reports and articles on the subject of thermal impedance measurements and heat flow analysis of semiconductor devices have been indexed and are being reviewed.

An existing system for measuring thermal resistance of low and medium power transistors by the I_{CO}, V_{EB}, and V_{CB} methods was reconditioned and modified, and is now nearing operational status. Further modifications to incorporate circuitry to measure current gain (h_{FE}) were considered because h_{FE} is sensitive to current density and therefore should be sensitive to the device hot-spot temperature insofar as current density is sensitive to junction temperature [2].

(F. F. Oettinger and S. Rubin)

Plans for the next quarter: The literature search and review of the methods of measurement of thermal impedance of semiconductor devices will continue. Modification of the existing thermal impedance measurement system will be completed and device measurements initiated. Thermal mapping of the devices by means of thermographic techniques is planned and equipment for this work will be assembled. Thermal mapping is necessary to confirm the junction temperature measurements made using the temperature sensitive electrical parameters of the device and to relate hot spot temperature to thermal impedance. Application of thermal measurements to the problem of evaluating the uniformity and quality of die attachment will be considered.

---

1. M. R. P. Young and D. A. Peterman, "Reliability Engineering," *Microelectronics and Reliability*, 7, 91-103, (1968).
2. J. T. Nelson and J. E. Iwerson, "Measurement of Internal Temperature Rise of Transistors," *Proc. IRE*, 46, 1207-1208, (1958).

15. MICROWAVE DIODE MEASUREMENTS

Objective: To determine if NBS can be of service to industry in defining and resolving some of the problems associated with the measurement of the characteristics of microwave mixer diodes.

Background: Mixer diodes are used to heterodyne weak microwave signals with a strong local oscillator signal to produce an intermediate frequency. Among the critical parameters of mixer diodes are the conversion loss, output noise ratio, and i-f impedance. These largely determine the noise figure of the receiver in which the diode is used.

In the past, because of the lack of microwave noise standards traceable to NBS, diode manufacturers relied on standard diodes as references to calibrate test equipment, diode holders, test procedures, measurements, etc. Originally, these standard diodes were calibrated by one of several government laboratories, but more recently with the termination of this service by these laboratories, a round-robin testing program among the manufacturers on the qualified products list has been used. While differences in noise figure of 0.1 dB can be resolved by this method, the accuracy is no better than 1.5 dB.

Since 1964, military specifications for semiconductor devices have contained the requirement that "guard bands" be used in establishing working limits so that the true values of the measured parameters are within the absolute values specified in the limits [1]. In addition, MIL-S-19500D requires that the standard mixer diodes and standard mixer holders used in the qualification testing of UHF and microwave mixer diodes shall be calibrated at least once in each successive 12-month period at a laboratory acceptable to the government [2].

In the discussions between the Defense Electronics Supply Center and the manufacturers, represented by JEDEC Committee JS-3 (UHF and Microwave Diodes) which followed the adoption of these standards, it became evident that there were no military laboratories which were equipped to calibrate mixer diodes, and that the commercial laboratories contacted were not interested in furnishing calibration services. Furthermore, the poor accuracy of the round-robin method of establishing reference diodes necessitated guard bands that were larger than the difference in overall (average) noise figure between consecutive mixer diodes in the same family, e.g. between the 1N21F and 1N21G.

(G. J. Rogers)

Plans for the next quarter: Representatives of NBS, both Washington and Boulder, the military services, and industry (through JEDEC Committee JS-3) will meet to resolve these questions and to plan future work.

From the information available it appears that the Boulder Labs can provide calibration of microwave noise sources at the frequencies presently required, 3.06 and 9.375 GHz. Other problems still to be resolved include:

1. Improvement of the accuracy of the primary standard.

2. Improvements in secondary standards used by industry and in the field to obtain the desired accuracy and stability.

3. Refinement of the test procedure for the measurement of mixer output noise ratio and overall (average) noise figure.

4. Simplification of procedures for the application of the standards and the test methods for noise measurements to facilitate their use by industrial and military personnel.

