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

Quarterly Report

July 1 to September 30, 1969

U.S.
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Methods of Measurement for Semiconductor Materials, Process Control, and Devices

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July 1 to September 30, 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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METHODS OF MEASUREMENT FOR SEMICONDUCTOR MATERIALS, PROCESS CONTROL, AND DEVICES

Quarterly Report
July 1 to September 30, 1969

ABSTRACT

This quarterly progress report, fifth of a series, describes NBS activities directed toward the development of methods of measurement for semiconductor materials, process control, and devices. Principal emphasis is placed on measurement of resistivity, carrier lifetime, and electrical inhomogeneities in semiconductor crystals; evaluation of wire bonds; and measurement of thermal properties of semiconductor devices. Other tasks involve study of infrared measurement methods, deep-lying impurities in InSb, and gold in silicon; 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 properties of microwave devices. 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. Laboratory procedures for use and calibration of a capacitor microphone to measure vibration amplitude of the tool tip of an ultrasonic wire bonder are also described in a separate appendix.

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

1. INTRODUCTION AND HIGHLIGHTS

This is the fifth quarterly report to the sponsors of the Joint Program on Methods of Measurement for Semiconductor Materials, Process

Control, and Devices. The report is subdivided according to tasks which have been identified as parts of the Program. Sections 2 through 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.

Resistivity is the principle parameter used in specifying both bulk silicon wafers and epitaxial layers. Detailed study of the traditional four-probe method for application both to bulk silicon wafers and epitaxial layers continued. Results of a round robin conducted with Committee F-1 of the Americal Society for Testing and Materials (ASTM) to extend the range of applicability of the standard four-probe method to silicon wafers with higher and lower resistivity values were analyzed. For measurements on inhomogeneous wafers, the importance of making the measurement at the center of the wafer was demonstrated. In addition, serious errors were shown to be introduced into the measurement of radial resistivity variations when the specimen is slightly mispositioned.

Renewed interest in carrier lifetime measurements in ASTM Committee F-1 led to the reactivation of the carrier lifetime section at the September meeting. This interest was stimulated by the need to review for possible improvement the existing standard method for measuring carrier lifetime by the photoconductive decay method and by the increased desire to be able to determine carrier lifetime in epitaxial layers. There is also considerable interest among the radiation effects community for additional understanding of the carrier lifetime in transistors. To reflect the renewed interest work has been initiated on measurements of carrier lifetime in epitaxial layers in addition to continuing the efforts on measurements in bulk crystals, diodes, and transistors.

The emphasis in the inhomogeneities task has been reoriented toward establishment of the suitability of applying the photovoltaic method to the measurement of radial resistivity variations in germanium and silicon wafers. The apparatus has been improved to permit the measurement to be made on lapped wafers with room temperature resistivity 1 Ω-cm or higher. The feasibility of extending these measurements to specimens with room temperature resistivity down to 0.1 Ω-cm has been demonstrated. The quantitative evaluation of the relationship between photovoltage and resistivity gradient along the diameter of a wafer is continuing.

Substantial progress was made in the characterization of ultrasonic bonding systems by means of the capacitor microphone technique. Results of the studies demonstrate that the vibration amplitude of the tip of the bonding tool can be very different even when the bonder is set up in ostensibly the same fashion. Difficulties with absorption of ultrasonic power into the transducer mount were noted. In addition variations in both standing wave pattern and vibration amplitude of the tip were observed when the bonding tool was removed from the horn and reinserted either to the same or a slightly different extension. These studies further confirmed that the unloaded Q of the transducer, which is normally specified, has no significance insofar as the vibration characteristics of the bonding tool are concerned. Some dependence of both resonance frequency and tip vibration amplitude on the temperature was also observed.

This work to date has been a laboratory characterization of ultrasonic systems employed in bonding wire leads to silicon devices. However preliminary studies suggest that the capacitor microphone technique can be used to determine tip vibration amplitude during bonding. This approach is expected to provide a more direct means for obtaining both optimum and reproducible conditions for bonding. This phase of the program which is directed toward the development of procedures for obtaining reproducible bonds has been restricted to the aluminum-aluminum ultrasonic process. The bonds produced are intended for use as the vehicle for evaluating the effectiveness of various test methods in identifying weak bonds. It is intended that the test methods under consideration will also be applicable to other bonding systems such as the gold thermocompression system.

