NBS TECHNICAL NOTE

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

Quarterly Report
October 1 to December 31, 1968

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

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NBS TECHNICAL NOTE 475

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

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National Bureau of Standards
Washington, D.C. 20234

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† Related Projects

ABSTRACT

This second quarterly progress report in this series 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 aluminum metallization, wire bonds, and wafer die attachment; 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 services, and publications are included as appendixes.

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

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

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October 1 to December 31, 1968

1. INTRODUCTION

This is the second quarterly report to the sponsors of the Joint Program on Methods of Measurement for Semiconductor Materials, Process Control, and Devices which is supported jointly by the National Bureau of Standards [1], the National Aeronautics and Space Administration [2], the Defense Atomic Support Agency [3], and the U. S. Naval Ammunition Depot, Crane, Indiana [4]. The Joint Program was undertaken earlier this 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, such as uniformity of response to radiation effects.

This report is subdivided according to tasks which have been identified as parts of the Program. Sections 2 through 9 deal with methods of measurement for materials; sections 10 through 14, with methods of measurement for process control; and sections 15 through 19, with methods of measurement for devices. 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. 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 program planning.

In the first report of this series [5] background information was given for the Program and for 15 of the tasks. In this quarter work was begun on three new tasks. One of these, the task on Metallization Evaluation, had been identified during the April 1968 program review. The other two tasks, Die Attachment Evaluation and Thermographic Measurements, evolved from the task on Thermal Properties of Devices.

The objective of the task on Infrared Methods has been broadened to include work on detection of certain impurities in silicon and germanium by infrared absorption. This work is in support of American Society for Testing and Materials Committee F-1 on Materials for Electron Devices and Microelectronics which is developing standards for such measurements.

Efforts on the task on Wire Bond Evaluation have been substantially increased during this quarter. The tasks on Metallization Evaluation and Die Attachment Evaluation were established, in part, because of the importance of the overall bond failure problem. Program staff members participated in several briefings related to various aspects of this problem and Dr. J. A. Coleman attended the Fact-Finding Conference on Radiation Effects in Semiconductor Devices (sponsored by the Defense Electronics Supply Center) at General Electric, Syracuse, New York, on November 19, 1968 and the Symposium on Transient Radiation Effects and Techniques for Circuit Hardening (sponsored by the Defense Atomic Support Agency) at the Naval Research Laboratory, Washington, D. C., on December 9-13, 1968.

Considerable committee work was carried on during this period in addition to items specifically mentioned in connection with various tasks. Although there were no meetings of ASTM Committee F-1, four documents were reviewed editorially for the committee, six were reviewed editorially for various subcommittees, and four were reviewed informally. Revisions were prepared for both methods for determining crystallographic perfection of silicon by etching to extend the method to larger diameter crystals. A manuscript on "Measurement Standards for Integrated Circuit Processing" was prepared together with the chairman of ASTM Committee F-1 for submission to a special issue of the *Proceedings of the IEEE*. At the November meeting of the Nuclear Instruments and Detectors Committee of the IEEE Nuclear Science Group a revised standard for testing Geiger-Mueller tubes was reviewed in detail.

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.

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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 808-69. (NBS Project 4259522)
4. Through Cost Reimbursement Order P09-0016. (NBS Project 4259533)
5. "Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, July 1 to September 30, 1968," NBS Tech. Note 472, December 1968.

2. RESISTIVITY

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

Progress: The round robin based on the ASTM four-probe method [1], which is being conducted in cooperation with ASTM Committee F-1 on Materials for Electron Devices and Microelectronics, has not been completed because of delays encountered at several participating organizations. Six of the nine participants in the round robin have completed the measurements.

Measurements for an F-1 sponsored round robin based on a proposed two-probe method [2] for measuring the resistivity of cylindrical silicon crystals are being made. It is necessary to take 120 measurements on each of eight crystals. A calculator program has been prepared to speed processing of the data. About half the crystals have been measured.

(F. H. Brewer)

Preliminary results have been obtained in the study of the dependence of resistivity of silicon wafers as measured by the four-probe method on different currents. Pronounced, though not fully consistent, variations have been observed when the current is changed in the range between 0.1 and 10 times the current recommended in the standard method. Specimens with resistivity greater than 100 Ω-cm showed decreasing resistivity with increasing current, while specimens of less than 100 Ω-cm resistivity usually showed decreasing resistivity with decreasing current. Although there was a tendency to exhibit a constant resistivity as current was increased in the range 5 to 10 times the recommended value, a 100 Ω-cm n-type specimen exhibited increasing resistivity with increasing current in this range. A digital voltmeter has been ordered so that data for this study can be taken more rapidly.

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

A series of measurements was made to try to determine how the "forming" of the metal-semiconductor contact influences the measurement of resistivity of epitaxial layers by the voltage-breakdown method. Results so far are inconclusive. The observation by another laboratory that the value of resistivity measured depends on the precise details of forming and that forming should therefore be avoided is generally substantiated by the measurements. However on some specimens, particularly those of resistivity between 1 and 5 Ω-cm, no definite breakdown could be observed until the contact had been formed to some extent. The reason for this necessity to form contacts in these cases is not understood.

The relation of pulse length to voltage breakdown levels was also investigated. Indications at present are that triangular voltage pulses should be limited to about 100 μs because of the onset of forming and the possibility of probe and wafer deterioration at the high power levels which accompany longer pulses.

An existing probe holder was modified to enable it to be used for measurements at various points on the specimen surface, with all other contacts remaining fixed. This probe holder also allows interchanging high quality probes of various radii in order to evaluate the effect of probe radius on observed breakdown voltage. Problems with mechanical vibration of the probe at breakdown have been encountered when using this holder and so satisfactory measurements have not yet been made.

Some measurements have been made with available pieces of silicon carbide as breakdown probe material. It has been reported that much more reproducible voltage breakdown values can be obtained with this material than with most other materials [3]. Available material has not yielded satisfactory results. (J. R. Ehrstein)

Plans: After work on the two-probe resistivity round robin is completed the study of the effect of various parameters on four-probe resistivity values will be extended by measurements at various probe pressures with the range of current values already used. The four-probe measuring system will be modified to include the new digital voltmeter.

It is expected that tabulation and analysis of the data from the four-probe round robin will be completed before the June meeting of ASTM Committee F-1.

The experiments on the voltage-breakdown method will be continued. Additional efforts will be made to eliminate the mechanical vibration of the modified holder at breakdown. The availability of silicon carbide of suitable and reproducible form and doping level for voltage breakdown work will be investigated.

Because measurement of spreading resistance is an important and related alternative to the voltage-breakdown method for measuring resistivity of epitaxial layers, the feasibility of obtaining a measurement system for this technique will be investigated. It is expected that such a system would be compatible with the requirements of the draft method for measurement of resistivity by means of spreading resistance which is now being developed by ASTM Committee F-1.

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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.
2. For a general description of the two-probe method see "Method of Test for Resistivity of Semiconductor Materials" (ASTM Designation: F43-67T), *ibid.*
3. P. A. Schumann, Jr., and A. Dupnock, "An Evaluation of Point Material for the Three-Point Probe," *Electrochem. Tech.* 6, 218-219 (1968).

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.

Progress: Experimental work on the determination of bulk lifetime from filament lifetime as measured by the photoconductive decay (PCD) method [1] was continued. A 22 Ω-cm, p-type germanium specimen with lapped surfaces was measured at its initial size of 1.0 by 1.0 by 2.15 cm. The specimen cross section was reduced and the decay time remeasured. It was found that if the diffusion coefficient was chosen as 101.7 cm^2/s, the computed bulk lifetime values agreed within 7 per cent for three specimen sizes. Earlier difficulties in a similar experiment on a 4 Ω-cm p-type silicon specimen are attributed to inhomogeneities within the specimen.

A mathematical analysis was made of the effect of bulk inhomogeneities on lifetime measurement by the PCD method. The specimen was first assumed to be divided into incremental lengths with uniform lifetime within each increment. The measured lifetime was found by averaging over the specimen length. Second, the specimen was assumed to be composed of constant-lifetime parallel filaments. In both cases, the analysis revealed that in the presence of inhomogeneities the use of chopped light in which a steady-state non-equilibrium carrier distribution is set up prior to decay should always produce a longer measured lifetime than the use of light pulses which have duration smaller than the carrier lifetime.

A study of recombination models was made in the hope that PCD measurements could be used to identify a particular specimen with a particular model. It would be helpful for instance to be able to estimate the density of trapping (as opposed to recombination) centers in a specimen. It appears that this and similar information can be obtained only by combining PCD measurements with other lifetime measurements.

A summary of the results to date is being prepared. This summary considers the problems associated with the PCD method in three groups: (1) effects of the surface, (2) effects of inhomogeneities, and (3) the dependence of lifetime on excess carrier density. (R. L. Mattis)

Construction and testing of the circuitry for making voltage decay carrier lifetime measurements on diodes was completed. A series of measurements was made on germanium and silicon switching and zener diodes with lifetimes in the range 5 to 80 μs to compare results of carrier lifetime measurements by the reverse recovery and voltage decay methods. Although both are expected to yield the same result, lower values for carrier lifetime were obtained from the voltage decay measurements. The circuitry is being modified in an effort to eliminate the problem.

With balanced forward and reverse currents at 4.0 mA with a 1 kHz square wave input, the reverse recovery method was shown to be capable of measuring lifetimes as low as 1 μs, while the voltage decay method using 10 V, 20 μs pulses at a repetition rate of 4000 pulses per second is capable of measuring lifetimes as low as 0.1 μs. Other reverse recovery circuitry now being studied is able to measure values of carrier lifetime below 10 ns.

One area of difficulty encountered during these tests was the interpretation of the graphical output of the reverse recovery measurement. A typical reverse recovery curve is shown in Fig. 1. The plateau is used to determine the carrier lifetime. Experiments showed that the most consistent results were obtained when the width of the plateau was taken as the time between the current zero (point A in Fig. 1) and the beginning of the current decay (point B). (A. J. Baroody)

Low temperature steady-state photomagnetoelectric and photoconductivity (PME-PC) measurements on indium antimonide specimens revealed inconsistencies which measurements of the specimen resistance *in situ* showed were due to failure of the specimen to follow the temperature variations of the copper holder. The cause of this failure was traced to the 0.12 mm-thick glass slide which provided electrical insulation between specimen and holder. After the slide was replaced with a sapphire wafer the temperature differential between the specimen and holder was reduced to a value less than the uncertainty in the temperature as measured by a thermocouple imbedded in the copper holder. Following this modification of the apparatus, several runs were successfully completed (see Section 7).

(W. E. Phillips, J. L. Scales, and A. W. Stallings)

Plans: The summary of work on the problems associated with the PCD method will be completed.

Instrumentation modifications for the voltage decay method will be tested. After the problems have been resolved, measurements on p-i-n and surface barrier diodes will be started. Modifications will be made in the circuitry used for reverse recovery measurements so that the ratio of forward to reverse current can be varied continuously between 0.01 and 100. The dependence of the measured response time upon the frequency and amplitude of the input square wave will also be studied.

The PME-PC equipment will be physically rearranged for convenience in operation and consolidated with the surface photovoltage (SPV) equipment. Following this, measurements by the SPV method will be resumed.

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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.

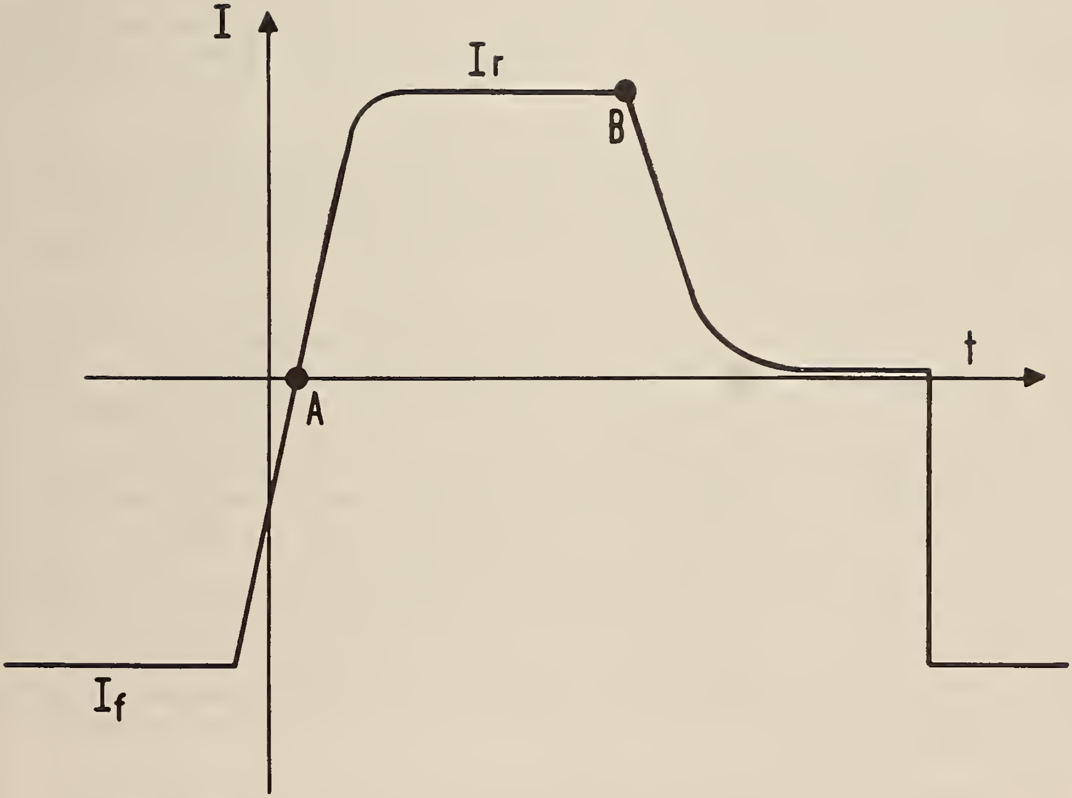

Fig. 1 Typical current vs time curve for the reverse recovery method.

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: While a number of resistivity profile measurements made on p-type germanium and n- and p-type silicon specimens with the photovoltaic technique did give results which agreed to within less than 1 per cent with the corresponding two-probe resistivity profile measurements, there were other cases where apparent surface effects prevented such precision. In particular, the photovoltage in these cases changed magnitude and sometimes even polarity with time (hours to days) and with surface preparation. These effects produced significant changes in the calculated resistivity profile. Attention has therefore been shifted toward a study of the effects of surface conditions on the photovoltage rather than methods for preparing the surface to minimize the surface recombination velocity and thereby maximize the photoresponse from the specimen.

Preliminary measurements on 60 Ω-cm n-type silicon indicate that at least for high resistivity material it may be possible to obtain a sufficiently large photoresponse to determine resistivity profiles without having to etch or otherwise treat the surface of the specimens after lapping with 5 μm alumina.

Resistivity measurements on a p-type silicon rectangular bar with significant variations in resistivity perpendicular to its long axis (along the width of the specimen) served to underscore the limitation of the photovoltaic technique for such a case. Here the photovoltaic resistivity profile along the length of the specimen differed by as much as 3 per cent from an average of three two-point resistivity profiles measured along three different lines on one face of the specimen.

Work on the theory of a photovoltaic method for measuring resistivity gradients along the diameter of a circular specimen was begun. The method makes use of an ohmic contact at each end of a diameter to be probed. These contacts are used to measure the photovoltage generated by resistivity gradients along the diameter when probing with a small circular spot of light.

The effect of geometry on the photovoltage as it is measured at the ohmic contacts was calculated. In the calculation it was assumed that a constant voltage dipole could be used to simulate the action of the light probe in a specimen which has a constant resistivity gradient along the measurement diameter. The solution for the photovoltage consists of two factors. One factor represents the potential difference which appears at the contacts when the light probe is at the center of the wafer. This factor is a function of the voltage dipole and therefore is implicitly dependent on such parameters as the resistivity gradient and the excess electron-hole pair concentration introduced by the light probe. The other factor is a form factor in that it is essentially dependent on the position of the light probe along the measurement diameter. This factor shows how the photovoltage, simulated by the dipole, is modified by the geometry of the specimen.

(H. A. Schafft, D. L. Blackburn, and L. J. Swartzendruber)

Plans: Measurements on p-type germanium and n- and p-type silicon bar specimens will be continued to improve the technique. Special emphasis will be placed on overcoming the apparent surface-induced problems which have caused inconsistent resistivity profile measurements.

Work on the theory of a photovoltaic method for measuring resistivity gradients in circular specimens will be continued. Determination of the resistivity gradient along the measurement diameter from photovoltage measurements will require the derivation of an expression for the center position factor which will involve, explicitly, the resistivity gradient and the excess electron-hole pair concentration or photoconductivity. As a first approach the photovoltage generated by a resistivity gradient (non-zero only along the measurement diameter) will be considered for the case when the light probe is located at the center of the wafer. Because a separate measurement of the photoconductivity of the specimen will be needed, a form factor to take into account the position of the light probe must be calculated theoretically or obtained with the use of an electro-mechanical analog. One such analog which has been considered is a metal disk with a means of altering the resistance at points along a diameter to simulate the photoconductive effect of the light probe. As in the semiconductor wafer, the total resistance is determined from the potential drop between the ends of the same diameter.

The results of these calculations will be used to determine the kind of preliminary quantitative measurements that will be made on circular specimens. If appropriate, measurements on rectangular bar specimens where the light probe does not extend completely across the width of the specimen, postponed this quarter, will be undertaken.

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-1 in extending the applicability of infrared absorption as a method for detecting impurities such as oxygen and carbon in silicon and germanium.

Progress: A liquid helium cryostat for the impurity photoconductivity work was received from a commercial supplier. Additional gratings and filters to extend the wavelength range of the monochrometer system to 40 μm were ordered.

Tests were made to determine if the signal-to-noise ratio of the double-beam spectrophotometer used for absorption measurements could be improved by using a lock-in amplifier in place of the corresponding electronics in the spectrophotometer. No significant improvement was obtained.

The limit of detection for oxygen in germanium was determined for the differential absorption method. In this, the most sensitive of the various absorption procedures which can be used, the test specimen is placed in the sample beam of the spectrophotometer, a specimen of the same thickness but without the impurity is placed in the reference beam, and the difference in transmission (absorption) is read directly by the instrument. A detection limit of 8×10^{14} cm^{-3} at room temperature and 2×10^{14} cm^{-3} at 20 K was found when 10-mm slices were used.

Absorption measurements were made at 78 and 300 K on three silicon specimens containing oxygen in conjunction with a round robin being conducted by ASTM Committee F-1. In addition to the air reference method called for in the round robin, oxygen concentration was determined by using the procedures specified in the existing standard method [1] and by using the differential absorption technique. (W. R. Thurber)

Plans: The equipment for low temperature photoconductivity measurements will be assembled and preliminary measurements made on germanium specimens diffused with copper or gold. Several impurity concentrations, verified by neutron activation analysis or other means, will be used in these experiments.

The absorption data obtained by the various measurement methods from the three silicon specimens described above will be analyzed. In addition results obtained previously will be examined and the need for more work on the various methods will be considered. A value for the ratio of the 9 μm absorption coefficient due to oxygen in silicon at 80 K to that at 300 K will be obtained.

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1. "Method of Test for Oxygen Content in Silicon" (ASTM Designation F45-64T), *1968 Book of ASTM Standards*, Part 8, November 1968.

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 automatic temperature control system for the Hall effect experiment was completed, and it is now possible to measure the Hall coefficient and resistivity of a specimen at 48 different temperatures without an operator. The output data are punched on cards which are processed at the NBS computer facility. Programs have been written which enable the computer to prepare a magnetic tape from which the results can be machine plotted. Many plots have been made in connection with the Deep-Level Studies (see Section 7).
(W. R. Thurber, J. L. Scales, and R. L. Gladhill)

A draft of the report which describes the coupling between the Hall experiment and the time-shared computer system has been written in cooperation with Computer Center personnel [1].

Work on a report concerning Hall measurements and their interpretation is continuing. Emphasis has been on the relationship between Hall coefficient and carrier concentration in gallium arsenide, silicon, germanium, and indium antimonide. (W. R. Thurber and W. M. Bullis)

Plans: Material related to measurement and interpretation of the Hall effect in gallium arsenide will be discussed at the January meeting of the Optoelectronics Task Force of ASTM Committee F-1.

Experimental tests will be conducted to determine if it is practical to increase the input impedance of the automatic system by using an electrometer input. Hall coefficient and resistivity measurements will be made on high-purity germanium specimens which have very high resistance below room temperature (see Section 9).

The two reports now being prepared will be completed.

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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).

7. DEEP-LEVEL STUDIES

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

Progress: Lithium diffusion experiments were performed on specimens of high resistivity p-type indium antimonide. The specimens became n-type after diffusion, passed through a precipitation phase lasting from two to three weeks, and then became p-type again as shown in Fig. 2. Analysis of Hall data gave clear indication that the untreated samples had had a deep-lying donor impurity in a concentration from 0.8 to 1.2 x 10^{14} cm^{-3}. After lithium treatment, similar Hall measurements showed complete absence of the donor level.

PME-PC lifetime measurements (see Section 3) were performed on both treated and untreated specimens. With the same magnetic flux density, sample dimensions, and light intensity in the two sets of measurements, the electron lifetime was found to be increased by four orders of magnitude in the lithium-treated material. This was further confirmation of the idea that lithium had neutralized the donor impurities that had been acting as recombination centers. The lifetime of holes was increased slightly by the lithium treatment, but the principal change was in the variation with temperature. In untreated material, the hole lifetime showed a pronounced decrease around 125 K. This dip was absent in the data on treated material. (J. L. Scales)

Plans: Additional lifetime measurements will be made on treated and untreated material. The problem of calibration of the light source will be given further consideration. At the conclusion of these experiments the work will be prepared for publication.