---

1. "Test Methods for Semiconductor Devices," MIL-STD-750A, May 1964, ammended by Notice 1, August 1965, and Notice 2, September 1966, par. 4.2.3; "Semiconductor Device, Diode, Silicon, Mixer, Types 1N21WG, 1N21WGM, and 1N21WGMR," MIL-S-19500/321A, 20 March 1967, par. 4.5.2; "Semiconductor Device, Diode, Silicon, Mixer, Types 1N23WG, 1N23WGM, and 1N23WGMR," MIL-S-19500/322a, 20 March 1967, par. 4.5.2; "Semiconductor Device, Diode, Silicon, Mixer, Types 1N3747W, 1N3747WM, and 1N3747WMR," MIL-S-19500/281A, 6 July 1967, par. 4.5.2.
2. "General Specification for Semiconductor Devices," MIL-S-19500D, May 1964, par. 4.4.3.

16. SILICON NUCLEAR RADIATION DETECTORS†

Objective: To establish the necessary facilities and undertake a continuing 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 during reporting period: Because of the poor quality of most large-diameter silicon crystals, detectors with areas greater than 10 cm^2 are not available from commercial sources at the present time. Two silicon crystals with diameters greater than 60 mm, resistivity $\sim$50 Ω-cm, and carrier lifetime $\sim$50 μs have recently been received. Crystals which are used for detectors customarily have carrier lifetime $\sim$1000 μs. As a result of the low lifetime value in the large-diameter crystals, it is unlikely that Si(Li) detectors made from these crystals will have desirable low-noise characteristics. A thorough study of these crystals is planned.

The equipment and plans for a study of electron damage effects in silicon surface-barrier detectors are nearly completed.

(B. H. Audet and J. A. Coleman)

Plans for next quarter: Silicon intended for use in large-area Si(Li) detectors will be characterized. Properties to be measured include resistivity profile, carrier lifetime, oxygen density by infrared absorption, lithium mobility, and detector characteristics. Electron damage studies will begin. Irradiations will be made with electrons of the following energies: 50, 100, 400, 600, and 800 keV. In addition, life-testing apparatus for silicon detectors will be assembled for use by the sponsor.

---

† Supported by Goddard Space Flight Center, National Aeronautics and Space Administration. (NBS Project 4254429)

Appendix A

JOINT PROGRAM STAFF

Coordinator: J. C. French

Semiconductor Characterization Section

Dr. W. M. Bullis, Chief

A. J. Baroody, Jr.
D. L. Blackburn
F. H. Brewer
M. Cosman
Dr. J. R. Ehrstein
R. L. Gladhill
G. G. Harman
F. R. Kelly*
H. K. Kessler
Mrs. K. O. Leedy
R. L. Mattis
Dr. W. E. Phillips
Miss D. E. Sanders§
J. L. Scales†
H. A. Schafft
A. W. Stallings
Dr. L. J. Swartzendruber
W. R. Thurber

Secretaries:

Miss T. A. Poole
Miss P. K. Smith
Miss R. E. Young

Semiconductor Processing Section

Dr. J. A. Coleman, Chief

B. H. Audet
H. A. Briscoe
R. D. Clayton*
W. K. Croll
H. E. Dyson§
W. J. Keery
E. I. Klein
J. Krawczyk
T. F. Leedy
Dr. A. H. Sher
L. M. Smith
G. P. Spurlock

Secretary:

Mrs. S. W. Davis

Electron Devices Section

J. C. French, Chief

Mrs. J. K. Moffitt
F. F. Oettinger
M. Phillips
G. J. Rogers
S. Rubin
M. Sigman
L. R. Williams

Secretaries:

Mrs. C. F. Bolton (P. T.)
R. S. Brown*
Miss B. Hope

---

* Summer Student
§ YOC Program
† Guest Worker

Appendix B

COMMITTEE ACTIVITIES

ASTM Committee F-1:

- 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, Epitaxial Thickness, etc.
- J. C. French, Chairman, Subcommittee VIII, Editorial
- W. E. Phillips, Task Forces on Crystal Perfection, Encapsulation, Thin and Thick Films, etc.
- A. H. Sher, Task Force on Germanium†
- M. Sigman, Editor, Subcommittee V, Semiconductor Processing Materials
- L. J. Swartzendruber, Task Force on Resistivity
- * W. R. Thurber, Task Forces on Conductivity Type†, Impurities in Semiconductors†, Optoelectronic Materials, etc.

Note: * Attended Fall Meeting, September 1968 in Denver
† Participated in Round-Robin Experiments this Quarter

Electronic Industries Association:

- MED 32, Active Digital Circuits: F. F. Oettinger, TG 32.5, Thermal Resistance Test Methods

Joint Electron Device Engineering Council (EIA-NEMA):

- JS-3, UHF and Microwave Diodes: J. C. French, Correspondence on Microwave Diode Specification Problems
- JS-6, Power Transistors: H. A. Schafft, Consultant on Second Breakdown Specifications
- JS-9, Industrial Signal Transistors: F. F. Oettinger, Panel Discussion on Thermal Time Constants
- JS-14, Thyristors: F. F. Oettinger, Thermal Resistance of SCR's

IEEE:

- Nuclear Science Group, J. A. Coleman: Administrative Committee
  Radiation Instruments and Detectors Committee
- Magnetics Group, S. Rubin: Galvanomagnetic Standards Subcommittee

IEC:TC47:

- S. Rubin, Galvanomagnetic Devices

NAS-NRC Semiconductor Detector Panel:

- J. A. Coleman

Appendix C

SOLID-STATE TECHNOLOGY & FABRICATION SERVICES

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

1. Solid-state technology - (W. K. Croll)
   a. A silicon internal reflection element was prepared for the Vacuum Measurements Section.
   b. A zirconium anode was soldered for the Atomic Physics Section.

2. Ultrasonic fabrication - (J. Krawczyk)
   Several piezoelectric devices for turbulence studies were made for the Naval Ship Research and Development Center.*

3. Germanium detectors - (A. H. Sher)
   a. A 5 cm^3 Ge (Li) gamma-ray detector was made and tested for the Nuclear Spectroscopy Section.
   b. A Ge (Li) gamma-ray detector was reprocessed for the Radiochemical Analysis Section.

4. Quartz fabrication - (E. I. Klein)
   a. A synchrotron injector was repaired twice for the Linac Operations Section.
   b. A calorimeter window was prepared for the Solid State Physics Section.
   c. Glass components were made for a micrometer system for the Vacuum Measurements Section.
   d. A liquid nitrogen dewar flask was repaired for the Radiation Thermometry Section.

5. Gunn devices - (G. G. Harman, K. O. Leedy, and H. K. Kessler)
   In this continuing project for the Harry Diamond Laboratories, attempts were made to fabricate Gunn diodes from three gallium arsenide epitaxial wafers. For different reasons, none of the wafers yielded satisfactory diodes.†

---

* NBS Project 4254431

† NBS Project 4251424

Appendix D

ELECTRON DEVICES DATA SERVICE †

The Electron Devices Section of the National Bureau of Standards Electronic Technology Division maintains an Electron Devices Data Service. This service was originally established in order to provide technical information relative to electron tubes. It was later expanded and now includes data on semiconductor devices and integrated circuits in its information file on approximately sixty thousand devices of foreign and domestic origin. Assistance is available, without charge, to individuals in government and industry who may be interested in obtaining information regarding the technical specifications, availability, manufacturers, or other pertinent data relative to specific devices.

This quarter, the Data Service has published a revised version of its tabulation of the characteristics of U.S.S.R. devices extracted from available foreign publications [1]. In this tabulation, which has been prepared as a quick reference source on these devices, information is presented on receiving, power, microwave, cathode ray, and miscellaneous tubes as well as on semiconductor devices. J. K. Moffitt)

---

† Supported by NBS. (Project 4252121)

1. J. K. Moffitt, "Tabulation of Published Data on Soviet Electron Devices," NBS Tech. Note 441, July 1968.

Appendix E

JOINT PROGRAM PUBLICATIONS

W. M. Bullis, F. H. Brewer, C. D. Kolstad, and L. J. Swartzendruber, "Temperature Coefficient of Resistivity of Silicon and Germanium near Room Temperature," *Solid-State Electronics*, 11, 639-646 (1968).