Studies of both the pull test and the high-current pulse test methods continued during the quarter. A scanning electron microscope is now available for use by the wire bond evaluation and other tasks in this program. Initially, work with this machine was devoted principally to operator training, but its usage is expected to increase considerably in the near future.

Preparation of the bibliography and critical review survey paper on wire bond evaluation has taken longer than anticipated. It is now expected that these will be completed before the end of this fiscal year.

Measurements of d-c current gain as a function of collector emitter voltage have shown a hysteresis effect which appears to be subject to interpretation on the basis of hot-spot formation. If the collector-emitter voltage of a transistor operated in a normal non-constricted current mode is increased to the point where a hot spot is formed and then reduced, the transistor will operate in a stable constricted current mode. It is thought that both the reversal of the current gain-temperature dependence at high current density and temperature levels as well as the dependence of current gain on current density are responsible for the hysteresis effect. These studies together with related studies in which the surface temperature distribution is being investigated by means of thermographic phosphors are continuing.

Work on the high field effects task has been suspended indefinitely in order that the staff originally assigned part time to it may devote all their efforts to the wire bond evaluation task. A preliminary survey to determine the problems associated with measurements of the properties of the silicon oxide films used in the semiconductor device industry was undertaken. Many different problems were encountered particularly in connection with thin silicon oxide and silicon nitride films used on MIS devices. The establishment of a task to study these measurement problems is being considered.

Further plans were made for the Symposium on Silicon Device Processing to be held in June, 1970, under the joint sponsorship of ASTM Committee F-1 and National Bureau of Standards. Acceptances have been obtained from all the speakers invited to participate in the opening session of the symposium which will consist of papers on silicon oxides and nitrides. A large number of papers is being considered for presentation at subsequent sessions which will deal with the unit processing operations, epitaxy and diffusion, from the standpoint of the required techniques and facilities as well as the properties and characterization of their product, surface preparation, and interdependence of the unit processing operations. Final selection of these papers will be made early in 1970.

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. The Program is supported by the National Bureau of Standards [1], the National Aeronautics and Space Administration [2], the Defense Atomic Support Agency [3], and the U. S. Navy Strategic Systems Project Office [4]. Because of the cooperative nature of the Program, there is not a one-to-one correspondence between the tasks described in this report and the projects by which the Program is supported. Although all sponsors subscribe to the need for the entire basic program for improvement of measurement methods for semiconductor materials, process control, and devices, the concern of certain sponsors with specific parts of the Program is taken into consideration in planning.

Additional background information on the Program and individual tasks may be found in earlier reports in this series (see Appendix D). A summary of early work which led to the establishment of the Joint Program has been prepared [5]. This summary contains a bibliography which includes all NBS publications which were issued in this field prior to the formation of the Joint Program.

Besides the tasks sponsored under the Joint Program, this report contains descriptions of activity on related projects supported by NBS or other agencies. Although the specific objectives of these projects are different from those of the Joint Program, much of the activity undertaken in these projects will be of interest to Joint Program sponsors. The sponsor of each of these related projects is identified in the description of the project.

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1. Through 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 808-70. (NBS Project 4259522)
4. Administered by U. S. Naval Ammunition Depot, Crane, Indiana through Cost Reimbursement Order P09-0016. (NBS Project 4259533)
5. W. M. Bullis, "Measurement Methods for the Semiconductor Device Industry — A Review of NBS Activity," NBS Tech. Note 511, December, 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: Detailed study of the four-probe method for application to bulk silicon wafers and epitaxial layers continued. Effort was increased on both the spreading resistance and capacitance-voltage methods which are intended primarily for use on epitaxial layers. In addition, capability was established for measuring impurity concentrations in bulk silicon by an infrared reflectance method which can be applied to measurement of the resistivity of epitaxial substrates. At present it is possible to measure impurity concentration in the range between 5×10^{18} cm^{-3} and 2×10^{20} cm^{-3}.