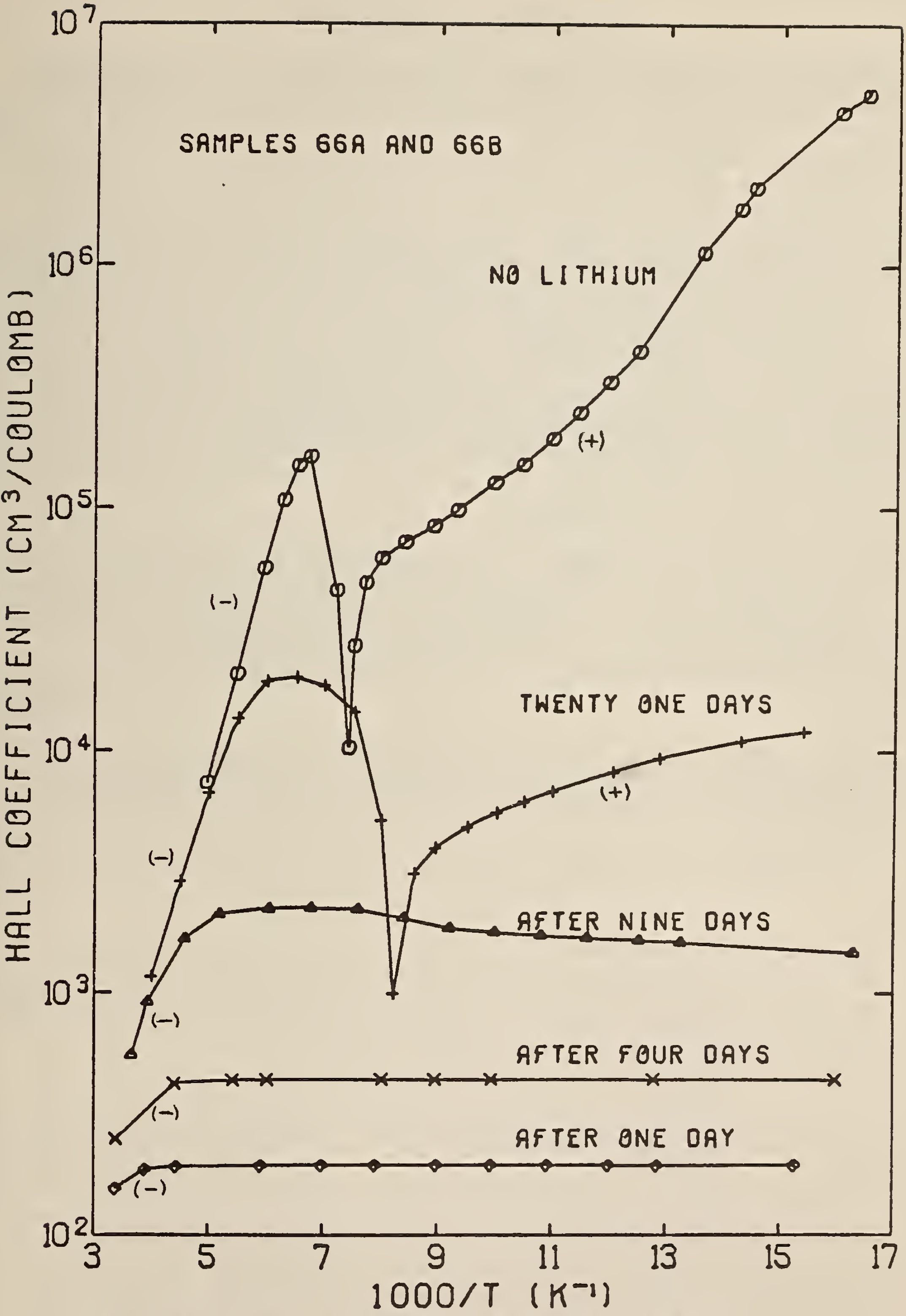

Fig. 2 Hall coefficient vs temperature showing the effects of lithium precipitation. Initially and after 21 days the Hall coefficient is negative at higher temperatures and positive below the transition point. After 1, 4, and 9 days it was negative at all temperatures.

8. HIGH FIELD EFFECTS

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

Progress: Many silicon specimens were prepared and tested for oscillation. These were first oxide masked and then had boron or phosphorous diffused into the contact ends. Specimens of different resistivity, carrier type, and length were prepared, each with the long axis oriented 22° from a <100> direction on a {100} surface. Both ohmic and junction specimens of 1 and 100 Ω-cm were tested at 77 K. None oscillated. The 10 Ω-cm junction specimens did oscillate at about 30 MHz, but the amplitude of the oscillation was only 2 to 3 per cent of the d-c current level. The orientations of several of these specimens, determined by a precision X-ray technique, were compared with the known orientations of several specimens which had previously been shown to have amplitudes about 10 per cent of the d-c current level. All were found to be very close to the desired 22° so there is no obvious reason for the variation in oscillation amplitude.

Oxide passivated specimens displayed no current change with increasing magnetic field. Therefore previously observed current changes can be attributed to surface recombination. The oscillation threshold and frequency did change as on the unpassivated specimens. These changes appear to be fundamental to the oscillation phenomenon.

(G. G. Harman)

Plans: Measurements will be completed on the newly prepared specimens. The effect of strong magnetic fields will be investigated, and the exact temperature dependence of the oscillation will be studied.

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.

Progress: The data on the 50 ingots presently being examined in this study has been transferred to punched cards. Single-crystal, p-type germanium for nuclear radiation detectors obtained from nine domestic and foreign suppliers are represented among the 50 crystals. The summary includes data on resistivity, lifetime, dislocation density, method of crystal growth, dopant, electron drift mobility, Hall (hole) mobility, lithium drift mobility, oxygen concentration (as determined by measurements of infrared absorption, lithium precipitation, and lithium mobility), and measured characteristics of Ge(Li) detectors fabricated from the test crystals. (A. H. Sher and H. E. Dyson)

A manuscript entitled "Lithium Ion Drift-Mobility in Germanium" was completed and submitted for publication to the *Journal of Applied Physics*. Studies of the change in capacitance of a drifting germanium p-i-n diode as a function of time revealed a third distinct field region at the junction which occurred at the beginning of the drifting process in addition to the linear-graded junction field normally found at early stages in the drifting process and the uniform field normally found after charge compensation. (A. H. Sher)

Hall coefficient and resistivity measurements were made as a function of temperature using the automated data acquisition system on two high-purity germanium specimens grown by R. N. Hall [1]. The results obtained from one specimen were in good agreement with those of Hall's group. The other specimen exhibited very high contact resistance resulting in loading by the digital voltmeter. (W. R. Thurber)

Sufficient data has been obtained on the measurement of oxygen impurity concentration in germanium by infrared absorption, lithium precipitation, and lithium mobility [2,3] so that the range of applicability and expected degree of agreement among the three methods can be examined. (A. H. Sher, W. K. Croll, and W. R. Thurber)

The construction of a collimator capable of producing a narrow beam of gamma-rays was completed, and the apparatus was put into use in the testing of Ge(Li) detectors. A 15 mCi source of ^{137}Cs (662-keV gamma-rays) was encased in a lead housing which acts as personnel shielding and provides the primary collimation of the monoenergetic gamma-ray beam. A secondary collimator produces a beam 1 mm in diameter at the aperture. The collimator is adjustable in two perpendicular axes by means of calibrated micrometer screws so that both horizontal and vertical scans of a Ge(Li) detector can be made. Measurements of the beam spread as a function of the distance from the aperture using dental X-ray film, showed that the beam width at the center of the active region of the detector is between 1.2 and 1.3 mm. Detector characteristics which can be determined using such a collimator [4] include the dimensions of the active region of the detector, hole or electron trapping lifetime, and identification of the type of carrier, if any, being preferentially trapped.

A Ge(Li) detector of 2 cm^3 active volume (NBS-83-2) was fabricated for use as a reference detector for collimation measurements. Gamma-ray pulse height spectra have been obtained as a function of both the applied collection field and the position of the gamma-ray beam between the n^+-i and i-p junction boundaries, using the collimated 662-keV beam on this detector. The data are being evaluated to determine if the measurement of the Fano factor in germanium at 77 K is affected by the collection of either electrons or holes separately [5]. The Fano factor is a characteristic of the charge production process only; the results of a measurement, however, also include trapping effects which occur during the collection of the radiation-induced charge. Therefore, this study seeks to determine the effects of unequal charge collection on the measurement process. (A. H. Sher and W. J. Keery)

Plans: 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. Additional Hall measurements are planned after modifications to the Hall effect system are made (see Section 6). Manuscripts describing the results of the oxygen determination and the Fano factor studies will be prepared.

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

1. AEC Contract AT(30-1) 3870, High Purity Germanium for Gamma Detectors, with General Electric Research and Development Laboratory.
2. E. M. Pell, "Study of Li-O Interactions in Si by Ion Drift," *J. Appl. Phys.* 32, 1048-1051 (1961).
3. R. J. Fox, "Lithium Drift Rates and Oxygen Contamination in Germanium," *IEEE Trans. Nucl. Sci.* NS-13, No. 3, 367-369 (1966).
4. P. P. Webb, et. al., "Use of Collimated Gamma-Ray Beams in the Study of Ge(Li) Detectors," *Nucl. Instr. Meth.* 63, 125-135 (1968).
5. A. H. Sher and B. D. Pate, "Determination of the Fano Factor in Germanium at 77 K," (Submitted to *Nucl. Instr. Meth.*).

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: The second diffusion furnace was placed into operation and preliminary data were obtained using a phosphine source. The end-zone temperatures were further adjusted to improve the uniformity over the entire lengths of both diffusion furnaces. To enable the observation of any long-term trends in the tube temperature with acceptable resolution, a digital voltmeter with a precision of 2 μV (corresponding to a temperature uncertainty of about 0.2 degree) was set up to measure the thermocouple voltages. This voltmeter is coupled to a card punch so that all three temperature zones of a furnace are sampled and recorded in rapid succession.

The thermal oxidation facility is being modified so that oxidation may be carried out with wet or dry oxygen, or steam with a nitrogen or argon carrier gas. In wet oxygen, about four hours will be required to grow a 1-μm thick silicon oxide layer.

The deionized water distribution system was designed but estimates of installation cost exceeded expectations. Alternate methods for obtaining high-purity water are being investigated.
(J. Oroshnik and T. F. Leedy)

A substrate heater for the vacuum evaporation station has been built and installed. The heater operates from room temperature to somewhat in excess of 600°C. Aluminum films in the range 0.3 to 1.1 μm have been deposited on silicon at substrate temperatures of approximately 300°C. A quartz crystal thickness monitor was used to control the thickness.

A new multiple-beam interferometer has been set up in the microscopy area. This instrument is capable of measuring film thickness from 4 nm to 2 μm as well as surface roughness. A very sharp interference pattern is obtained (see Fig. 3) and film thicknesses can be measured with considerably greater precision than with a two-beam interferometer. In addition the quartz crystal thickness deposition monitor can now be calibrated periodically. Arrangements were made to obtain ellipsometric measurements of index of refraction of silicon dioxide layers.

Sputtering activities were hampered by problems with leaks inherent in the design of the commercially-obtained system. These problems have been eliminated by installing a heat exchanger and redesigned flexible tubing for the cooling system. (T. F. Leedy and W. K. Croll)

Plans: Development of diffusion techniques with diborane and phosphine will continue, with the immediate objective of reproducible surface carrier concentrations and junction depths. For the present, junction depths will be determined by the angle lapping and staining technique. Using the junctions depths so measured, approximate surface concentrations will be determined from sheet resistivity measurements in conjunction with Irvin's curves [1].

Aluminum evaporations will be made on silicon and silicon dioxide substrates in order to establish standard evaporation techniques. Modifications will be made on the substrate heater so as to provide more accurate temperature measurement and control.

Work will continue on establishing controlled sputtering techniques for depositing aluminum and silicon dioxide films.

Fig. 3 Interference pattern of a 1.7 μm-thick aluminum stripe as observed on multiple-beam interferometer.

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1. J. C. Irvin, "Resistivity of Bulk Silicon and of Diffused Layers in Silicon," *Bell System Tech. J.* 41, 387-410 (1962).

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.

Background: The only two methods presently in use throughout the thin film field for measuring film adherence to a substrate are the peel (pressure sensitive tape) test, and the scratch test. Neither test yields a well defined value for the adhesive forces between a thin film and its substrate. It is well established that one of the more important of the many failure areas in the wire bonding system is adherence failure of the aluminum thin film to the silicon dioxide substrate. A clear and tangible knowledge of these adhesive forces obtained by way of a suitable adhesion measurement should prove to be an invaluable asset in pursuing the problem of wire bond evaluation, since connections to semiconductor chips are all made on thin metal films.

Plans: Efforts will be made to devise a scheme for measuring the adhesive strength of thin metallic films. The object is to contrive a simple test that will yield unambiguous information, in contradistinction to the peel and scratch tests currently in use. Attention will be focused on the adhesion of thin aluminum films deposited by vacuum evaporation or sputtering on silicon or silicon dioxide.

12. WIRE BOND EVALUATION

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

Progress: The wire bond system includes all parts of the device which comprise the connection between the semiconductor chip and the post or pin which leads to the outside of the package. Among the many materials involved are the semiconductor itself, oxide films, metal films, bonding wire, and the post or pin with its plating or coating. Preliminary criteria have been established to aid in selecting reports and papers from the vast literature which exists on the various aspects of the system for the bibliography and review on wire bonding. Primary emphasis is being placed on documents which describe methods for evaluating the quality of the wire bond system and for monitoring the bonding procedure.

Special efforts are being directed toward finding documents which contain correlations between evaluation data and field or laboratory failures to obtain information on the effectiveness of particular methods in detecting bond systems that eventually fail. Unfortunately, however, little such information has been found, either in documents collected or on visits made to date. Documents with descriptions of bond evaluation procedures, comparisons between different evaluation procedures, and correlation between evaluation data and bonding procedures are also of great interest.

In addition, material is being collected on procedures and techniques for making wire bonds. For the purposes of the bibliography, information relevant to all types of wire bonds (such as thermocompression and ultrasonic) and various wire and pad materials will be sought.

The 170 papers collected during the previous quarter have been examined and many have been discarded on the basis of the above criteria. Other papers obtained this quarter have been added to the collection which now numbers about 100.

An increased effort has been made to locate pertinent contract reports where much of the latest information on the problem is believed to reside. To this end, the bibliographic search capabilities of NASA, the Defense Documentation Center (DDC), and the Clearinghouse for Federal Scientific and Technical Information (CFSTI) were investigated. CFSTI provides listings of reports based on a key word selected from a limited number of possibilities. The term "integrated circuits" was the available key word most closely related to the wire bond problem. A listing of reports issued since 1963 based on this key word was requested. Although a long list of documents was received, only a few were relevant to the bond problem. A list of the unclassified work units relating to the making and testing of wire bonds in semiconductor integrated circuits was requested from the DoD Work Unit Data Bank maintained by DDC. NASA search capabilities which were found did not appear to be more useful for obtaining material for the bibliography than a direct search of the cumulative subject indexes of *Scientific and Technical Aerospace Reports*.