J. A. Coleman, D. P. Love,[*] J. H. Trainor,[†] and D. J. Williams,[†] "Effect of Damage by 0.8 MeV - 5.0 MeV Protons in Silicon Surface-Barrier Detectors," *IEEE Trans. Nuc. Sci.*, NS-15, 363-372 (1968).

L. J. Swartzendruber, F. H. Ulmer, and J. A. Coleman, "Direct Reading Instrument for Silicon and Germanium Resistivity Measurements," accepted by *Rev. Sci. Inst.*

H. A. Schafft, "Second Breakdown in Transistors," accepted by Second Symposium on Reliability in Electronics, Budapest, October 1968.

W. M. Bullis, "Measurement Problems in Microcircuit Processing," accepted by Government Microcircuits Applications Conference, Gaithersburg, October 1968.

---

[*] Vitro Services, Greenbelt, Md. 20771

[†] NASA-GSFC, Greenbelt, Md. 20771

Appendix F

ASTM STANDARDS RELEVANT TO INTEGRATED CIRCUIT PROCESSING

Committee F-1 on Materials for Electron Devices and Microelectronics of the American Society for Testing and Materials is continuing the development of Recommended Practices, Specifications, and Methods of Test related to the electron device and microelectronics industry. Documents generated by this committee are published in Part 8 of the *ASTM Book of Standards*. This volume appears in November of each year. A few other relevant documents developed by other ASTM Committees appear in other parts of the *Book of Standards*.

At all times, many documents are in various stages of the process which leads to completed standards. Published and unpublished documents pertinent to silicon and silicon processing are listed below.

Titles of published documents which have been accepted by the ASTM as standards or tentatives are preceded by the ASTM serial designation. This designation consists of a letter and number which identify the document followed by the last two digits of the year of adoption and, for tentatives only, a T. Proposed documents published for the first time in 1968 will not have serial designations assigned until they are approved as either Tentatives or Standards.

The unpublished draft documents listed are now being actively considered by Committee F-1. Although most of these will eventually appear as published standards, it should be recognized that some will be dropped before final action is taken.

I. Semiconductor Crystals

A. Resistivity:

F43-67T Tentative Method of Test for Resistivity of Semiconductor Materials [Two-probe method now being revised; see F84 for improved four-probe method]

F81-67T Tentative Methods of Test for Bulk Semiconductor Radial Resistivity Variation

F84-68T Tentative Method of Test for Resistivity of Silicon Slices Using Four Pointed Probes

Draft Method of Test for Silicon Epitaxial Layer Resistivity by the Three-Probe Voltage Breakdown Technique

Draft Method of Test for Resistivity of Semiconductor Materials by the Two-Probe Method

Draft Method for Determination of Resistivity and Impurity Profiles in Epitaxial Silicon Layers by the Diode Capacitance-Voltage Technique

Draft Method of Test for Silicon Epitaxial Layer Resistivity by the Spreading Resistance Technique

Draft Method of Test for Silicon Epitaxial Layer Resistivity by the Four-Probe Method [For use on p/n or n/p wafers.]

B. Conductivity Type:

F42-68T Tentative Methods of Test for Conductivity Type of Extrinsic Semiconducting Materials [Includes hot thermoelectric probe, cold thermoelectric probe, and rectification methods.]

C. Crystal Perfection:

F47-68 Standard Method of Test for Crystallographic Perfection of Silicon by Preferential Etch Techniques

F80-67T Tentative Method of Test for Crystallographic Perfection of Epitaxial Deposits of Silicon by Etching Technique

Draft Method of Test for Stacking Fault Density in Silicon Epitaxial Layers by Interference Contrast Microscopy

Draft Method of Test for Crystallographic Perfection of Silicon by X-Ray Topography

D. Carrier Lifetime:

F28-66 Standard Method for Measuring the Minority-Carrier Lifetime in Bulk Germanium and Silicon [Photoconductive decay method, see also Proc. IRE 42, 1292 (1961).]