Four-Probe Method — Results of a round robin based on the ASTM four-probe method for measuring resistivity of silicon wafers [1] completed in cooperation with ASTM Committee F-1 have been tabulated and reported at the September meeting of the committee. The round robin was conducted to determine the precision of the method for wafers with resistivity down to 0.001 Ω-cm and up to 2000 Ω-cm. The analysis was based on data submitted by six of the eight participants; two participants failed to follow certain aspects of the test procedure. It was found that the precision specified in the present version of the method (3-sigma limit of ±2 percent) can be achieved for measurements on the low-resistivity wafers. Wider limits were found for wafers with resistivity above 120 Ω-cm. These results and the results of several previous round robins are summarized in Table 1.

There is some indication that wafer inhomogeneity was at least partially responsible for the increased scatter in the higher resistivity ranges. Because this had been suspected from earlier results an inhomogeneous 1 Ω-cm wafer was included in the present round robin. The scatter in resistivity reported for this wafer was considerably greater than would have been expected for a homogeneous wafer of this resistivity. The resistivity profile of this wafer as determined by four-probe measurements along two perpendicular diameters is shown in Fig. 1a. To demonstrate the importance of correctly centering the wafer when making a radial profile, a second set of data was taken with the center of the wafer displaced about 0.9 mm to the right along each of the perpendicular measurement diameters. If this were done inadvertantly, the off-diameter correction factors [2] would be applied as though the center of the wafer had been correctly placed with the result illustrated in Fig. 1b. If the displacement distance is known, of course, appropriate correction factors can be used. In this case as would be expected, the result, illustrated in Fig. 1c, is quite similar to that of Fig. 1a.

Table 1 - Results of Four-Probe Resistivity Round Robins[a]

Specimen Number	Specimen Type	Number of Laboratories Participating	Average Resistivity (Ω-cm)	s(%)[b]
B-2	*p*	4	10.023	0.20
B-4	*p*	4	10.133	0.36
K-4	*p*	4	14.494	0.13
605603-3	*n*	5	0.008367	0.69
601333-2	*n*	5	0.08514	0.51
71983-2	*n*	5	101.08	0.63
71166-2a	*n*	3	838.0	0.48
71166-2b	*n*	3	932.	9.24
600200-2	*p*	5	0.0077426	0.41
607075-2	*p*	5	0.10881	0.34
70877-3	*p*	5	7.909	0.65
49445-2	*p*	5	11.877	0.63
66969-1	*p*	5	112.59	0.91
16603-2	*p*	5	944.8	1.02
1165-2	*n*	6	.000992	0.29
612552-1	*p*	6	.000979	0.56
2062	*n*	6	1.057	1.18
K.0.1	*n*	6	342.3	0.89
48290-2	*p*	6	1223.6 [1195.1][c]	5.86 [1.54][c]
40/1-1	*n*	6	1915.1 [1884.9][d]	4.04 [1.37][d]

[a] All results recomputed from raw data

[b] Relative sample standard deviation

[c] Lab 8 omitted from computation

[d] Lab 2 omitted from computation

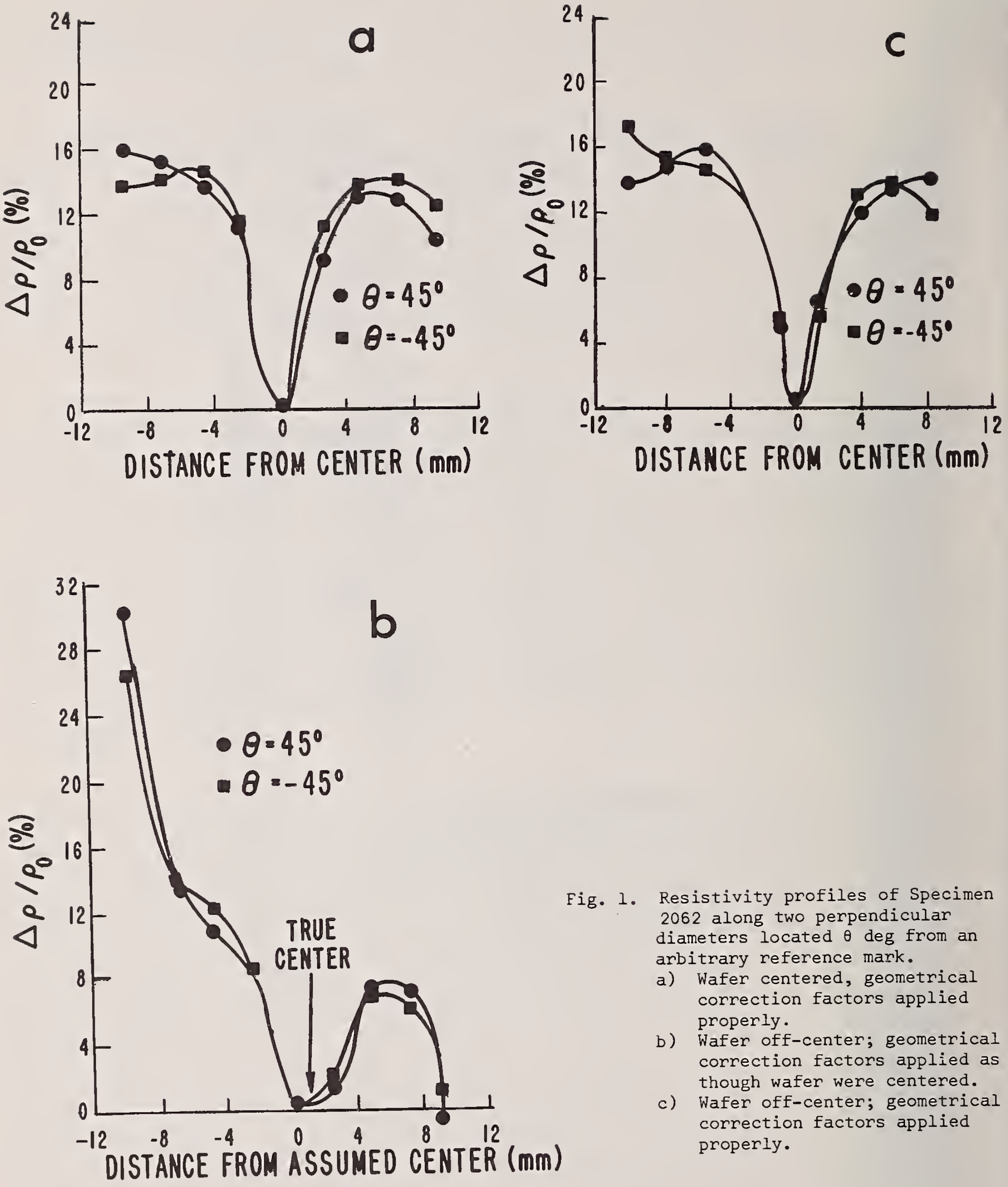

Fig. 1. Resistivity profiles of Specimen 2062 along two perpendicular diameters located θ deg from an arbitrary reference mark.

a) Wafer centered, geometrical correction factors applied properly.

b) Wafer off-center; geometrical correction factors applied as though wafer were centered.

c) Wafer off-center; geometrical correction factors applied properly.

The effect of improper centering on the measurement of the resistivity at the center of inhomogeneous wafers was also investigated. In each of five cases, resistivity measurements were taken at ten equiangular intervals as the pedestal on which the wafer was mounted was rotated through 360 deg. In case 1 both the probe and the wafer were carefully centered on the pedestal. In case 2 the probe was centered over the pedestal but the specimen was moved 1.5 mm (about one probe spacing) off center. As the pedestal was rotated, the measurement was always made about the same off-center point on the specimen but due to the inhomogeneity the effective resistivity as seen by the probe changed. In cases 3 and 4 the specimen was centered on the pedestal and the probe was displaced 1.5 mm from the center of rotation. In case 3 the displacement was in a direction perpendicular to the line of probes so that the probes were tangent to the circle of rotation, while in case 4 the displacement was in a direction along the line of probes so that the probes always lay along a radius. In case 5 both the probe and the specimen were displaced from the center of rotation by 1.5 mm in such a way that the probe position varied from the center of the specimen to a point 3 mm from the center. No corrections for off-center displacement were made. The results, summarized in Fig. 2, confirm that failure to place both the wafer and the probe at the center of rotation can degrade measurement precision if the wafer is not homogeneous. Interlaboratory precision would therefore be degraded if different operators measure the specimen at slightly different locations. It should be noted that, in this study, no allowance is made for the intrinsic errors introduced into the four-probe readings by the inhomogeneity of the specimen. This problem has been discussed by Swartzendruber [3] and, more recently, by Vieweg-Gutberlet and Schönhofer [4].

ASTM Committee F-1 is considering a round robin to extend the four-probe resistivity method to the 10,000 Ω-cm resistivity range. Since higher resistivity material tends to have poor homogeneity which leads to degradation of measurement precision, resistivity profiles were measured on several 6,000 to 10,000 Ω-cm specimens to determine the best specimens for use in the round robin.

The study of the current and probe-force dependence of the resistivity as measured by the four-probe method, preparatory to the use of this method for measuring the resistivity of epitaxial layers deposited on opposite conductivity type substrates, is continuing. Both resistivity and contact resistance are to be measured on wafers with lapped, mechanically polished, and chemically polished surfaces. Some measurements made on lapped surfaces ahve shown an erratic behavior, hence they are being repeated before proceeding to polished surfaces.

(F. H. Brewer, W. M. Bullis, and D. R. Ricks)

Spreading Resistance Methods — This technique is widely used for measuring resistivity of epitaxial layers. Several different methods for

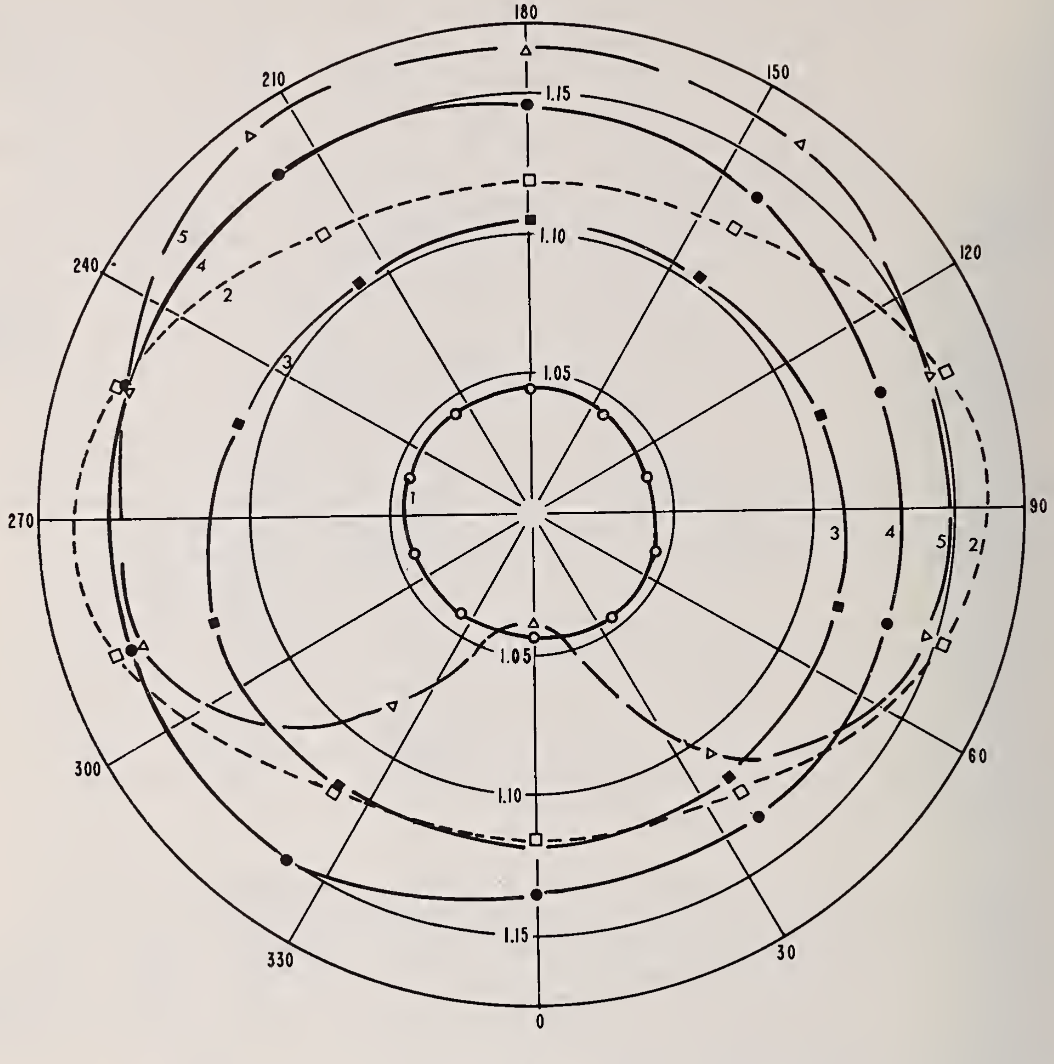

Fig. 2. Resistivity (Ω-cm) vs. angle (deg) for Specimen 2062 showing effects of misorientation of wafer and probes when measuring specimens with large radial resistivity gradients.

Case	Symbol	a	b	Probes	Specimens
1	○	1.0445	0.13	Centered	Centered
2	□	1.135	1.53	Centered	Displaced
3	■	1.111	0.63	Displaced[c]	Centered
4	●	1.142	0.74	Displaced[d]	Centered
5	△	1.132	3.73	Displaced	Displaced

a) Resistivity corrected to 23°C (Ω-cm)
b) Relative sample standard deviation (%)
c) Probes perpendicular to radius
d) Probes along radius

measuring spreading resistance have been reported [5, 6]. Although each of these is reported to yield highly reproducible results, the factors which must be controlled to enable adequate precision on intercomparison of the various methods are not fully recognized. The present effort is directed toward identifying these factors and establishing the technical basis necessary to the development of a standard procedure for this measurement. This effort is being carried out in cooperation with ASTM Committee F-1 which is now developing such a standard procedure.

A basic spreading resistance apparatus has been assembled. The purpose of this assembly is to evaluate and solve anticipated measurement problems relating to high electrical isolation of the measurement system, reduction of electrical noise below 1-μV, and attainment of reproducible contacts between probes and specimen with minimum damage to each. At present noise has been reduced to the 10-μV level by checking the circuit stage by stage for thermally sensitive contacts, proper grounding, and shielding. Isolation between stages has been maintained higher than 10^9 Ω. The question of reproducible contacts has not yet been investigated. (J. R. Ehrstein)

Capacitance-Voltage Method — This is an alternate technique for measuring resistivity of epitaxial layers. Initial measurements of capacitance have been made as a function of voltage with a 1-MHz capacitance bridge on shallow diffused mesa diodes fabricated on 8 Ω-cm *p*-type silicon. These measurements are intended to evaluate the available measurement equipment and to test the adequacy of a program used to compute doping density from the data [7]. (G. N. Stenbakken)

Plans: The investigation of the current and probe-force dependence of resistivity as measured by the four-probe method will continue. After consistent results have been achieved on wafers with lapped surfaces, measurements will be made for the same range of variables on mechanically and chemically polished surfaces.

Further four-probe resistivity measurements will be made on the 6,000 to 10,000 Ω-cm wafers in order to evaluate the single laboratory precision which can be expected on such wafers.

At the request of ASTM Committee F-1, a round robin will be coordinated on the measurement by the four-probe method of the resistivity of silicon epitaxial layers deposited on opposite type substrates.

Necessary steps will be taken to reduce further the electrical noise in the spreading resistance apparatus. After verifying that the probe impressions made with the apparatus are of a quality appropriate to the procedure being developed by ASTM Committee F-1, the effect of probe pressure and current levels will be investigated on several well-profiled, mechanically-polished, bulk wafers. The initial point material will be an osmium alloy and a three-probe measurement configuration will be used. Later these results will be compared against those obtained from a two-probe configuration.

Acquisition of capacitance data for the study of the capacitance-voltage technique will be expedited by the installation of a direct-reading capacitance meter which will be interfaced with a scanner-digital voltmeter system which is already operational. With such a system the entire range of bias voltages required to allow profiling the material under the fabricated diodes will be generated sequentially and recorded automatically along with the corresponding capacitance values. Planar and mesa diodes will be fabricated on bulk silicon wafers for which the radial resistivity uniformity has been determined by the four-probe method. Capacitance-voltage measurements on these diodes will be made to identify and investigate the importance of factors which affect the precision of the method.

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1. "Method of Test for Resistivity of Silicon Slices Using Four Pointed Probes" (ASTM Designation: F84-68T), *1969 Book of ASTM Standards*, Part 8, November, 1969. This document covers the range 0.05 to 120 Ω-cm.
2. L. J. Swartzendruber, "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples," NBS Tech. Note 199, April, 1964.
3. L. J. Swartzendruber, "Four-Point Probe Measurement of Non-Uniformities in Semiconductor Sheet Resistivity," *Solid State Electronics* 7, 413-422 (1964).
4. F. Vieweg-Gutberlet and F. X. Schönhofer, Grenzen der Anwendbarkeit des 4-Spitzen-Gleichstrom-Messverfahrens an Silicium-Proben, Teil I u. II," *Archiv für techn. Messen.* No. 369, 237-240 (October, 1966), No. 370, 259-262 (November, 1966).
5. R. G. Mazur and G. H. Dickey, "A Spreading Resistance Technique for Resistivity Measurements on Silicon," *J. Electrochem. Soc.* 113, 255-259 (1966).
6. J. M. Adley, M. R. Poponiak, C. P. Schneider, P. A. Schumann, Jr., and A. H. Tong, "The Design of a Probe for the Measurement of the Spreading Resistance of Semiconductors," *Semiconductor Silicon,* R. Haberecht and E. Kern, Eds., The Electrochemical Society, New York, 1969, pp. 721-731.
7. J. Hilibrand and R. D. Gold, "Determination of the Impurity Distribution in Junction Diodes from Capacitance-Voltage Measurements," *RCA Review* 21, 245-252 (1960).

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: Renewed interest in carrier lifetime measurements in ASTM Committee F-1 led to the reactivation of the Carrier Lifetime Section with Dr. W. E. Phillips as chairman at the September meeting of the Committee. This interest was stimulated by two factors: (1) the need to review for possible improvement the existing standard method [1] for measuring carrier lifetime by the photoconductive decay method, and (2) increased desire to be able to determine carrier lifetime in epitaxial layers. In addition there is increased interest among the radiation effects community for additional understanding of the carrier lifetime in transistors. To reflect this renewed interest the task has been organized into four areas according to the kind of structure being studied.

Bulk Crystals — Work is continuing on the evaluation and improvement of the photoconductive decay (PCD) method. To study the effect of a high density light source, nine specimens were measured by the PCD method with a HeNe (20 mW at 1.15 μm) laser as the light source. Comparison of these measurements with those made with a tungsten filament light source showed that a longer lifetime value is measured with laser illumination. That the longer lifetime could be attributed to the higher excess carrier density which is present with the laser was demonstrated by making several measurements with the laser defocused. With the resulting reduction of excess carrier density, the lifetime values agreed with those made with the tungsten filament light to better than 10 percent.

As reported last quarter (NBS Tech. Note 495, pp. 9-10), the apparatus for one of the contactless techniques [2] is operational. Additional work both on the extension of the PCD measurement capability to shorter lifetimes and on the two contactless methods for measuring lifetime by PCD has been postponed at least until work is completed on the revision of the existing ASTM method for PCD measurements [1] which is now being prepared. An experiment has been designed to establish a multi-operator, single-laboratory precision for the revised PCD procedure. The specimens of various resistivity, type, and geometry listed in Table 2 have been selected for study. Each specimen is to be measured four times by each of five operators following the revised procedure.

Another important task is the intercomparison of bulk lifetime measurements made by the various methods on the same set of specimens. The initial group, tabulated in Table 3, has been measured by the PCD method and surface photovoltage (SPV) measurements have been begun. The optical system of the SPV equipment was modified to permit a measurement of light

Table 2 - Specimens for Determination of the Precision of the Revised PCD Procedure

Specimen	Type and Material	Resistivity (Ω-cm)	Nominal Lifetime (μs)	Width (cm)	Thickness (cm)	Length (cm)
SS17	*n* Si	1	400	0.5	0.5	2.5
16444/2	*p* Si	1500	3600	diameter:	1.99	5.0
HS-1	*n* Si	7	(trapping)	1.0	0.56	3.4
SS6	*p* Si	270	35	0.25	0.25	2.5
SS10	*n* Ge	8	350	1.0	1.0	2.5
NG-13#4	*p* Ge	18	600	0.5	0.5	2.5
SS11	*n* Ge	8	100	0.5	0.5	2.5
SS12	*n* Ge	8	100	0.25	0.25	2.5

Table 3 - Specimens for Intercomparison of Lifetime Values by Various Measurement Methods

Specimen	Type and Material	Resistivity (Ω-cm)	Nominal Bulk Lifetime (μs)	Width (cm)	Thickness (cm)	Length (cm)
609651B	*n* Si	0.1	50	0.25	0.25	2.5
29021	*p* Si	1.0	60	1.0	1.0	1.9
605971B	*p* Si	4	85	1.0	1.0	2.5
31150	*p* Si	9.2	75	1.0	1.0	1.9
HG-1	*n* Ge	45	75	1.0	0.28	3.3
44853A	*p* Si	185	60	0.5	0.5	2.5
612225	*n* Si	9.6	8	1.0	1.0	2.5
322J	*n* Ge	7.4	33	1.0	1.0	2.3
HS-2	*n* Si	11	220	1.0	0.5	3.7
678C	*p* Ge	10	> 350	1.0	1.0	2.5

intensity without moving the illumination area on the specimen. The reproducibility of data was improved significantly.
(R. L. Mattis and W. E. Phillips)

Epitaxial Layers — Two methods for measurement of carrier lifetime in epitaxial layers are being studied: the metal-oxide-semiconductor (MOS) capacitance method [3] and the SPV method. The instrument requirements and limitations of the MOS capacitance technique are being investigated through study of published literature on the method to establish the appropriate directions for further investigation. (R. L. Mattis)

SPV measurements have been made on 9 to 11-μm thick epitaxial layers of 6 and 0.6 Ω-cm p on p^+ and 5 and 0.6 Ω-cm n on n^+ silicon. Although the data yield straight-line plots of photon intensity against penetration depth, or reciprocal absorption coefficient, as predicted [4] for a thick specimen, the theoretical consequences of the violation in thin epitaxial layers of the condition that the diffusion length be less than the thickness of the sample must be investigated before the intercept can be interpreted in terms of minority carrier diffusion length as it can in the case of a thick specimen. (W. E. Phillips)

Diodes — Emphasis during this period continued on studies intended to lead to a determination of the relevance of carrier lifetime of bulk silicon crystals to the characteristics of nuclear radiation detectors fabricated from the crystals. Present effort is concentrated in the area of the voltage decay technique for measuring carrier lifetime. Computer calculations were made of the dependence of the carrier lifetime as measured by this technique upon injection levels. In these calculations, various approximations used in computing the voltage decay following a forward current pulse were tested to determine their limits of validity. In particular the dependence of the shape of the voltage decay curve on the amplitude of the forward pulse and on the nature of the diode barrier was studied. Some conditions were found in which the linear voltage decay found for p-n step-junction diodes also occurs for surface barrier diodes. Several surface-barrier diodes have been fabricated and tested, but the conditions needed to obtain the linear voltage decay have not been achieved.

Fabrication of alloyed and shallow-diffused diodes for voltage decay and reverse recovery measurements of carrier lifetime in p-n step-junction diodes was begun. The mathematical analysis of the reverse recovery method was also continued. Additional data were taken in the experimental study of the reproducibility over an extended period of time of the reverse recovery technique for measuring carrier lifetime in p-n step-junction diodes. (A. J. Baroody)

Transistors — In order to make meaningful carrier lifetime measurements for device purposes, the interrelationships of the device structure and the measurement procedures must be more fully understood. A study of the literature on methods for measuring minority carrier lifetime in transistor structures was begun. (W. E. