NBS TECHNICAL NOTE

488

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

Quarterly Report
January 1 to March 31, 1969

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

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

Quarterly Report
January 1 to March 31, 1969

Edited by W. Murray Bullis

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

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† Related Projects

ABSTRACT

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

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

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

Quarterly Report
January 1 to March 31, 1969

1. INTRODUCTION

This is the third 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 last year to focus NBS efforts to enhance the performance, interchangeability, and reliability of discrete semiconductor devices and integrated circuits through improvements in methods of measurement for use in specifying materials and devices and in control of device fabrication processes. These improvements are intended to lead to a set of measurement methods which have been carefully evaluated for technical adequacy, which are acceptable to both users and suppliers, and which can provide a common basis for the purchase specifications of government agencies. In addition, such methods will provide a basis for controlled improvements in essential device characteristics, 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 10 deal with methods of measurement for materials; sections 11 through 15, with methods of measurement for process control; and sections 16 through 20, 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 the second report [6] background information was included for three additional tasks. During this quarter work was begun on a single new task on Gold-Doped Silicon, which had been identified during the April, 1968, program review.

One of the important functions of the Program is the stimulation of exchange of information on measurements and measurement methods among members of the various governmental and industrial communities which have an interest in high reliability electronic devices. Symposia on specialized topics are one means of accomplishing this function. At the January meeting of ASTM Committee F-1, D. E. Koontz, Chairman of Subcommittee X, proposed holding a Symposium on Silicon Processing under the joint spon-

sorship of the Committee and NBS. Preliminary plans call for sessions devoted to techniques and facilities for, and to properties and characterization of, both diffused and epitaxial layers. It is expected that the symposium will be held in June, 1970, at NBS, Gaithersburg, and that organizational plans will be completed by mid-1969.

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 PO9-0016. (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.
6. "Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, October 1 to December 31, 1968," NBS Tech. Note 475, February, 1969.

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: Two more industrial laboratories have completed measurements for the round robin based on the ASTM four-probe method [1] for measuring resistivity of silicon wafers which is being conducted in cooperation with ASTM Committee F-1 on Materials for Electron Devices and Microelectronics. Eight of the nine participants have now 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 have been completed and the results will be sent to the coordinator of the round robin. (F. H. Brewer)

Studies of the current dependence of the resistivity of silicon wafers as measured by the four-probe method have been resumed. A significant decrease in measured resistivity at high current levels was found in measurements on 60 Ω-cm *n*-type silicon crystal ends by a major silicon supplier. The results of a series of measurements on both wafers and crystal ends suggest that the change occurs as a result of injection from the current contacts. The effect was more pronounced on thicker specimens (crystal ends compared with wafers), when using closer spaced probes (25 mil rather than the standard 62.5 mil), and on longer lifetime material. If the change in measured resistivity were due to heating effects, it would be expected that the change would have been in the other direction. These observations reemphasize the need for establishing appropriate current levels and other characteristics when nonstandard conditions are employed in resistivity measurements.
(F. H. Brewer and W. M. Bullis)

In the study of the three-probe voltage breakdown method for measuring epitaxial layer resistivity, the possibility was investigated that actual resistivity variations over small distances were responsible for at least part of the scatter in breakdown voltage values. Measurements taken at 2-mil spacings over 20 × 40-mil grids indicated that there were indeed small scale variations in resistivity. However, several effects were observed which require investigation before any quantitative results can be obtained:

1. At random positions on the grids, it was necessary to form the contacts before any voltage breakdown could be seen.
2. There were noticeable fluctuations in the rate of voltage ramp, which were thought to affect the value of breakdown voltage.

An attempt was made to determine the conditions under which a distinct breakdown voltage could be obtained without prior need to form the contacts. Measurements were made as a function of probe radius and voltage ramp time. Random exceptions persisted but it was tentatively concluded that, under equal probe force, 1-mil radius probes require forming less often than do 2-mil radius probes (0.5 mil-radius probes were also tested but discarded due to fragility). The possibility exists, but has not been investigated, that the change in probe pressure which may occur when the probe radius is changed also may contribute to this effect. Also, contact forming was required more often with a nominal 100-μs long ramp than with a nominal 10-μs long ramp. Furthermore, separation of the reverse biased probe from the other two by a distance the order of 1 cm also caused need for contact forming. The reason is not understood nor has a critical distance been defined. Many of the random exceptions which were noticed could be eliminated, however, simply by inserting a new probe, or by "recleaning" the specimen surface.

Ramp-rate instability and the dependence of breakdown voltage on ramp rate were examined. Ramp rate fluctuations were not due solely to the power supply and it was not possible to eliminate them. Change in breakdown voltage was measured as a function of time to reach breakdown (ramp-rate time). As the time to reach breakdown was decreased from 40 μs to 6 or 8 μs there was an increasing sensitivity of breakdown voltage to further 1-microsecond decreases in ramp time. This higher relative sensitivity at shorter ramp times results in greater relative variation in breakdown voltages due to ramp-rate instabilities of any given magnitude.

Modifications were made which improved the general quality of the mount for the reverse-biased probe. It has not been possible to eliminate the probe vibration, believed to be entirely electrical in origin [3], which occurs when the reverse biased probe is taken to (or near) breakdown potential.

Initial work has been done to investigate the possibility of a more durable substitute for tungsten carbide as a breakdown probe. Doped synthetic diamond was considered and rejected since it is only available in very high resistivity ranges. Silicon carbide is being considered although no supplier of controlled doping level silicon carbide has yet been found.

ASTM has completed work on a tentative method of test [3] based on the voltage breakdown technique. The precision of this method is not adequate for its consideration as a reference or referee method. Work performed to date by Committee F-1 indicates that the likelihood of achieving significant improvements in precision of this method is small. Furthermore, the complex nature of the dependence of this measurement on various crystal and measurement parameters experienced here have led to a decision to shift the emphasis of this task toward other methods of measuring epitaxial layer resistivity.

An initial survey has been taken of suppliers of instrumentation needed for establishment of a spreading resistance capability to measure epitaxial resistivity. (J. R. Ehrstein)

Plans: It is expected that the final participating laboratory will have completed its measurements and that the results of the four-probe resistivity round robin will be tabulated and analyzed by the June meeting of ASTM Committee F-1.

Measurements will be continued on the dependence of four-probe resistivity values on current level and probe spacing.

Emphasis will be placed on the development of facilities for both the capacitance-voltage and spreading resistance methods for epitaxial resistivity measurement. Instrumentation for capacitance-voltage measurements will be selected. Consideration of instrumentation for spreading resistance measurements will be completed and orders for components will be placed. The search for a more durable probe material will be continued with emphasis on probes for spreading resistance measurement.

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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. A. A. Gundjian, "Electrostriction in Germanium," *Solid State Communications* 3, 279-281 (1965); "The Electrostrictor - A New Type of Electromechanical Semiconductor Oscillator," *IEEE Trans. Electron Dev.* ED-13, 866-873 (1966).
4. "Method of Test for Resistivity of Silicon Epitaxial Layers by the Three-Probe Voltage Breakdown Method" (ASTM Designation: F108-69T), to be published in the *1969 Book of ASTM Standards*. Single copies can be purchased from ASTM, 1916 Race Street, Philadelphia, Pa. 19103.

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: The surface photovoltage (SPV) equipment was combined with the steady-state photomagnetoelectric and photoconductivity (PME-

PC) equipment. The new arrangement eliminates the need to duplicate major items of equipment and it also centralizes the control instrumentation. (W. E. Phillips and A. W. Stallings)

Work on the measurement of carrier lifetime by the photoconductive decay (PCD) method [1] was continued. A draft summary of results to date was prepared, and the writing of a revised procedure for PCD lifetime measurements was begun.

A mathematical analysis was made of the effect of light turn-off on lifetime measurement. It was found that linear light decay, such as would occur using chopped light, produces no error in the lifetime measurement after the light is completely extinguished. This conclusion is consistent with work of Susila [2] and Penchina and Levinstein [3]. In the case of exponential light decay, which is approximately descriptive of pulsed light turn-off, the measurement error depends upon the ratio of the light turn-off time constant to the lifetime and upon the extent to which decay has proceeded when the measurement is made.

Work was begun on a contactless PCD method described by Nishizawa, *et al.* [4] and by Miyamoto and Nishizawa [5]. Experiments thus far indicate that the measurement is sensitive to the position of the specimen on the holder, elevation of the specimen above the holder, and lamp intensity. Although lifetime measurements on some specimens agree with the data obtained using the standard method, experimental conditions have not yet been found by which contactless measurements consistently agree with measurements made by the standard PCD method. Analysis from the standpoint of the equivalent circuit which the specimen and holder present to the oscillator circuit appears to explain qualitatively some of the deviations of the contactless method from the standard method. (R. L. Mattis)

As stated last quarter, the lifetime values obtained from voltage decay measurements were lower than the values measured by the reverse recovery technique. It was found that the lifetime values obtained are dependent upon the densities of injected carriers. A study of p-n junction theory to ascertain this dependence for each method is in progress. The effect of external circuit parameters on the measurement is also being studied. In the voltage decay method the lifetime is measured while the diode is open circuited following a pulse of forward current. On the other hand in the reverse recovery method, the lifetime is measured in the presence of a reverse current. Thus the voltage decay value is dependent only upon the recombination time of carriers; the reverse recovery value, however, is dependent also upon the external circuitry. Because of this, it was concluded that the voltage decay apparatus is operating satisfactorily. Preliminary measurements of carrier lifetime by the voltage decay technique in a 6 Ω-cm p-type silicon point-contact diode did not yield a straight-line voltage-time curve. The cause of this effect is still being investigated. (A. J. Baroody)

Plans: PME-PC measurements on indium antimonide will be resumed in the 100 K to room temperature range (see Section 7). Following completion of these measurements, SPV measurements will be resumed. In addition to comparisons with other methods for determining carrier lifetime in bulk silicon, application of the SPV method to the measurement of carrier lifetime in silicon epitaxial layers will be investigated.

The detailed procedure for measuring carrier lifetime by PCD will be completed. The experimental and analytical work on the contactless PCD method will be continued. Consideration will be given to the modification of the present apparatus to permit measurement of shorter lifetimes.

The theoretical and experimental investigation of injected carrier densities in diode recovery measurements will be continued along with studies of the effect of external circuit parameters. The reverse recovery circuitry will be modified to permit forward to reverse current ratios between 0.01 and 100. The dependence of the measured response time on the frequency and amplitude of the input square wave will be studied.

In connection with the Silicon Detector Task (see Section 20) the theory of diode recovery measurements as applied to point-contact, surface-barrier, diffused, and alloyed junctions will be studied. These types of junctions will be fabricated and lifetimes measured with the voltage decay method. Additional studies of voltage decay measurements on point contact diodes will be carried out.

---

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. G. Susila, "A Method for the Determination of Short Lifetime of Carriers in a Photoconductor from the Transient Photoresponse", *Indian J. Pure Appl. Phys.* 2, 44-47 (1964).
3. C. M. Penchina and H. Levinstein, "Measurement of Lifetimes in Photoconductors by Means of Optical Beating", *Infrared Phys.* 6, 173-182 (1966).
4. J. Nishizawa, Y. Yamoguchi, N. Shoji, and Y. Tominaga, "Application of Siemens Method to Measure the Resistivity and the Lifetime of Small Slices of Silicon," *Ultrapurification of Semiconductor Materials*, MacMillan Co., New York, 1962, pp. 636-644.
5. N. Miyamoto and J. Nishizawa, "Contactless Measurement of Resistivity of Slices of Semiconductor Materials," *Rev. Sci. Instrum.* 38, 360-367 (1967).

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: Problems that have been encountered in some *p*-type germanium and *n*- and *p*-type silicon specimens are still present. In an attempt to overcome these problems which are apparently related to the surface condition, improved cleaning procedures have been developed for use prior to treatment of the surface to reduce the surface recombination velocity. Such treatments are now being performed only on a lapped surface (5-μm alumina); treatments on etched surfaces have been found to give inconsistent results.

Attempts were made to reduce the relative number of carriers generated at the surface of the specimen by using light with a greater penetration depth. A monochrometer using a 40-W tungsten (quartz iodine) lamp was tried as a light source but the light intensity was insufficient. Detectable signals were obtained when an infrared transmission filter was used with an incandescent lamp. The surface related effects were still observable. (D. L. Blackburn)

Work on the theory of the photovoltaic method for measuring resistivity gradients along the diameter of circular specimens was continued. In this work it is assumed that a voltage dipole can be used to simulate the photovoltage generated by a light probe illuminating a region having a resistivity gradient along the diameter being scanned.

The dipole moment is implicitly dependent on the resistivity gradient and the excess electron-hole pair concentration introduced by the light probe. The theoretical results obtained for bar-shaped geometries may be applied to circular geometries where the light probe does not extend across the width of the specimen, if (1) the resistivity gradient perpendicular to the scanning direction is negligible near the diameter being scanned, (2) the effect of the circular geometry on the photoconductivity measurement is included, and (3) the effects of the shunting current on the photovoltage measurement are included.

Two sets of calculations were completed. These allow the theoretical results obtained for bar-shaped specimens to be applied to circular specimens. The effect of the geometry on the photoconductivity measurement was calculated without resorting to the use of an electromechanical analog mentioned in the last report. The existence of a shunting current and the way this current is perturbed by the specimen results in a spatial dependence of the photovoltage measured at the ohmic contacts at the ends of the measurement diameter. This dependence was calculated in the previous quarter. The shunting current also reduces the photovoltage at the region probed below that which would be calculated solely on the basis of the resistivity gradient. An estimate of this reduction was made by calculating the ratio of the resistance of the region probed to the resistance of the remainder of the specimen and considering these resistances to operate as a voltage divider for the photovoltage that would be measured if no shunting currents were present. The following expression was obtained for the resistivity gradient, $d\rho/dx$, which

applies to extrinsic wafers as long as the light probe is several diameters distant from the ohmic contacts:

$$\frac{d\rho}{dx} = \frac{3\pi}{16} \cdot \frac{B}{C} \cdot \frac{1}{1-(y/r)^2} \cdot \frac{tR^2}{r} \cdot \frac{V}{\Delta R} \qquad (1)$$

where: $B = \frac{q}{2kT}(1 + A)$,

q = charge of the majority carrier (C),
k = Boltzmann's constant (J/K),
T = absolute temperature (K),
A = ratio of the mobility of the majority carrier to mobility of the minority carrier,
$C = \left[\ln \frac{2 - (a/r)}{(a/r)}\right]^2$
a = radius of the small semicircular ohmic contacts at the ends of the measurement diameter (assumed to be equal),
y = distance of light spot from specimen center (cm),
r = specimen radius (cm),
t = specimen thickness (cm),
R = specimen resistance as measured at the ends of the measurement diameter (Ω),
V = photovoltage (V), and
ΔR = photoinduced change in resistance of the wafer (Ω).

Quantitative resistivity profiles of two *n*-type germanium wafers (one 10 Ω-cm, the other 40 Ω-cm) have been calculated using Eq. (1). The general features of the resistivity profiles agree well with those of the corresponding profiles obtained from four-point probe measurements. It was found in both cases, however, that the calculated resistivity gradients were about one-half the values indicated by the four-point probe measurements.

The success in extending the theoretical analysis to circular geometry has obviated the need for the previously planned measurements on bar-shaped specimens in which the light probe does not extend completely across the specimen width.

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

Plans: Measurements on other *n*-type germanium circular specimens will be made to substantiate the usefulness of Eq. (1) in the measurement of resistivity gradients in circular specimens. A more intense incandescent light source for the light probe will be used because of the generally smaller values of the photovoltage and photoconductivity that are measured on circular specimens, especially near the center.

Work will continue on the problems encountered on *p*-type germanium and *n*- and *p*-type silicon. Different surface treatments will be investigated. To reduce surface instabilities that may be causing these problems, a controlled ambient atmosphere may be required.

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: The additional gratings and filter to extend the wavelength range for the impurity photoconductivity work were installed in the monochrometer and calibrated. The specimen holder for the liquid helium cryostat was designed and fabricated. Provision was made for a filter which will be cooled to approximately the same temperature as the specimen to eliminate undesirable background radiation.

Analysis of the infrared absorption data obtained by the air reference method, the differential absorption method, and the single beam method [1] on silicon specimens indicates that results from the different methods are in agreement when appropriate correction factors are applied and allowance is made for the experimental errors of each technique. More experimental work on the methods does not seem necessary at this time.

The ratio of the 9-μm absorption coefficient due to oxygen in silicon at 80 K to that at 300 K was found to be 2.5 ± 0.4 from measurements on several specimens. It may be possible to compute a more precise value from the results of a round robin now being conducted by Committee F-1.

(W. R. Thurber)

Plans: Germanium specimens will be diffused with copper or gold and the photoconductivity resulting from these impurities will be measured at liquid helium temperature. Specimens with several different impurity concentrations, verified by neutron activation analysis or other means, will be used.

A summary report on the work on oxygen determinations will be prepared.

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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: Modifications to the Hall apparatus to permit automatic measurements on high-resistivity materials are being planned. It was found that the input impedance of the automatic system can be increased by using an electrometer input, but that measurement times are unusually long because of the large capacitance of the circuit. A proposed solution [1] is under consideration which will greatly reduce the measurement time.

The report concerning the use of a time-shared computer to control the Hall experiment has been revised and work on the final draft has begun. Work on the report concerning Hall measurements and their interpretation is continuing. (W. R. Thurber and W. M. Bullis)

Plans: The design of the modifications of the Hall apparatus will continue. The system will be used for measurements of high-purity germanium (see Section 10) and gold-doped silicon (see Section 8).

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

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1. D. Colman, "High-Resistivity Hall Effect Measurements," *Rev. Sci. Instrum.* 39, 1946-1948 (1968).

7. DEEP-LEVEL STUDIES

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

Progress: Previously it was reported that lithium added to specimens of *p*-type indium antimonide interacted with a deep-lying residual center. This was evident from measurements of Hall coefficient and electron and hole lifetimes.

A third indication of the interaction is shown by the Hall mobility data of Fig. 1. In the untreated specimen, 66A, the mobility increased as temperature decreased, reached a maximum, and then decreased as the temperature was further decreased. This was interpreted as the mobility that would result from a combination of acoustic phonon and ionized impurity scattering. In the lithium-treated specimens, the mobility did not show the decrease at low temperatures attributed to ionized impurity scattering. Instead the mobility continued to increase as the temperature decreased. This was clear indication that the scattering center had been neutralized by the addition of lithium.

The difference between the two lithium-treated specimens was that 64B received a bake-out treatment, to drive off excess lithium, whereas 66B did not. Consequently the latter specimen would be expected to contain more interstitial, electrically neutral lithium that may have caused reduction in mobility due to neutral-impurity scattering. The observed

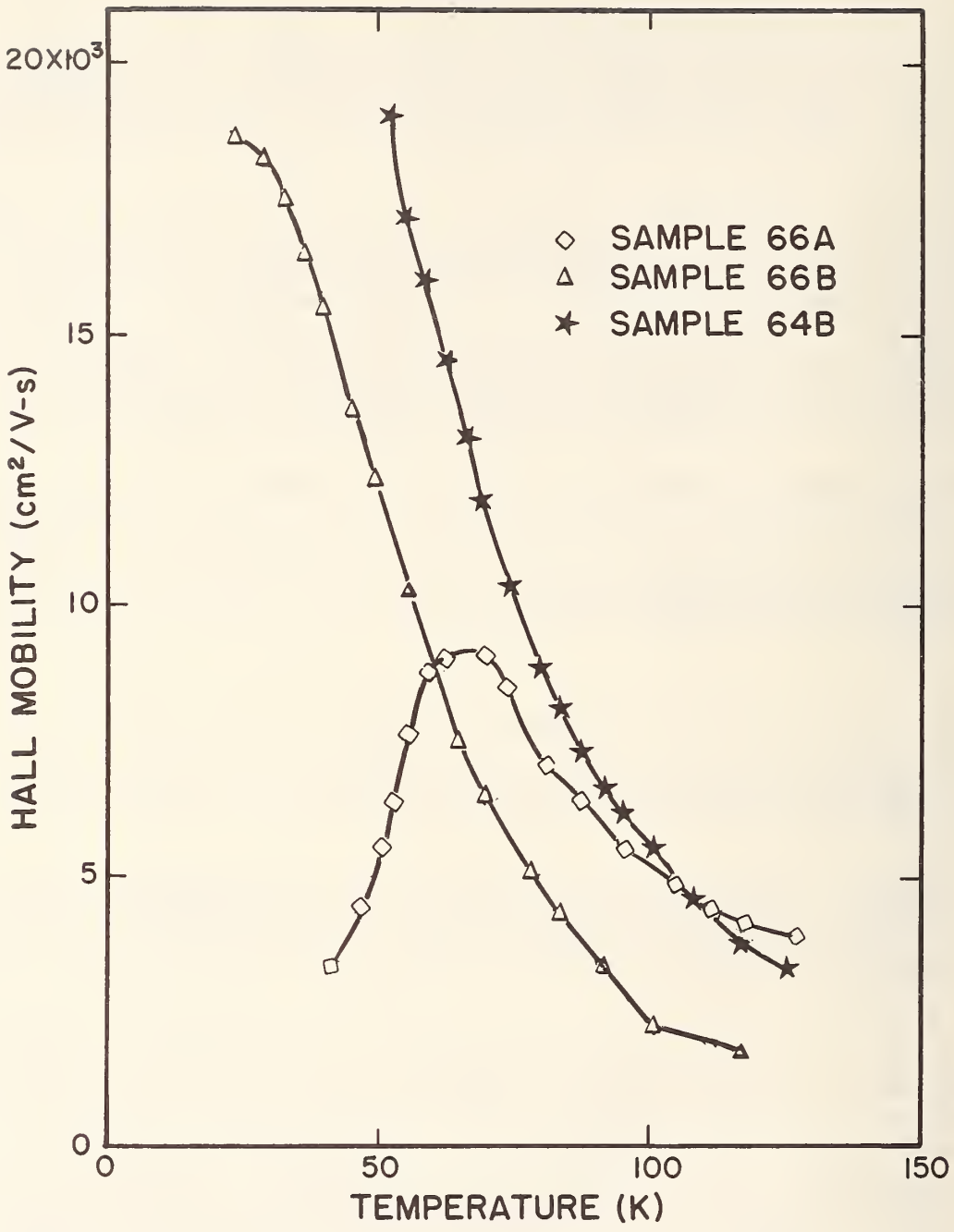

Fig. 1. Hall mobility vs. temperature for three specimens of *p*-type indium antimonide. Specimen 66A was untreated; lithium was added to specimens 66B and 64B.

difference in mobility between samples 66B and 64B was fairly constant over the temperature range shown, consistent with the neutral-impurity scattering hypothesis.