E. Orientation:

F26-66 Standard Methods for Determining the Orientation of a Semiconductive Single Crystal [Includes both X-ray and optical methods.]

F. Hall Mobility:

F76-68 Standard Method for Measuring Hall Mobility in Extrinsic Semiconductor Single Crystals

G. Impurity Content:

F45-64T Tentative Method of Test for Oxygen Content of Silicon [Now being revised to include a new calibration curve and a differential method to increase the sensitivity of the test.]

Draft Recommended Practices for Infrared Absorption Analysis of Impurities in Semiconductor Crystals

Draft Method of Test for Substitutional Carbon in Silicon

Draft Method of Test for Boron, Phosphorus, and Arsenic in Silicon

H. Thickness:

F95-68T Tentative Method of Test for Thickness of Epitaxial Layers of Silicon on Substrates of the Same Type by Infrared Reflectance [Suitable for n/n^+ and p/p^+ epitaxial layers.]

Draft Method of Test for Thickness of Epitaxial and Diffused Layers by the Angle Lap and Stain Technique

Draft Method of Test for Thickness of Epitaxial Layers by Stacking Fault Edge Measurement

I. Test Ingots:

F40-68 Standard Method for Preparing Monocrystalline Test Ingots of Silicon by the Vertical Pulling (Czochralski) Technique

F41-68 Standard Method for Preparing Silicon Single Crystals by the Floating-zone Technique

J. Surface Condition:

F21-65 Standard Method of Test for Hydrophobic Surface Films by the Atomizer Test

F22-65 Standard Method of Test for Hydrophobic Surface Films by the Water-Break Test

F24-65 Standard Method for Measuring and Counting Particulate Contaminants on Surfaces

F59-68T Standard Method for Identification of Metal Particulate Contaminants Found in Electronic and Microelectronic Components and Systems Using the Ring Oven Technique with Spot Tests

Draft Method of Test for Bowing of Silicon Wafers

Draft Methods for the Examination and Characterization of Defect Structures and Contaminants as Seen on Specular Silicon Surfaces

II. Photomasking:

F66-66T Tentative Methods of Testing Physical Properties of Photoresists Used in Microelectronic Fabrication

F93-68T Tentative Recommended Practice for Preparation of Specifications for the Procurement of Photomasks

Proposed Definitions of Terms Relating to Photomasking Technology for Microelectronics

Draft Method for Dimensional Inspection of Photomasks

Draft Method for the Visual Inspection of Emulsion Photomasks

Draft Method of Test for Photomask Flatness

Draft Recommended Practice for Determining Multiple Photomask Registration

III. Water:

D1069-66 Standard Methods of Test for Particulate and Dissolved Matter in Industrial Waste Water

D1125-64 Standard Methods of Test for Electrical Conductivity of Industrial Water and Industrial Waste Water

D1129-68 Definitions of Terms Relating to Industrial Water and Industrial Waste Water

D1193-66 Standard Specifications for Reagent Water

D1688-68 Standard Method of Test for Copper in Industrial Water and Industrial Waste Water

D1888-67 Standard Methods of Test for Particulate and Dissolved Matter in Industrial Water

F57-68 Standard Method of Concentrating and Measuring Trace Quantities of Copper in High-Purity Water Used in the Electronics Industry

F60-68 Standard Methods for Detection and Enumeration of Microbiological Contaminants in Water Used for Processing Electron and Microelectronic Devices

IV. Other Processing Materials:

F52-65T Tentative Method of Test for Silting Index of Fluids for Processing Electron and Microelectronic Devices

F58-68 Standard Method of Measuring Specific Resistivity of Electronic Grade Solvents