The search functions of this task have been combined with those of Die Attach Evaluation (see Section 13) in order to avoid duplication of effort. (H. A. Schafft, K. O. Leedy, and M. Sigman)

Visits to two organizations were made to discuss the wire bond system problem. This brings the total of such visits to twelve. The evaluation of information obtained is continuing. (H. A. Schafft)

A motor-driven bond puller has been designed and is being assembled. This puller is mounted on a bonder platform and uses the bonder micropositioner. A mechanical dynamometer is used as the indicator. Initially a simple hook is used to catch the bond wire, but consideration is being given to grasping the wire by other means so that the direction of pull can be controlled. Assembly of equipment and construction of test circuits for conducting burn-in screen tests based on method 1015 of MIL-STD-833 [1] have been initiated.

Aluminum has been vacuum deposited and sintered into several silicon substrates for use in connection with the study of conditions which will produce good or bad bonds. It was decided to limit this study initially to aluminum-aluminum ultrasonic bonds because of the importance of this bonding system in radiation-resistant devices.

Equipment for vibration tests of small devices by methods 2005 and 2007 of MIL-STD-883 [1] has been assembled and placed into operation. This facility may be useful in detecting bonds with poor electrical contacts.

Further consideration has been given to the 100 per cent, nondestructive pull test incorporated directly into the bonding machine. A possible approach to this type of test would be to mount a small counterbalanced magnetic chuck between the capillary and the wire spool. After the bond is made, the chuck would hold the wire during a programmed ascent of the tool. When the chuck deflected a preset amount, equivalent to 1 or 2 grams tension, electrical contact would be broken and the chuck would release. Weak bonds would fail prior to release. In addition, any severe defects in the wire would result in rupture.
(G. G. Harman, H. K. Kessler, and K. O. Leedy)

Plans: About 10 more visits to government and industrial laboratories will be scheduled for next quarter as part of the field review of the current status of wire bonding and wire bond system evaluation procedures. Collection and review of documents for the bibliography will continue, and the listing of work units will be examined after it is received to locate contracts which might bear on the bond problem. It is expected that the bibliography will be completed during the next two quarters. Work will begin on analyzing and summarizing the information obtained from the visits made and documents collected. When this is completed, a critical review of the status of the wire bond problem will be prepared.

Assembly of the facilities for pull testing and burn-in will be completed. Aluminum-aluminum ultrasonic bonds will be made under a variety of conditions and tested with these facilities. Facilities for a high-current, pulse method for nondestructive bond testing will be assembled and preliminary tests of the method will begin. The use of aluminum wire ribbon in ultrasonic bonding will be investigated [2]. Special tungsten carbide bonding tools for ribbon wire will be designed.

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1. "Test Methods and Procedures for Microelectronics," MIL-STD-883, 1 May 1968.
2. Previous experience with gold ribbon indicates that such wire produces bonds that are stronger at the heel, are more flexible in the direction of greatest movement, and can absorb torque that would otherwise be transmitted to the bond.

13. 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.

Background: Voids or other faults in the die attachment are a significant cause of failure in high-reliability semiconductor devices. These faults usually increase the thermal resistance of the path between the active area of the device and the case. Thermal resistance measurements as well as thermographic, radiographic, and metallographic techniques are presently used, in varying degrees, for the detection of poor die attachment. The measurement of thermal resistance and the use of radiographic techniques lend themselves to 100 per cent testing by both supplier and user. These techniques are not extensively used but merit further study to evaluate their applicability and reproducibility for various types of semiconductor devices (see Section 16).

Progress: A literature search on the techniques utilized in evaluating the uniformity and quality of semiconductor device die attachment was initiated. This search is being carried out in conjunction with the Wire Bond Evaluation search (see Section 12).

(F. F. Oettinger and M. Sigman)

Plans: The literature search on the techniques utilized in evaluating the uniformity and quality of semiconductor device die attachment will continue. A study of the feasibility of fabricating semiconductor diodes with known voids will be undertaken.

14. 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: Early returns of the questionnaire circulated by ASTM Committee F-1 on use and adequacy of ASTM standards for microcircuit processing and of other test methods in the NASA compilation [1] have been tabulated and studied. On the basis of this small sample (25) it appears that there are several test methods which are widely accepted and used but which have not been standardized. These include measurements of oxide thickness and stability and of photoresist processes. The responses also tentatively identify some unsatisfactory methods on which further work is needed. These include temperature measurements of wafers during epitaxial deposition and diffusion processes.

An interim method for measuring oxide thickness on silicon wafers by interference measurements on a metallized step formed after etching away part of the oxide layer is being prepared in a standard format. This method was selected because, although it is destructive, it is widely used and the procedures appear to be satisfactory.

(W. E. Phillips)

Plans: Analysis of the questionnaire returns will continue. Preparation of the interim method for measuring oxide thickness will be completed.

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

15. 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: An invited paper titled "Second Breakdown in Transistors" was presented at the Second Symposium on Reliability in Electronics which was held in Budapest, Hungary, in October. In addition, the author led a round table discussion on the same subject. (H. A. Schafft)

Plans: A first draft of a manuscript on "Failure Modes" will be written. This will be a section in the chapter titled "Users Guide for Power Transistors" of "Recommended Standards for Power Transistors" which is being prepared by the JEDEC Committee JS-6 on Power Transistors.

16. THERMAL PROPERTIES OF DEVICES

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

Progress: The literature search and review of the methods of measurement of thermal impedance of semiconductor devices were continued.
(F. F. Oettinger and M. Sigman)

The system for determining thermal resistance from measurements of pulsed and steady-state h_{FE} was built and is being debugged. Initial measurements utilizing methods based on h_{FE} and V_{EB} for measuring thermal resistance indicated that further work is needed to improve the precision of the test procedures. The original temperature-controlled test fixture is limited to an upper temperature of 75°C, making it necessary to design a new test fixture which is capable of operating up to a temperature of 275°C in order to enable calibration of the temperature sensitive parameter at hot-spot temperatures.
(F. F. Oettinger, S. Rubin, and R. L. Gladhill)

Plans: The literature search and review of the methods of measurement of thermal impedance of semiconductor devices will continue.

Additional modifications will be made on the h_{FE} and V_{EB} thermal resistance measuring circuits to improve the precision of the measurement as well as to increase the maximum temperature capabilities of the system. Transistors with selected geometries, lead configurations, and power handling capabilities will be utilized as test specimens for both the thermal resistance and thermographic measurements (see Section 17).

17. THERMOGRAPHIC MEASUREMENTS

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

Background: The use of phosphors to display two-dimensional temperature distributions was first reported by Urback, Nail, and Pearlman [1]. The utility of these phosphors to determine the current or power dissipation distribution by means of the associated temperature distribution in discrete semiconductor devices and in integrated circuits has been largely overlooked by the electronics industry. Some work has been reported in their use as a screening tool for passive components [2,3] as well as for making qualitative determinations of the current distribution prior to and after the initiation of second breakdown in transistors [2,4]. However, no serious attempts have been reported in the literature to develop the technology for using these phosphors to make quantitative measurements over areas comparable with those of the active regions of semiconductor devices.

To obtain a two-dimensional temperature distribution, a thin layer of the phosphor powder is applied to the surface to be studied. The temperature-dependent luminescence of the phosphor, which is in the visible range, is excited by near ultra-violet radiation. The phosphors have the property that the intensity of the luminescence will decrease and finally quench as the temperature of the phosphors is increased. Four phosphors are commercially available which cover a temperature range from room temperature to about 400°C [5]. The temperature range over which a phosphor can be used can be shifted somewhat by changing the intensity of the exciting radiation. Increasing the intensity shifts the range to a higher temperature.

With refinement in the technology for using these phosphors, the detection of temperature differences of about 1 degree would be expected. The average diameter of the phosphor particles is 9 μm. With the ability to apply a thin, uniform layer of phosphor it should be possible to observe temperature distributions with a spatial resolution of 25 μm. These two capabilities are significant especially when the relative simplicity and low cost of the method and the advantages of measuring a visible display are all taken into consideration.

Progress: Measurements of transistor hot-spot temperature made with thermographic phosphors have demonstrated that quantitative measurements can be made with this technique although some problems remain to be solved. Hot spots are clearly revealed, but the intensity of the luminescence over the metallization differs from that over the oxide as can be seen in Fig. 4. For quantitative measurements the illuminated phosphor was photographed on a 35-mm film strip and the density was measured point by point with a microdensitometer. The film strip was calibrated by photographing the device at various elevated temperatures

without power. This process is both cumbersome and time consuming, and so it would not be suitable for routine scanning purposes.
(D. B. Brenner,[*] G. J. Rogers, and F. F. Oettinger)

Plans: Improved methods of applying the temperature sensitive phosphors and measuring their light output will be studied. Suitable light intensity measuring equipment will be ordered and construction of a test fixture initiated.

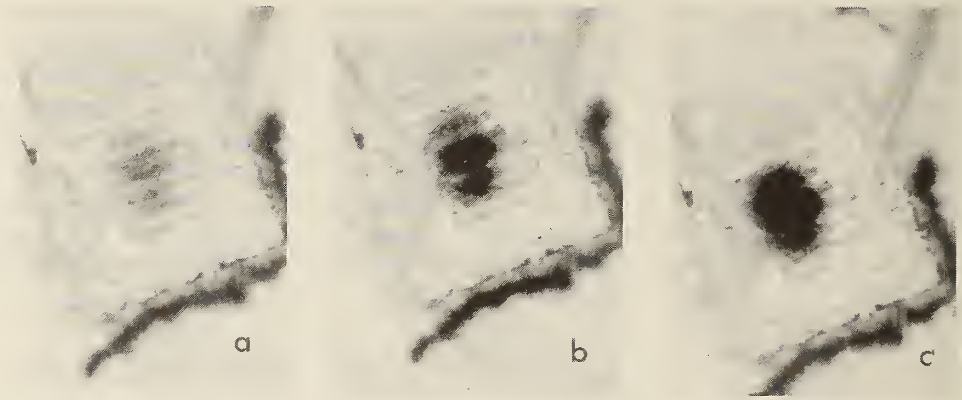

Fig. 4 A 20-watt planar transistor without its cap is shown covered with a thin semitransparent layer of a temperature sensitive phosphor to display the temperature distribution for different operating conditions:

(a) V_{CE}=20 V, I_C=800 mA, P=16 W
(b) V_{CE}=40 V, I_C=400 mA, P=16 W
(c) V_{CE}=60 V, I_C=255 mA, P=15.3 W

The heat sink temperature is 35°C. The semiconductor chip is approximately 1.5 mm on a side. The peak temperature in (a) is about 180°C and in (c) is about 190°C. The dark area adjacent to the chip is a scratch in the phosphor used for orientation purposes.

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[*] NBS Measurement Engineering Division

1. F. Urback, N. R. Nail, and D. Pearlman, "The Observation of Temperature Distributions and of Thermal Radiation by Means of Non-linear Phosphors," *J. Opt. Soc. Am.* 39, 1011-1019 (1949).
2. H. D. Frazier, "Temperature Dependent Flourescent Paints—A Graphic Display of Temperature Distribution," *IEEE Wescon Convention Record* 7, part 3.1, 1-5 (1963).
3. J. W. Ballard, "Detection and Prediction of Malfunction of Electronic Components by Contact Thermography," *Proc. National Aerospace Electronics Conference* (1964), pp. 154-163.
4. H. A. Schafft and J. C. French, "Second Breakdown and Current Distributions in Transistors," *Solid-State Electronics*, 9, 681-688 (1966).
5. W. H. Byler and F. R. Hays, "Fluorescence Thermography," *Nondestructive Testing* 19, 177-181 (1961).

18. MICROWAVE DIODE MEASUREMENTS

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

Progress: Representatives of the Navy and NBS met at NBS to review the status of microwave diode measurements. It was concluded that the measurement of noise figure appears to require immediate attention and that there was a misunderstanding in industry of what services are available from NBS.

At a subsequent meeting of representatives of EIA, the Naval Applied Science Laboratory, and NBS the history of the mixer diode noise figure measurement problem was discussed, calibration services required and available were outlined, and measurement facilities used by industry were examined. It was concluded that many of the needs for calibration of gas-tube noise standards can be met by existing or soon to be available services at the NBS Boulder Laboratories, and that NBS can be of service in three ways:

1. Continue to provide and expand measurement services for noise sources and other basic standards,
2. Thoroughly study and evaluate methods and equipment used for testing diodes, and
3. Establish and maintain a measurement service for standard reference diodes.

The Naval Applied Science Laboratory was visited to discuss apparatus and techniques used for noise measurement there. It was concluded that it would be most practical for NBS with existing relevant capabilities and experience to supply the above listed services.

At a meeting of JEDEC Committee JS-3 on UHF and Microwave Diodes these conclusions were outlined and material was distributed describing measurement services already available from NBS. The Committee concurred with the conclusions.

A study was begun to determine the theoretical limits of the uncertainties in diode noise figure determinations made by different laboratories with a finite number of measurements. Recent developments in explaining uncertainties in frequency measurements appear to be applicable to measurements of noise in non-linear devices where a broad band of frequencies is present. (R. C. Powell and G. J. Rogers)

Plans: The objectives of the present task have been met. If it is determined that the scope of the effort should be expanded, apparatus will be assembled for an experimental investigation of problems involved in mixer noise measurement, the theoretical study of uncertainties will continue, and other problems associated with measurement of mixer characteristics will be considered.

19. SILICON NUCLEAR RADIATION DETECTORS†

Objective: To conduct 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: A platform and shroud for mounting silicon detectors in vacuum ($<10^{-9}$ torr) during life testing has been designed so that the temperature of the detectors can be cycled between -20 and +50°C or be held constant to within a few tenths of a degree within this range. This system is being established so that static electrical parameters and counting performance can be determined for each detector over extended periods of time in order to provide a basis for predicting the long-term stability of semiconductor detectors in space.

Two ingots of silicon with diameters of approximately 67 mm are being evaluated for use in large-area Si(Li) detectors. The resistivity of each ingot varied between 60 and 100 Ω-cm over a distance of ∿6 cm. Determination of carrier lifetime by the photoconductive decay method showed two components, a short-time value of ∿200 μs and a long-time value of ∿2000 μs. The short-time value is typical of a normal recombination lifetime, whereas the long-time value is an indication of either single or multiple trapping of carriers. (B. H. Audet)

A 1.0-mm thick, silicon, surface-barrier, transmission detector was irradiated with 600-keV electrons incident on the front contact at fluences of 10^{12} to 10^{16} electrons/cm^2. The detector leakage current and capacitance, as shown in Fig. 5, and the noise and counting characteristics were determined after each increment of fluence. The counting response to alpha particles from ^{241}Am incident on the front contact decreased with increasing fluence, and the ability to detect alpha particles through the front contact was lost after a fluence of between 1.5×10^{13} and 1.2×10^{14} electrons/cm^2. In contrast to this, the response to alpha particles incident on the rear contact improved with fluence until noise due to damage began to degrade the energy resolution. (J. A. Coleman)

Plans: Construction of the detector mount and shroud for high-vacuum life testing will begin. The radial resistivity profile, oxygen content, lithium mobility, and detector performance characteristics of the 67-mm diameter silicon will be measured. A second 1.0-mm thick silicon, surface-barrier, transmission detector will be irradiated with 600-keV electrons incident on the rear contact. The effects of radiation damage on the detector performance for this exposure will be compared with those observed for the irradiation of the front contact.

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

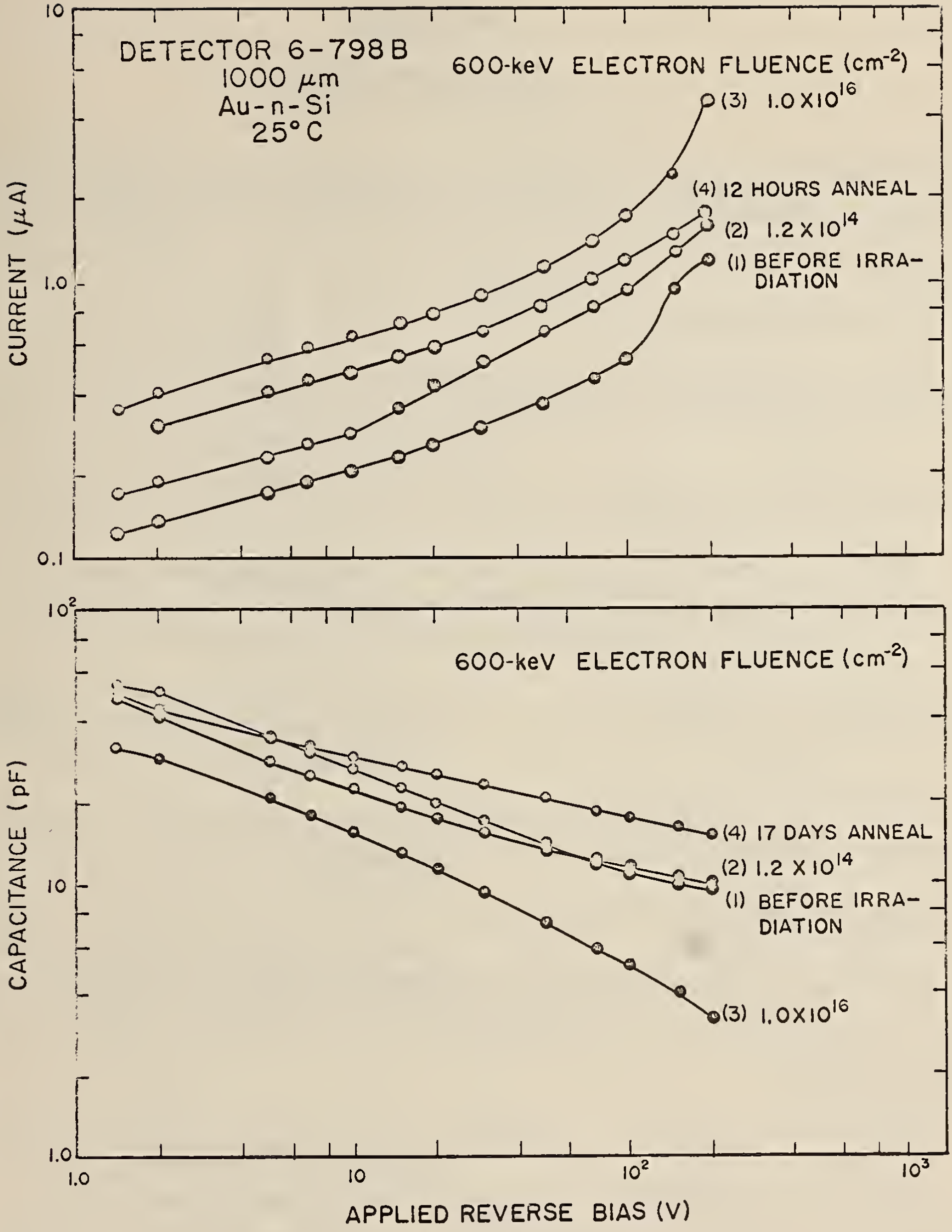

Fig. 5 Leakage current and capacitance of a silicon, surface-barrier, transmission detector (1) before irradiation, (2, 3) after irradiation with fluences as marked, and (4) after annealing in one atmosphere of dry nitrogen at room temperature. Normal operating bias is 150 V. Data taken after irradiation with a fluence of about 10^{12} electrons/cm^2 deviated only slightly from data taken before irradiation. Although both of these parameters tend to improve with annealing following irradiation, the counting characteristics did not recover so rapidly.

Appendix A

JOINT PROGRAM STAFF

Coordinator: J. C. French

Semiconductor Characterization Section

Dr. W. M. Bullis, Chief

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M. Cosman
Dr. J. R. Ehrstein
R. L. Gladhill
G. G. Harman
H. K. Kessler
Mrs. K. O. Leedy
R. L. Mattis
Dr. W. E. Phillips
J. L. Scales†
H. A. Schafft
A. W. Stallings
Dr. L. J. Swartzendruber
W. R. Thurber

Secretaries:

Miss T. A. Poole
Miss R. E. Young

Semiconductor Processing Section

Dr. J. A. Coleman, Chief

B. H. Audet
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W. K. Croll
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W. J. Keery
E. I. Klein
J. Krawczyk
T. F. Leedy
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Dr. A. H. Sher
L. M. Smith
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Electron Devices Section

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S. Rubin
M. Sigman
L. R. Williams

Secretaries:

Mrs. C. F. Bolton*
Miss B. S. Hope

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* Part Time
† 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.

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: R. C. Powell, 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
  - Nuclear 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

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† Participated in Round-Robin Experiments this Quarter

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 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. Solid-state technology - (B. H. Audet)

   Several Al-p-Si surface-barrier detectors are being developed and tested for use at very low temperatures by the Nuclear Spectroscopy Section. The principal difficulty with these detectors has been the poor adherence of contacts at temperatures near 4 K.

2. Quartz and glass fabrication - (E. I. Klein)
   a. A graded-glass seal and semi-ball joint was added to a PbS cell for the Radiation Thermometry Section.
   b. A glass manifold was constructed and repairs were made to several vacuum systems for the Vacuum Measurements Section.
   c. Repairs were made to a variety of equipment for the Building Research Division.
   d. Glass-to-kovar seals were fabricated for the Supply Division.

3. Ultrasonic fabrication - (J. Krawczyk)

   Additional elements for piezoelectric device arrays were made for the Naval Ship Research and Development Center An attempt to produce thin-film piezoelectric elements will be made during the next reporting period.*

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* NBS Project 4254431

Appendix D

JOINT PROGRAM PUBLICATIONS

L. J. Swartzendruber, F. H. Ulmer, and J. A. Coleman, "Direct Reading Instrument for Silicon and Germanium Resistivity Measurements," *Rev. Sci. Instr.* 39, 1858-1863 (1968).

H. A. Schafft, "Second Breakdown in Transistors," *Proc. Second Symposium on Reliability in Electronics,* Budapest, (1968), pp. 225-1 to 225-14.

W. M. Bullis, "Measurement Problems in Microcircuit Processing," *1968 Government Microcircuits Applications Conference, Digest of Papers,* pp. 215-217.

U. S. GOVERNMENT PRINTING OFFICE : 1969—341-024/105

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, $[illegible].00; foreign, $4.00*.

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

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.

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