Plans: Additional PME-PC lifetime measurements will be made on treated and untreated specimens in order to verify certain features of the model. The low-temperature Hall effect will be further investigated to characterize the shallow acceptor level more completely. After the measurements are completed a final report on the task will be prepared.

8. GOLD-DOPED SILICON

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

Background: Gold is frequently introduced into high-speed silicon devices in order to reduce the carrier lifetime [1]. Gold may also be introduced into high-resistivity silicon in order to increase the resistivity still further and, at the same time, to permit high-resistivity to be maintained at temperatures below room temperature.

The extensive early work on this subject has been summarized in a review paper [2]. Although this work led to a model which explains the gross features of gold-doped silicon, extensive disagreement between the theoretically predicted characteristics and the measured characteristics has been noted. Subsequent work [3-5] has clarified some aspects of the problem but has not led to a thorough understanding.

The approach to be employed in this study is the measurement of resistivity, Hall effect, and minority and majority carrier lifetimes in gold-doped silicon. Both *n*- and *p*-type silicon will be used. Wafers will be selected from crystals with a variety of room temperature resistivities. It is expected that about 10 crystals of each type with resistivities ranging between 0.01 and 5,000 Ω-cm will be employed in the study. Gold will be introduced by diffusion at various temperatures; the resulting gold concentration will be determined by an independent technique such as neutron activation analysis. At least 5 different gold concentrations will be introduced into specimens from each of the crystals. Electrical measurements will be made at room temperature and at such other temperatures as may be appropriate.

Progress: Design of the initial experiments has been completed. Construction of a diffusion furnace for introducing gold into the silicon wafers at temperatures between 750 and 1350°C has been started. A preliminary search of literature in the period following the review article has been completed. (W. M. Bullis)

Plans: Following completion of the diffusion furnace facility, procedures for introducing gold into silicon wafers will be established.

It is expected that this phase will be completed and that electrical measurements will begin during the third quarter of this year.

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1. A. E. Bakanowski and J. H. Forster, "Electrical Properties of Gold-Doped Diffused Silicon Computer Diodes," *Bell System Tech. J.* 39, 87-104 (1960).
2. W. M. Bullis, "Properties of Gold in Silicon," *Solid-State Electronics* 9, 143-168 (1966).
3. W. M. Bullis and F. J. Strieter, "Electrical Properties of *n*-Type Silicon Doped with Gold," *J. Appl. Phys.* 39, 314-318 (1968). This paper presents resistivity and activation analysis data.
4. A. Vapailli, "Silicium Dopé a l'Or: Étude de la Conductivite et de la Durée de Vie des Porteurs en Excès," *Ann. Phys.* 3, 13-25 (1968). This paper presents Hall effect, resistivity, and lifetime (conductivity decay) data on samples in which the gold concentration exceeds that of the shallow dopant.
5. S. F. Cagnina, "Enhanced Gold Solubility Effect in Heavily *n*-Type Silicon," *J. Electrochem. Soc.* 116, 498-502 (1969).

9. HIGH FIELD EFFECTS

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

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

(G. G. Harman)

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

10. SPECIFICATION OF GERMANIUM†

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

Progress: Due to the large number of germanium crystals which have been received for examination (presently 65) and the resultant quantity of data generated, more reliance has been placed on computer analysis of the data. To aid in determining if meaningful correlations exist between the results of various measurements made on the crystals, computer-generated "scatter plots" have been made. These plots have

shown that for 15 specimens, no direct correlation exists between lithium ion drift mobility at 24°C and electron drift mobility at 77 K in contrast to previous reports by Armantrout [1].

(A. H. Sher and H. E. Dyson)

Nomographs have been prepared which relate the drifted depth of Ge(Li) structures to time, temperature, and the applied voltage during drift. These charts are revisions of previously published ones [2], and take into account recent experimental measurements of the lithium ion drift mobility in germanium between 24 and 60°C [3].

A model for the drift rate of lithium in germanium is being investigated. It attempts to explain drift rates which are less than expected on the basis of theory in terms of the loss of lithium, possibly by precipitation, in the compensated region during drift. If a term proportional to the drifted depth, W, is added to the equation for the rate of change of W (cm/s), one obtains:

$$dW/dt = \mu_{Li}E - \lambda W = \mu_{Li}E - W/\tau \qquad (1)$$

where μ_{Li} is the lithium mobility ($cm^2/V\text{-}s$), E is the electric field (V/cm), and λ is the "loss rate constant" equal to the reciprocal of the "loss time constant," τ (s). Integration of Eq. (1) yields:

$$W = [\mu_{Li}V\tau(1 - e^{-2t/\tau})]^{\frac{1}{2}}. \qquad (2)$$

Drift curves can be fitted with Eq. (2) to obtain values for both μ_{Li} and τ. For short drift times ($t \ll \tau$), i.e., for germanium in which little precipitation takes place, expansion of the exponential term in Eq. (2) yields the drift equation for the "ideal" case:

$$W = (2\mu_{Li}Vt)^{\frac{1}{2}}, \qquad (3)$$

and μ_{Li} can be obtained from the curves in the usual way.

The postulated lithium loss mechanism in germanium during drifting is being examined in greater detail to include effects of impurities, vacancies, and lithium solubility. A manuscript which discusses the aforementioned nomographs as they apply to the drifting of germanium gamma-ray detectors and the problem of lithium "mobility" versus "driftability" [Eq. (3) vs Eq. (2)] is being prepared for publication.

(A. H. Sher and J. A. Coleman)

The results of the measurements of oxygen concentration in germanium by infrared absorption, lithium precipitation, and lithium mobility are being critically examined in preparation for a report on the usefulness of these methods. Results obtained by measurements of infrared absorption have been refined by using the complete transmission expression, which includes the effects of multiple reflections, in the calculation; values of oxygen concentration thus obtained are generally lower than in the previously uncorrected cases.

The effect of the acceptor concentration on the determination of oxygen concentration by lithium mobility measurements is being investigated. In germanium, the acceptor concentration has a more profound effect in the calculation of oxygen concentration than in the case of silicon. The equation used is [4]:

$$N_O = n(1 - f) + C(f^{-1}-1) \qquad (4)$$

where N_O is the oxygen concentration, n is the total donor concentration (taken to be equal to the initial acceptor concentration as suggested by Fox [5], f is the ratio of the observed lithium ion drift mobility to the maximum drift mobility (3.04×10^{-10} cm^2/V-s at 23.8°C [3]), and C is the dissociation constant for the LiO^+ complex (3.4×10^{12} cm^{-3} at 27°C [6]). In *p*-type germanium, the range of acceptor concentration encountered in the starting materials commonly used is 1×10^{14} to 9×10^{14} atoms/cm^3 (30 to 5 Ω-cm). Plots of Eq. (4) for various resistivities are shown in Fig. 2. For cases in which $N_O < 10^{15}$ atoms/cm^3, the dependence of Li mobility on oxygen concentration is affected greatly by the acceptor concentration because the first term in Eq. (4) is the dominant one. The situation in *p*-type silicon is reversed; $C = 5.8 \times 10^{15}$ cm^{-3} [7], and the acceptor concentration of material commonly used for lithium drifting is in the range of 1×10^{13} to 1×10^{14} atoms/cm^3. Thus, the second term in Eq. (4) is expected to be the dominant one, and the acceptor concentration does not seriously affect the relationship between oxygen concentration and lithium mobility. (A. H. Sher, W. K. Croll, and W. R. Thurber)

Measurements of Ge(Li) detector characteristics are proceeding using the collimated gamma-ray beam (^{137}Cs). The secondary collimator has been modified so that the beam width at the aperture is now 0.75 mm, and the secondary collimator itself is adjustable in two perpendicular axes. Measurements of beam spread as a function of distance from the secondary collimator using dental x-ray film indicate that the beam width at 6 cm is approximately 1 mm. Work is proceeding to allow stepping of the detector bias through preselected values with external control of the multichannel analyzer to permit the automatic recording of data for measurements of detector resolution and peak shift as a function of bias at a specified collimated beam scanning position.

Preparation of the manuscript on the effects of separate collection of electron and hole currents in Ge(Li) detectors on the measurement of the Fano factor has been suspended temporarily pending implementation of a computer program for data analysis. (A. H. Sher and W. J. Keery)

Visits to four national laboratories, a military laboratory, and a leading commercial detector manufacturer were made to review work on the fabrication of Ge(Li) detectors, use of detectors in nuclear physics, materials problems, and methods of measurement useful for specification of detector-grade material. (A. H. Sher)

Plans: Lithium mobility studies (including effects of surface type and detector performance measurements with emphasis on carrier trapping utilizing a collimated gamma-ray beam and subsequent analysis of the peak

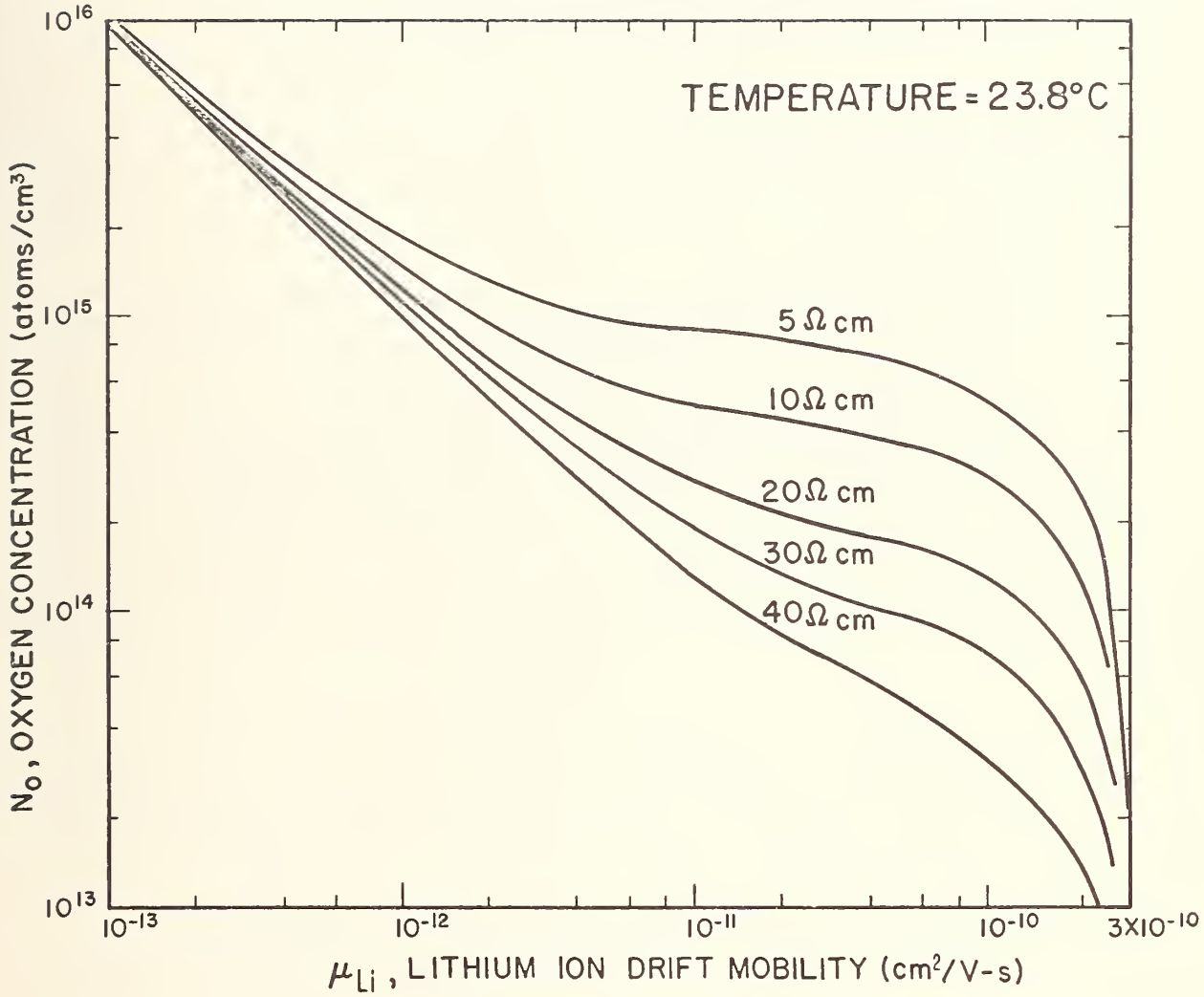

Fig. 2 Oxygen concentration, N_O, as a function of lithium ion drift mobility, μ_{Li}, at 23.8°C for different specimen resistivities, from Eq. (4).

shape will be continued. Impurity photoconductivity measurements will be made on germanium crystals which charge-trapping experiments have indicated may contain undesirable impurities (see Section 5). Infrared response measurements at 77 K will be made on Ge(Li) detector structures fabricated from the same material in order to determine if correlations exist between the two infrared analytical techniques.

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

1. G. A. Armantrout, "Correlation Between Lithium Drift Mobility and Minority-Carrier Drift Mobility in Germanium," *IEEE Trans. Nucl. Sci.* NS-13, No. 3, 370-372 (1966).
2. J. Takacs, "The Depletion Depth of Lithium-Ion Drift Detectors as a Function of the Time, Voltage, and the Diffusion Coefficient," *Nucl. Instr. and Meth.* 33, 171-172 (1965).
3. A. H. Sher, "Lithium Ion Drift-Mobility in Germanium," *J. Appl. Phys.*, to be published (May, 1969).
4. H. Reiss and W. Kaiser, "Solid State Reactions of Oxygen in Silicon," *Properties of Elemental and Compound Semiconductors*, H. C. Gatos, ed., Vol. 5, Interscience, New York, 1960, pp. 103-119.
5. R. J. Fox, "Lithium Drift Rates and Oxygen Contamination in Germanium" *IEEE Trans. Nucl. Sci.* NS-13, No. 3, 367-369 (1966).
6. R. J. Fox, "Determination of Oxygen in Germanium by Lithium Precipitation, *Semiconductor Nuclear-Particle Detectors and Circuits*, W. L. Brown, *et. al.*, eds., National Academy of Sciences, Washington, D. C. 1969, pp. 198-200.
7. E. M. Pell, "Study of the Li-O Interaction in Si by Ion Drift," *J. Appl. Phys.* 32, 1048-1051 (1961).

11. METALLIZATION EVALUATION

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

Progress: Current efforts are directed toward aluminum metallization vacuum evaporated onto silicon or silicon dioxide. The vacuum evaporation system has been modified so that aluminum films may be produced under carefully controlled conditions of substrate temperature, deposition rate, and film thickness.

Considerable work has been accomplished on the control of the substrate temperature. The heater being used consists of an array of quartz infrared lamps with a suitable reflector. The temperature sensor is a thermocouple carefully sandwiched between two silicon wafers. The size, surface, and mass of this sensor have been chosen so that the properties of the sensor essentially duplicate those of the silicon working wafers. Thus far, substrate temperatures of 580°C have been attained with reliable control although these is some question of temperature uniformity across the wafers. Also, low thermal inertia has been obtained by using quartz jigging for supporting the silicon work pieces and the temperature sensor. Calibration of the instrumentation for monitoring film thickness and deposition rate and of the substrate temperature sensor is presently under way.

A literature search on thin film adhesion was initiated. The object is to achieve a comprehensive understanding of the work completed in recent years on adhesion testing and adhesion measurements. Information on adhesion testing by way of tensile forces applied perpendicularly to the metal film-substrate interface is of special interest.

(W. K. Croll and J. Oroshnik)

An interim method for measuring thickness of oxide and metal films is being prepared (see Section 15).

Plans: Instrumentation for the control of substrate temperature, deposition rate, and film thickness are expected to be completed.

Presently under consideration is a method for making a bond to small aluminum dots for a normal pull test for adhesion. The requirements placed on this bonding scheme are that the aluminum-substrate interface and the bulk of the aluminum film shall in no way be disturbed physically or chemically by the bonding process. In addition, an adhesion test using the method of film scratch with a stylus will be set up; evaluation studies of this method will begin. The literature search on thin film adhesion will continue.

12. 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: Detailed evaluations were made of diffusions that were produced using the gaseous diffusion system. The *p*-type (boron) furnace was used to produce a predeposition for a base diffusion of approximately 10 Ω/square. A problem in its operation, however, is the formation of B_2O_3 glass which fouls and shortens the life of the diffusion tube. This will be minimized by selection of an optimum oxygen flow rate.

Less difficulty has been experienced with the *n*-type (phosphorous) diffusion furnace. Diffusions were produced with sheet resistances between 1.7 and 5.0 Ω/square in a 30-min diffusion cycle using different phosphine concentrations. Junction depths of approximately 2.5 μm were obtained; such a diffusion is suitable for a transistor emitter.

Laminar air-flow hoods were installed in the photoresist area so that wafers can be processed in dust-free conditions. A deionized water system is being installed by laboratory personnel in this area to facilitate the etching of oxides and metallizations. A liquid nitrogen boil-off system is being installed to provide a constant source of pure, dry nitrogen carrier gas for the diffusion furnaces. Improvements has also been made in the metallization facilities (see Section 11).

(T. F. Leedy and J. Krawczyk)

Plans: Efforts will be concentrated in two areas: photomasking and junction delineation. A mask alignment system suitable for producing simple geometries will be obtained. This will permit metallization patterns to be aligned over oxide openings.

Accurate determination of junction depths is necessary to characterize fully the diffusion parameters. Staining techniques proposed by NASA [1] and ASTM [2] for delineating the location of diffused junctions will be studied and standard techniques will be developed.

Ellipsometric studies will be deferred to a future date when more meaningful determinations can be made. These were not begun last quarter since a reappraisal led to the conclusion that this information is not essential now.

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1. Methods 6030D and 6060 of NASA-STD-XX-3, *Test Standards for Microcircuits*, Draft, December 1, 1968.
2. "Method of Test for Thickness of Epitaxial or Diffused Layers in Silicon by the Angle Lapping and Staining Techniques," now in final stages of consideration by ASTM Committee F-1.

13. 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: Eighteen different organizations were visited during the last quarter. In all, 30 visits have been made to a total of 27 organizations (13 industrial semiconductor device suppiers and users, 6 government installations, and 8 equipment manufacturers). This essentially completes the field visits which were planned to review the current status of microwire bonding and procedures for evaluating such bond systems. Analysis of the information obtained during these visits has begun. (H. A. Schafft, G. G. Harman, and H. K. Kessler)

A tentative outline of classes of key words to be used to identify entries in the bibliography has been selected and is shown below:

I. Test Methods
   - A. Type
     1. Destructive
     2. Non-Destructive
   - B. Description and Procedures
   - C. Evaluation and Correlation
     1. With Laboratory or Field Stress
     2. Between Test Methods

II. Materials and Fabrication (pertinent to bond system quality)
   - A. Material Characterization
   - B. Bonding Procedures
   - C. Post-Bonding Procedures

III. Failure Analysis
   - A. Origin of Failures (Mechanisms)
   - B. Types of Failures (Modes)
   - C. Stress Condition
     1. Mechanical
     2. Electrical
     3. Thermal
     4. Radiation

For each main class a key work will be included to identify the wire bond system considered. Some of the test methods which have already been found and will be identified with key words are: visual inspection, pull, shear, centrifuge, vibration, electrical noise, low current-voltage, thermal cycle, thermal shock, and ultrasonic monitoring. Some of the mechanisms responsible for failure of the bond system which will be identified with key words are: intermetallic compound growth, the Kirkendahl effect, wire anneal, dispersion of the wire alloy, wire deformation, work hardening, and contamination. Finally the types of failure modes will be identified according to the location of the break.

The papers to be included in the bibliography will fall in one or more of the three main subject categories. All papers dealing with test methods will be included in the bibliography; the number of such papers is not expected to be large. Selected papers dealing with the other subject categories will be listed; these will include both papers of a review or general nature as well as those concerned with specific devices.

To aid in the use of the bibliography, the relative importance of the papers in the second and third categories will be indicated. If only a portion of the paper is of interest that part will be so identified. Appropriate key word indexes and an author index will be included with the bibliography. Although considerable progress has been made in locating some of the report literature, reports and papers both on correlation of bond test results with device failure and on the types of experimental work being carried out under this task have been very sparse. Contributions of reports on these subjects from readers would be appreciated by the compiler of the bibliography.

The list of unclassified work units relating to the making and testing of wire bonds was received from the DoD Work Unit Data Bank maintained by the Defense Documentation Center (DDC). The list was too short to be useful, and it was established that a more detailed description of project requirements will be necessary if a list of the desired scope is to be obtained. (H. A. Schafft)

Construction was completed of a versatile wire bond pulling system which is designed to hold the substrate or device at any angle with respect to the pulling force. Pulling is accomplished by an electrolytically etched 2-mil wire hook or by the special wire gripper described below. Precision alignment of the hook with respect to the wire is accomplished by mounting the hook on a micropositioner (A of Fig. 3). A switch-controlled motor that can be programmed to stop at a predetermined point does the actual pulling. Thus the important parameters of the system are independent of the operator. The pulling force is measured by a gram gauge dynamometer (C), but other measuring instruments, electronic or mechanical, could be installed on the micropositioner platform.

In order to determine the strength of a single bond, it has long been assumed that the wire must be pulled at the angle the bond was made in order to minimize perturbing effects such as peeling of the bond which are pull-angle dependent. For a thermal compression bond this direction is perpendicular to the surface, and for an ultrasonic bond it is typically 30° from the surface. As generally practiced, bond pulling is preformed with a hook placed somewhere near the middle of the loop. However, in devices the die and post are frequently at different levels and the exact loop height and length are seldom known. Thus it is almost impossible to determine the actual direction and magnitude of the force applied to the bond.

A very simple method has been devised to grip the wire without deforming it so that it can then be pulled in any desired direction. In

order to do this, a hairpin shaped loop of 5-mil nichrome wire was attached to the arm of a gram gauge. The tip of the loop was electrolytically etched down to a 2-mil diameter. This tip was then bent in the shape of a foot. A heating power of about 0.3 W was dissipated in the wire and the tip was wetted with a small dab of a special hot-melt glue. In operation the heated tip of this hot-melt puller is lowered over the free end of a wire that has one end bonded to a substrate. The power dissipation is terminated and the bond tensioned or pulled after the puller has cooled. The thermal time constant of the system is a fraction of a second. The choice of glue is very important. It is necessary to use one that has a discrete melting point and that is quite hard when cold. Most such glues will bond well to a 5-mil length of 1-mil diameter aluminum wire. However, gold wire is more difficult for glues to grip and larger bonding areas have been required to obtain sufficient strength for pulling the bond.

The hot-melt puller can also be used to test metallization adherance to a substrate. Considering an appropriate glue as having several thousand pounds per square inch tensile strength, the force applied to an aluminum metallization will be higher than is applied by

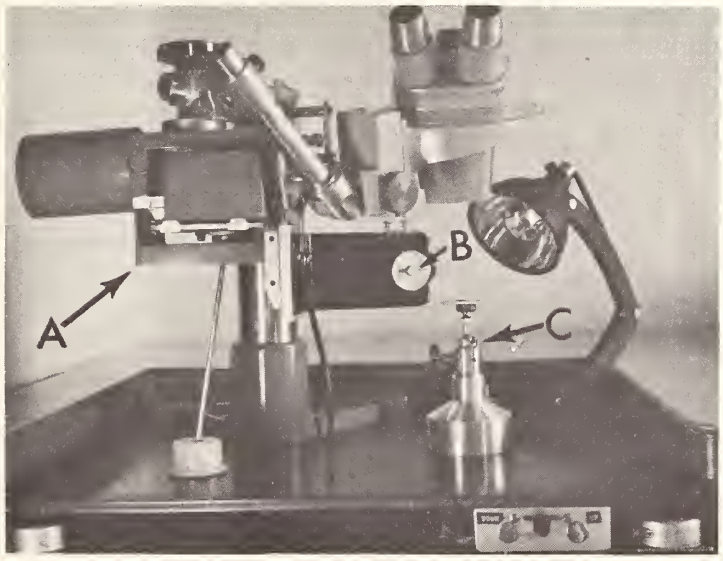

Fig. 3 Mechanical Wire Bond Pulling Machine

A-Micropositioner
B-Gram gauge dynamometer
C-Adjustable fixture for holding specimens.

the usual adhesive tape tests and can be applied to a much smaller, controlled area.

The hot-melt puller has been used to measure the tensile strength of 1-mil aluminum bonding wire. In initial tests with wire from a single spool, repeated pulls to rupture on the same (decreasing) lengths of wire gave approximately the same tensile strength for each successive pull. If this tentative result is substantiated the result indicates that prestressing the wire, even to the breaking point, does not appreciably weaken the remaining wire.

It is well known that a burn-in screen [1] anneals and weakens the wire-bond system. Information obtained during visits to manufacturers and from unpublished reports indicates that post burn-in bond pull strengths of from 2 to 6 g are typical for 1-mil diameter aluminum wire. Some state that 2 g *final* strength is necessary while others are satisfied with prestressing the bonds to 1 or 2 g *before* burn-in. Generally the loop height and wire length are not considered, so that the actual force applied to the bonds is unspecified. Many laboratory tests are accelerated by using temperatures much higher than the maximum specified for the device and thus may not be related to typical life conditions. Without definite knowledge of the weakening it is not possible to determine the maximum strength that should be expected from a well made bond after burn-in, or after several thousand hours of normal operation in a system. An investigation was started and thus far two samples containing about 150 bonds each, stitched back and forth across a large substrate, have been tested. Initially, every other bond was pulled, then the substrates were baked at 150°C for 168 hours and the remaining bonds were pulled. The bond strength had decreased significantly after heating, as expected.

An investigation was started concerning the use of aluminum ribbon wire for ultrasonic bonding. This was to have been initiated with ribbon having the same cross section (1.5 mil × 0.5 mil) as 1-mil round wire. However, long delivery schedules from the vendor have prevented this, and tests were started with 2.2-mil × 0.5-mil wire. A special ultrasonic tool was used that had a groove parallel to the wire. The resulting normalized bond strengths of the ribbon were higher than for round wire, even though it was determined that two dimensions of the tool should be changed for optimum bonding. This is encouraging, but since neither the wire cross sections nor the tools used were directly equivalent it is not possible to draw definite conclusions at present.

One major argument expressed against the use of ribbon wire was dispelled. Some have assumed that since this wire is wider in the beginning it will undergo a normal deformation and require a larger bonding pad than equivalent round wire. This would in turn lead to high capacitance and degraded high frequency characteristics. The fact is that ribbon wire conforms easily to the pad. The bond requires less pressure and less ultrasonic energy per unit area and results in a bond area that is less than or equal to that for a round wire of the same cross section.

It is well known that the use of wire with higher tensile strength (~20 to 24 g) results in higher bond strength. However, the higher ultrasonic energy and pressure required to form the bond result in damage to the die. Because less deformation occurs with ribbon wire, it should be possible to use high tensile strength wire without causing significant damage to the die. Hence it is expected that considerably higher bond strengths than are possible with round wire can be achieved.

Modifications were made on several bonding machines. Tungsten carbide capillaries and sapphire flame cut off nozzles were added to two older thermal compression ball bonders. The wire feed system and the ultrasonic horn were redesigned on an ultrasonic bonder.

It was decided not to modify an existing bonding machine in order to incorporate the 100 percent non-destructive pull test discussed previously. The basic operation sequence as well as physical location of parts would have had to be changed. This would entail a lengthy redesign which would tie up the machine and delay other objectives of the program. (G. G. Harman, H. K. Kessler, and K. O. Leedy)

Plans: A few additional visits will be made to suppliers of bonding equipment and materials. New requests for a search of the Work Unit Data Bank and for a bibliography of reports will be submitted to DDC. The information obtained from the field visits and from the papers and reports obtained for the bibliography will be analyzed and summarized. The first draft of this paper and the collection of papers for the bibliography should both be near completion by the end of the next quarter.

Investigation of the tensile strength of wire and wire bond systems after burn-in at various temperatures will continue. In some cases devices will be operated for greatly extended periods in order to simulate normal system operation. These tests will include wire obtained from several different sources. Cross correlations will be made between the optimum-angle pull-strength, the loop height, and the length of normal stitch bonds. The work on ribbon wire will continue when new bonding tools and wire are received.

The effect of metallization sintering time and temperature on ultimate wire bond strength will be investigated. Typical manufacturing practice varies in time from a few minutes to over a half an hour and in temperature from about 450 to 550°C. Many combinations of these conditions may be adequate from the standpoint of metallization adherance or electrical contact resistance, but there could be a considerable effect on bonding to the surface.

Some ultrasonic bond monitoring systems will be investigated and compared with wire pull and "push" tests. A high current pulse method for non-destructive wire bond testing will be investigated.

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1. Method 1015 of "Test Methods and Procedures for Microelectronics," MIL-STD-883, 1 May 1968.

14. DIE ATTACHMENT EVALUATION

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

Progress: The literature search on the techniques utilized in evaluating the uniformity and quality of semiconductor device die attachment was continued. This search is being carried out in conjunction with the Wire Bond Evaluation search (see Section 13). Visits to two industrial organizations were conducted to discuss methods of evaluating semiconductor device die attachment.

It was determined that the fabrication of semiconductor diodes with known voids is feasible and can be undertaken.

A method of introducing voids in the eutectic solder layer is to modify either the eutectic alloy preform or the evaporated gold layer to give various void configurations or to simply bond the chip off-center to the header (see Figure 4). Another technique for void generation is the introduction of impurities onto the bonding surfaces in order to produce poor wetting. This technique lacks the needed control and therefore should be used only as a secondary method.

(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 determination of chip size, mounting configuration, and case type for the test diodes will be made. Fabrication of diodes with known voids will begin and a facility for void detection by radiographic means will be established. Initial studies will use devices bonded by a eutectic process. The test diodes will be designed so that the major heat transfer path is through the solder layer to the header, thus insuring maximum thermal sensitivity to voids. Both thermal resistance and transient thermal response measurements will be made by a selection of techniques to determine the sensitivity of the measurement to voids.

15. NASA MEASUREMENT METHODS

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

Progress: The interim method for measuring oxide thickness by the multiple-beam interference method [1] has been prepared. The method is being revised to include metallization thickness measurements.

(W. E. Phillips)

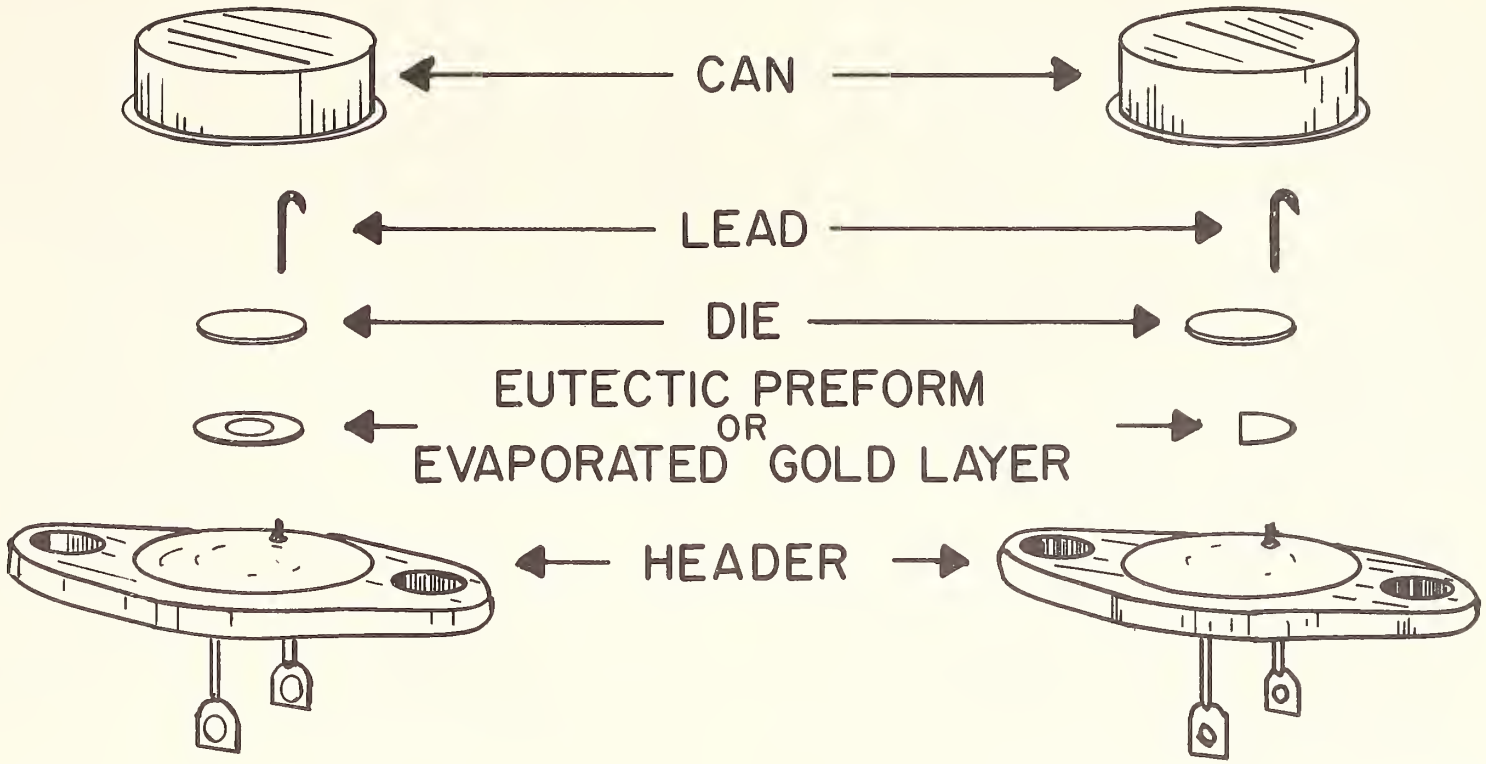

Figure 4. Exploded view of diode package with controlled voids in the solder layer.

Many different tests for fine and gross leaks in semiconductor cans have been proposed by such groups as ASTM Committee F-1, NASA, and the military. These can be grouped into a few distinctly different methods. The proliferation of tests has resulted in numerous variants of the basic methods with relatively minor but troublesome differences. Data on the various test methods is being collected and summarized. In addition, literature on limitations and repeatability of the various tests is being collected [2] and industry positions (as expressed through such groups as the Electronic Industries Association) are being identified. This material is being assembled to assist Committee F-1 to determine what, if any, action it should take in documenting the characteristics and applicability of the various methods.

A review of the December 1, 1968, draft of NASA-STD-XX-3, "Test Standards for Microcircuits" has been initiated. Several additional tests included in this document have been selected for detailed review. These include tests for junction depth (and base width) and for conductivity of thin metallic films. Active work related to both these measurements is underway in connection with other tasks (see Sections 2, 11, and 12). In addition, draft methods are being considered by Committee F-1. The purpose of the review of these methods is to identify problem areas and missing information which may impede the development of standard forms. (W. M. Bullis)

Plans: The interim method for measuring oxide and metallization thickness will be completed and sent to NASA for review.

The review of the draft of NASA-STD-XX-3 will be completed. Detailed review of methods for measuring junction depth will begin. Material on leak tests will be presented to Committee F-1 for action.

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1. S. Tolansky, *Multiple-Beam Interferometry*, Oxford University Press, London, 1948, pp 147-150.
2. The collection process was considerably simplified by the appearance of a review "Guidelines for Hermetic Seal Testing of Semiconductor Devices," prepared by Defense Electronics Supply Center, Dayton, Ohio, 1 April 1969.

16. SECOND BREAKDOWN

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

Progress: It was determined that the possibility of radiation-induced second breakdown is considered to be a significant problem in some environments. Hence, it was decided that a new review of the status of the second breakdown field with particular emphasis on radiation-induced effects is needed.

Plans: It is expected that the first draft of the manuscript on "Failure Modes" will be completed before the next meeting of the JEDEC Committee JS-6 on Power Transistors in June. This is to be included in a proposed JS-6 publication titled, "Recommended Standards for Power Transistors."

The review of the status of the second breakdown field will begin after work on the wire bond bibliography and survey (see Section 13) is complete.

17. THERMAL PROPERTIES OF DEVICES

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

Progress: The literature search and the review of the methods of measurement of thermal impedance of semiconductor devices were continued. It has been found that the majority of articles and reports on thermal measurements of semiconductor devices do not describe the proposed measurement techniques in sufficient detail for effective utilization. In some cases the information reported is not applicable to devices manufactured by present techniques. (F. F. Oettinger and M. Sigman)

Further modifications were made on the h_{FE} and V_{EB} thermal resistance measuring circuits to improve the accuracy and precision of the measurement as well as to increase the maximum temperature capabilities of the system. The following modifications were completed:

1. A high-temperature heat sink and temperature controller were designed and fabricated. The new heat sink is capable of raising a transistor in a TO-66 can to a temperature of 200°C for calibration purposes. It is also capable of

being held at 25±½°C while controlling the temperature of a transistor dissipating a maximum power of 35 W.

2. The mode of operation of the device under test, utilizing the base-emitter voltage as the temperature sensitive parameter, was changed from the common-base (V_{EB}) to the common-emitter (V_{BE}) configuration to facilitate direct comparison with the pulsed h_{FE} measurement technique.
3. A solid-state relay was designed and constructed to replace the mercury-wetted relay system used in the V_{BE} measuring system. This modification greatly reduces the oscillatory condition caused by the termination of the power pulse making it possible to measure V_{BE} within 20 μs of the pulse termination.

It had been decided that transistors with a chip 60 mils on a side and a power dissipation of 20 to 35 W will be used initially in this task and in the Thermographic Measurements task (see Section 18). Samples of devices with geometries suitable for these measurements have been obtained.

Initial comparison measurements made on a 20 W *n-p-n* silicon power transistor using the pulsed h_{FE} and the V_{BE} techniques showed, as anticipated, a 25 percent greater thermal resistance with the pulsed h_{FE} measurement method. The comparison was possible only for voltages below the inflection of the dc h_{FE} vs collector-emitter voltage curve caused by device thermal instability [1].

Visits to three industrial organizations (including device manufacturers and users) and one government laboratory were conducted to discuss various aspects of the problem of thermal measurements of semiconductor devices. The information obtained during the visits is being studied. (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. Both reports on government-sponsored studies and the open literature will continue to be searched.

Additional circuit modifications will be made to reduce the switching transients caused by the termination of the power pulse. Thermal resistance measurements will then be resumed on the 20 W transistors. This will include a study to determine the effects on the measured thermal resistance of the magnitude of low-level metering current and the time delay between the termination of the power pulse and the actual measurement of the temperature sensitive parameter. Thermal resistance versus power dissipation as measured using various V_{BE} techniques will be compared with the steady-state measurement of h_{FE} versus power to determine if the break points (the notable increase of thermal resistance or the decrease of h_{FE} with power), which are thought to indicate the onset of lateral thermal instability [1][2], show significant correlation when measured by the various methods.

The evaluation of the technical information obtained during visits to industrial and government facilities will continue.

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1. W. Steffe and T. Moutoux, "Avoiding Second Breakdowns in Power Transistors," *The Electronic Engineer* 26, 65-69 (December, 1967).
2. H. W. Rouhof, "Lateral Thermal Instability in Transistors Detected by Electrical Measurements," *Electronic Engineering* 40, 458-460 (1968).

18. THERMOGRAPHIC MEASUREMENTS

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

Progress: Thermographic phosphors have been used to determine hot-spot temperatures within an estimated ±2°C on a power transistor with a square chip approximately 60 mils on an edge. These measurements were made by photographing the illuminated phosphor and measuring the density of the film, point by point, by means of a microdensitometer. Most of the work was done in the temperature range 170 to 210°C using a zinc sulfide phosphor activated by copper and nickel. The principal drawback to this method is the time required for processing the photographs and evaluating the film density.

The test fixture illustrated in Fig. 5 was used for photographically recording the light output of the thermographic phosphor. The heat sink is used to heat the transistor under test uniformly for calibration measurements or to maintain the transistor case at a predetermined temperature while the transistor is dissipating power. A feedback circuit is used in conjunction with the ultraviolet light monitor to maintain the intensity of the near ultraviolet radiation at a uniform level. The chamber containing the transistor is vacuum tight although it has not been found necessary to evacuate it.

Two methods of coating the semiconductor chip were used. In the first, the decapped transistor is immersed in water whose surface is coated with a one-particle-thick layer of phosphor. When the transistor is raised from the water at an angle, the phosphor clings to the surface of the transistor. The semitransparency of the resulting coating makes it easy to locate the various parts of the transistor; a disadvantage of the thin coating is that the brightness of the fluorescence where the phosphor coats metallized regions is different from the brightness of the same phosphor at the same temperature where it coats oxide regions. The effects of these differences are minimized by making calibrating measurements with the entire transistor maintained at the same temperature.

Phosphor coats from 1 to 5 mils thick are obtained by the second coating method. In this method, the transistors to be coated are placed in an evaporating dish or other container and covered to a suitable depth

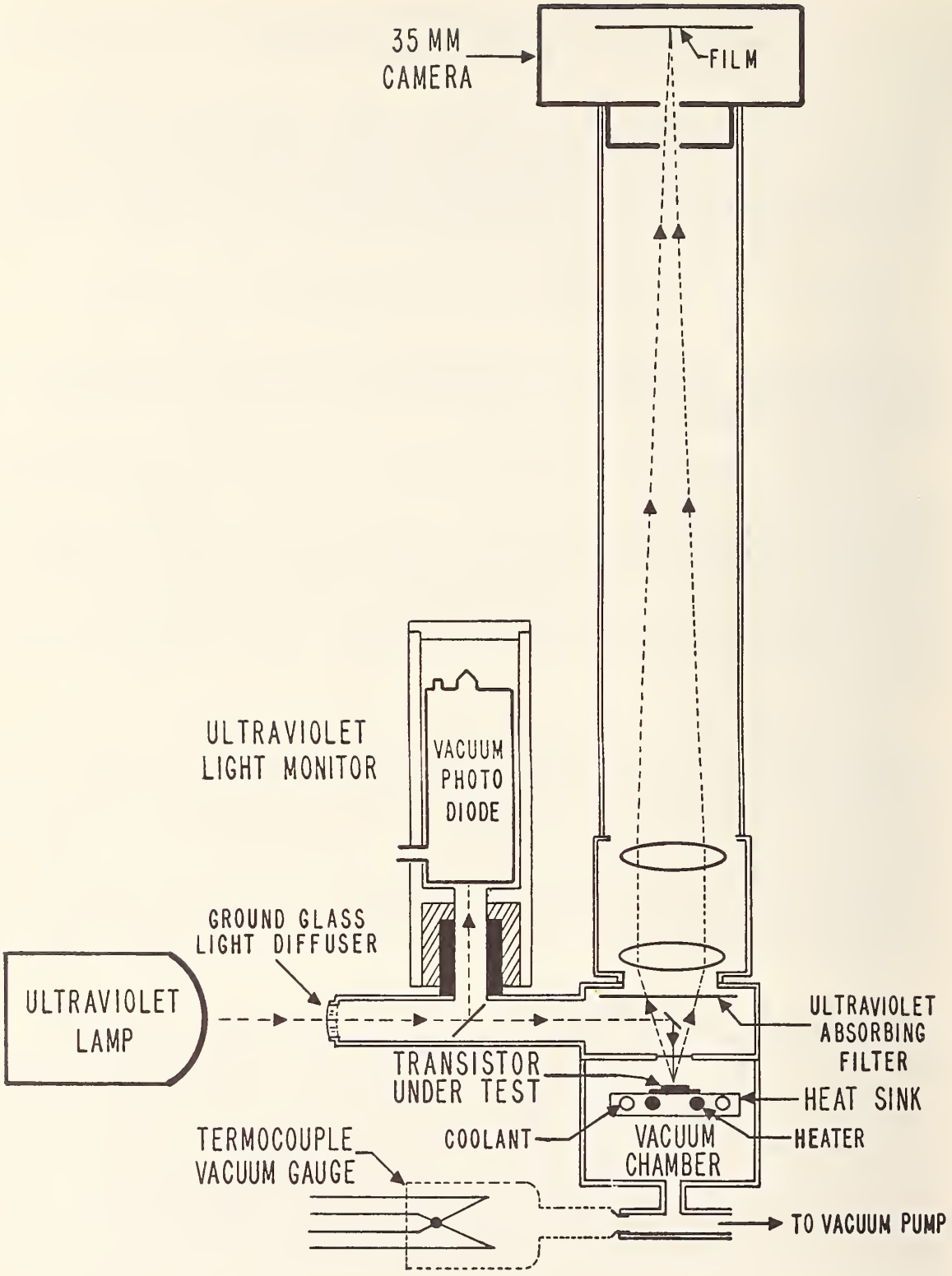

Fig. 5 Test fixture for photographically recording the visible light output of a thermographic phosphor which has been deposited on the surface of the chip of the transistor under test and is illuminated by ultraviolet light.

by a liquid in which the phosphor has been suspended. The thickness of the coating can be varied by the amount of phosphor suspended in the slurry and by the depth to which the semiconductor chip is covered. After 5 to 10 min, most of the solid particles will settle out of the liquid. The excess liquid is removed either by evaporation or by siphoning until only a meniscus is left on the semiconductor chip.
(D. B. Brenner,* G. J. Rogers, and F. F. Oettinger)

Plans: Methods of applying the temperature-sensitive phosphors will be refined. To reduce the time required for a measurement, a photometric microscope, which measures the light intensity directly and displays the results on an x-y recorder, will be installed. Experiments to determine the spatial resolution and temperature resolution as a function of the thickness of the phosphor coating will be begun.

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* NBS Measurement Engineering Division

19. MICROWAVE DIODE MEASUREMENTS

Objective: To study the problems and uncertainties associated with measurement of microwave mixer diode characteristics.

Progress: It was determined that the scope of this task should be expanded, and a new objective, given above, has been defined. Negotiations are continuing with the Navy and EIA to determine the problems involved in microwave semiconductor device measurements which should be studied.

Apparatus is being assembled to make measurements of diode noise using existing techniques.

The study of noise measurement theory shows that by using new analysis techniques, various types of noise can be differentiated. The degree to which power levels, impedances, and bandwidth must be stable and determined is not yet known. (R. C. Powell)

Plans: Assembly of the apparatus for an experimental investigation of problems involved in mixer measurement, the theoretical study of uncertainties, and consideration of other problems associated with measurement of mixer characteristics will be continued.

20. SILICON NUCLEAR RADIATION DETECTORS†

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

Progress: The assembly of the platform and shroud for life testing of detectors in a space-simulated environment is continuing. The baseplate with cooling channels, the thermal barrier, the coolant and electrical high-vacuum feedthroughs, and detector mounts have been constructed.

The design of a system for storing several hundred semiconductor detectors on bias and automatically monitoring their static electrical parameters on a daily basis has begun. This system will provide up-to-date information on the detector operational conditions and test data which can be used to predict the long-term stability of each detector. Initially, detector leakage current, noise, and temperature will be monitored at several preselected detector biases. Expansion of the system to include a determination of the counting performance of each detector is expected in the future.

Evaluation of the two 67-mm diameter silicon ingots for use in large-area detectors continued. Infrared absorption analysis showed that the oxygen concentration in these crystals was between 1.5×10^{18} and 1.8×10^{18} atoms/cm^3. Seven 19-mm diameter wafers were cut from a 2-mm thick slice of each crystal, and lithium was diffused into the surfaces. As expected from the high oxygen content of these crystals, the drifting of the lithium through the material at 500-V applied bias at a temperature of 125°C was extremely slow. It is estimated that the lithium mobility for these crystals is at least two orders of magnitude lower than the mobility for oxygen-free material ($\sim 5 \times 10^{-10}$ cm^2/V-s) under similar conditions. Thus, to drift completely through a 2-mm thick wafer would take more than 100 days as compared to approximately 22 hours for normal oxygen-free material under the same conditions.

Additional sources of detector-grade silicon crystals which have diameters greater than 50 mm are being sought. However, it has been found that most large-diameter silicon crystals are pulled from the melt in a quartz crucible. Some of the crucible material is usually dissolved in the molten silicon, thus providing the source of the oxygen contamination. (B. H. Audet)

Many of the surface-barrier detectors used in space radiation research are operated as totally-depleted, transmission detectors. Since the depletion depth of a detector is proportional to the square root of the product of the resistivity of the silicon and the reverse bias voltage applied, large variations of resistivity can cause very nonuniform depletion depths. The applied bias must be large enough to deplete completely the regions of lowest resistivity in order to eliminate dead layers in transmission detectors.

Float-zone refined, *n*-type silicon is normally used to make surface-barrier detectors. To determine the radial resistivity variation in typical high resistivity detector-grade silicon, two 0.3-mm thick wafers of *n*-type silicon with nominal resistivities of 10,000 Ω-cm were scanned with a four point probe. The results of the scan for one wafer is shown in Fig. 6. Similar results were obtained for the other wafer. Such

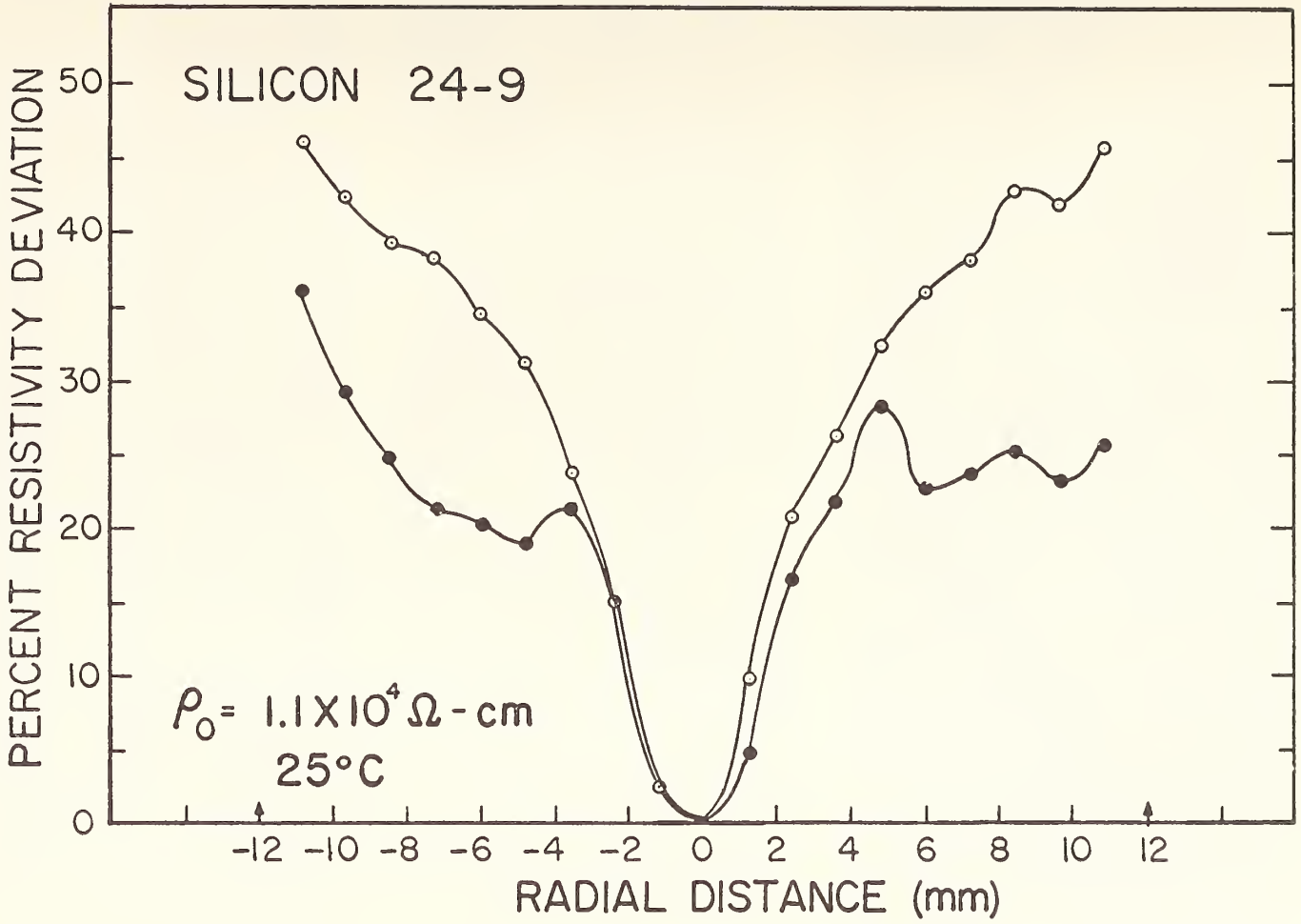

Fig. 6 Resistivity profile determined by means of the four-probe technique along two perpendicular diameters of a 1-mm thick wafer of *n*-type, float-zone refined silicon.

large variations of resistivity along a radius appear to be characteristic of high resistivity (>1000 Ω-cm), float-zone refined, *n*-type silicon.

A 1.0-mm thick silicon surface-barrier detector (Au-*n*-Si) was irradiated with 600 keV electrons on a 20-mm^2 spot at the center of the rear contact with fluences between 10^{13} and 10^{16} electrons/cm^2. The variation of the detector leakage current and capacitance with fluence and reverse bias is shown in Fig. 7. The counting response (collection efficiency and peak energy resolution) to alpha particles from ^{241}Am incident on the front contact degraded slightly with increasing fluence. However, at fluences above 3×10^{13} electrons/cm^2, the counting response to alpha particles incident on the rear contact was very much degraded. The effect of electron damage at the rear contact of a surface-barrier detector on the alpha particle counting response, as reported here, was opposite to that observed when the detector's front contact was damaged. These experiments have shown that the detector counting response was most seriously impaired when the alpha particles entered the contact which was irradiated. In these irradiations the range of the 600-keV electrons was approximately 85 percent of the thickness of the detector.
(J. A. Coleman)

Plans have been made to study the significance of initial carrier lifetime in silicon crystals on the characteristics of a radiation detector fabricated from the crystal. The plans call for initial measurements of carrier lifetime by the photoconductive decay (PCD) method. Four types of diodes (point-contact, surface-barrier, diffused, and alloyed) are to be fabricated from each crystal in the study and carrier lifetime measured by diode recovery methods. Detailed procedures for the preparation of diodes by these techniques are being selected. Lithium-drifted, surface-barrier, and diffused silicon particle detectors are to be fabricated from the same materials and characterized. In each case, correlations are to be sought between PCD lifetime value, diode recovery lifetime for each type of diode, and the characteristics of each detector.
(A. J. Baroody)

Plans: Assembly of the platform and shroud for high-vacuum life testing of detectors will be completed. Lithium mobility and precipitation in the large=diameter silicon ingots will be studied. An ingot of dislocation-free silicon will be evaluated for use in Si(Li) detectors. The radiation damage study in surface-barrier detectors will continue with irradiations by 1-MeV electrons. Work will continue on the study of the correlation of carrier lifetime in silicon with the characteristics of detectors made from the same material.

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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.

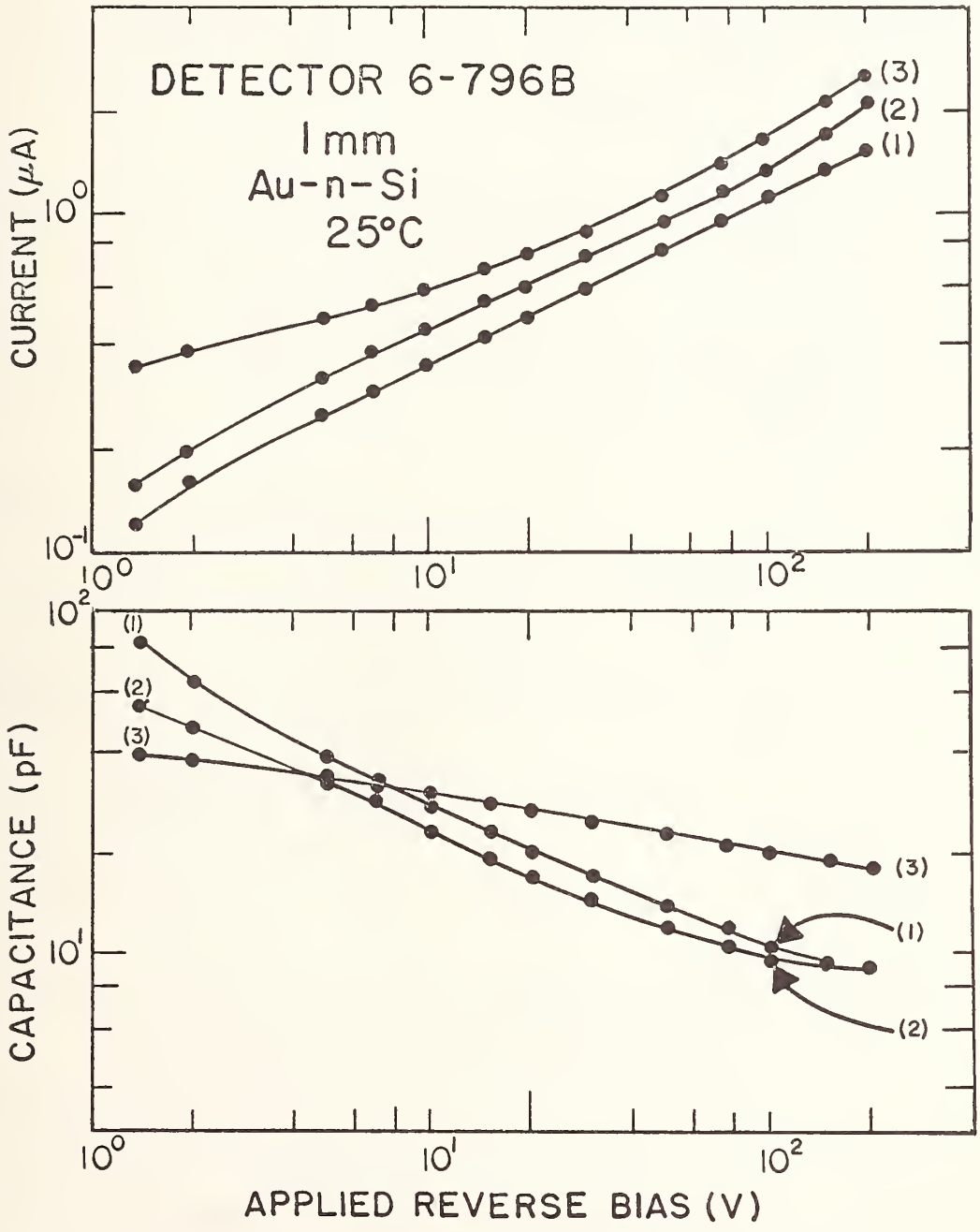

Fig. 7 Current- and capacitance-voltage characteristics of a 1-mm thick silicon surface-barrier detector before irradiation (1) and after damage by 10^{14} (2) and 10^{16} (3) 600-keV electrons/cm^2 incident on the rear contact.

Appendix A

JOINT PROGRAM STAFF

Coordinator: J. C. French

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M. Sigman
L. R. Williams

Secretaries:

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

---

* Part Time
† Guest Worker

Appendix B

COMMITTEE ACTIVITIES

ASTM Committee F-1; Materials for Electron Devices and Microelectronics
- F. H. Brewer, Task Force on Resistivity
- W. M. Bullis, Editor, Subcommittee IV, Semiconductor Crystals
- J. A. Coleman, Secretary, Subcommittee V, Semiconductor Processing Materials
- J. R. Ehrstein, Task Forces on Epitaxial Resistivity and Epitaxial Thickness
- J. C. French, Chairman, Subcommittee VIII, Editorial
- T. F. Leedy, Task Force on Photomasking
- J. Oroshnik, Task Forces on Thin Films, Thick Films, and Photomasking
- W. E. Phillips, Task Forces on Crystal Perfection, Encapsulation, Thin Films, and Thick Films
- A. H. Sher, Task Force on Germanium
- M. Sigman, Editor, Subcommittee V, Semiconductor Processing Materials
- W. R. Thurber, Task Forces on Impurities in Semiconductors and Germanium

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

Joint Electron Device Engineering Council (EIA-NEMA):
- JS-3, UHF and Microwave Diodes: R. C. Powell, Microwave Diode Specification Problems
- JS-6, Power Transistors: H. A. Schafft, Consultant on Second Breakdown Specifications
- JS-9, Low Power Transistors: F. F. Oettinger, Thermal Resistance Measurements
- 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; Editorial Board, *Transactions on Nuclear Science;* Chairman, 1970 Nuclear Science Symposium
- Magnetics Group: S. Rubin; Chairman, Galvanomagnetic Standards Subcommittee

IEC TC47, Semiconductor Devices and Integrated Circuits:
- F. F. Oettinger, U. S. Experts Advisory Committee
- S. Rubin, Technical Expert, Galvanomagnetic Devices

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

Appendix C

SOLID-STATE TECHNOLOGY & FABRICATION SERVICES

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

1. Silicon detectors - (B. H. Audet)
   Two 3-mm thick Al-*p*-Si surface-barrier silicon detectors were made for the Nuclear Spectroscopy Section. A wire bond which will remain attached to the thin Al film and have low noise at liquid helium temperature continues to be the major problem being encountered with these devices.

2. Quartz and glass fabrication - (E. I. Klein)
   a. A small-volume quartz cell which will be used for determining the dielectric constant for liquids was built for the Polymer Dielectrics Section.
   b. Ten He-Ne gas laser tubes with aligned end-windows were constructed for the Laboratory Astrophysics Division (Boulder).

3. Gunn devices - (G. G. Harman and H. K. Kessler)
   Twenty three hybrid mode devices were made for the Harry Diamond Laboratories. These were fabricated from epitaxial material and used a new all-metal electrode contact system. Several devices were life tested over 100 hours without any evidence of degradation.†

† NBS Project 4251424

Appendix D

JOINT PROGRAM PUBLICATIONS

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

G. G. Harman, "Topological Features of Hot-Carrier Induced Anisotropic Breakdown on Silicon Diode Surfaces," to appear in *J. Res. Natl. Bur. Standards*, Section A, May-June, 1969.

A. H. Sher, "Lithium Ion Drift Mobility in Germanium" to appear in *J. Appl. Phys.*, May, 1969.

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

Official SI Unit Names and Symbols

[For a complete statement of NBS practice, see NBS Tech. News Bull. Vol. 52, No. 6, June 1968.]

Name	Symbol	Name	Symbol
meter	m	newton	N
kilogram	kg	joule	J
second	s	watt	W
ampere	A	coulomb	C
kelvin[1]	K	volt	V
candela	cd	ohm	Ω
radian	rad	farad	F
steradian	sr	weber	Wb
hertz	Hz	henry	H
lumen	lm	tesla	T
lux	lx		

Additional Names and Symbols approved for NBS use

curie[2]	Ci	mho	mho
degree Celsius[3]	°C	mole	mol
gram	g	siemens[4]	S

[1] The same name and symbol are used for thermodynamic temperature interval. (Adopted by the 13th General Conference on Weights & Measures, 1967.)

[2] Accepted by the General Conference on Weights & Measures for use with the SI.

[3] For expressing "Celsius temperature"; may also be used for a temperature interval.

[4] Adopted by IEC and ISO.

Table for Converting U.S. Customary Units to Those of the International System (SI)[5]

To relate various units customarily used in the United States to those of the International System, the National Bureau of Standards uses the conversion factors listed in the "ASTM Metric Practice Guide", NBS Handbook 102. These are based on international agreements effective July 1, 1959, between the national standards laboratories of Australia, Canada, New Zealand, South Africa, the United Kingdom, and the United States.

To convert from:

(1) inches to meters, multiply by 0.0254 exactly.
(2) feet to meters, multiply by 0.3048 exactly.
(3) feet (U.S. survey) to meters, multiply by 1200/3937 exactly.
(4) yards to meters, multiply by 0.9144 exactly.
(5) miles (U.S. statute) to meters, multiply by 1609.344 exactly.
(6) miles (international nautical) to meters, multiply by 1852 exactly.
(7) grains (1/7000 lbm avoirdupois) to grams multiply by 0.064 798 91 exactly.
(8) troy or apothecary ounces mass to grams, multiply by 31.103 48 ...
(9) pounds-force (lbf avoirdupois) to newtons, multiply by 4.448 222 ...
(10) pounds-mass (lbm avoirdupois) to kilograms, multiply by 0.453 592 ...
(11) fluid ounces (U.S.) to cubic centimeters, multiply by 29.57 ...
(12) gallons (U.S. liquid) to cubic meters, multiply by 0.003 785 ...
(13) torr (mm Hg at 0 °C) to newtons per square meter, multiply by 133.322 exactly.
(14) millibars to newtons per square meter, multiply by 100 exactly.
(15) psi to newtons per square meter, multiply by 6894.757 ...
(16) poise to newton-seconds per square meter, multiply by 0.1 exactly.
(17) stokes to square meters per second, multiply by 0.0001 exactly.
(18) degrees Fahrenheit to kelvins, use the relation $t_K = (t_F + 459.67)/1.8$.
(19) degrees Fahrenheit to degrees Celsius, use the relation $t_C = (t_F - 32)/1.8$.
(20) curies to disintegrations per second, multiply by 3.7×10^{10} exactly.
(21) roentgens to coulombs per kilogram, multiply by $2.579\ 760 \times 10^{-4}$ exactly.

[5] Système International d'Unités (designated SI in all languages).

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