F61-68 Standard Method of Test for Phosphate in Electronic Grade Hydrogen-Peroxide Solutions

F62-68 Standard Method of Test for Tin in Electronic Grade Hydrogen-Peroxide Solutions

F94-68T Tentative Method of Test for Aluminum in Electronic Grade Hydrogen-Peroxide Solutions

Draft Method of Test for Impurities in Nitric Acid

Draft Method of Test for Purity of Boron Tribromide

Draft Method of Test for Purity of Silicon Tetrachloride

V. Cleaning Procedures

Draft Recommended Practice for the Cleaning of Silicon Slices

Draft Method of Test for the Cleanness of an Epitaxial Layer Deposition Apparatus

Draft Recommended Practice for Ultrasonic Cleaning

VI. Packaging and Associated Parts

F18-64 Standard Specification and Method for Evaluation of Glass-to-Metal Headers Used in Electron Devices

F72-66T Tentative Specification for Gold Wire for Semiconductor Lead Bonding

F78-67T Tentative Method for Calibration of Helium Leak Detectors by Use of Secondary Standards

F97-68T Tentative Method of Test for Hermeticity of Electronic Devices by Dye Penetration

F98-68T Tentative Method of Test for Hermeticity of Electronic Devices by a Bubble Test

F100-68T Tentative Method of Test for Shrinkage Stresses in Plastic Embedment Materials Using a Photoelastic Technique (for Electronic and Similar Applications)

F218-68 Standard Method for Analyzing Stress in Cylindrical Metal-to-Glass Seals

Proposed Method of Test for Embedment Stress Caused by Casting Compounds on Glass Encased Electronic Components

Draft Specification for Lead-Frame Strips for Integrated Circuit Packages

Draft Recommended Practice for Testing the Effect of Encapsulation Systems on Microelectronics

Draft Method of Measurement for Completeness of Cure-State of Crosslinked Resin Polymers for Electronic Devices by Means of Hot Indentation

Draft Method of Test for the Effect of Moisture on Electrical Insulation Varnishes for Plastics

Draft Method of Test for Hermeticity of Electron Devices by a Helium Mass Spectrometer Test

Draft Method of Test for Hermeticity of Electron Devices by a Radioisotope Test

Draft Method of Test for Reversion Characteristics of Elastomeric Encapsulation Systems

VII. Clean Rooms:

F25-68 Standard Method for Sizing and Counting Airborne Particulate Contamination in Clean Rooms and Other Dust-Controlled Areas Designed for Electronic and Similar Applications

F50-65T Tentative Method for Continuous Counting and Sizing of Airborne Particles in Dust-Controlled Areas by the Light-Scattering Principle (for Electronic and Similar Applications)

F51-68 Standard Method for Sizing and Counting Particulate Contaminants in and on Clean Room Garments

F71-68 Standard Method of Test Using Morphological Key for the Rapid Identification of Fibers for Contamination Control in Electron Devices and Microelectronics

F91-68T Tentative Recommended Practice for Testing for Leaks in Filters Associated with Laminar Flow Clean Rooms and Clean Work Stations by Use of a Condensation Nuclei Detector.

VIII. Hybrid Microcircuits

Draft Method of Test for the Sheet Resistance of Metallic Thin Films by the Four-Probe Technique

Draft Method for Determining the Adhesion of Metal Films to Hard Translucent Substrates by the Scratch Test

Draft Method of Test for the Thickness of Thin Films by the Stylus Method

Draft Definitions of Terms in Thin and Thick Film Technology

Draft Method of Test for Adhesion of Microcircuit Thick Films to Ceramic Substrates

Draft Method of Test for Sheet Resistance of Resistor Compositions for Thick-Film Circuitry

Draft Method of Test for Noise in Thin-Film Conductors

Draft Method of Test for Resistivity of Insulating Materials

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, $6.00; foreign, $7.25*.

- **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, $2.25; foreign, $2.75*.

- **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, $2.75; foreign, $3.50*.

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*.

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 quantitative 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

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

Order NBS publications from:

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

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

OFFICIAL BUSINESS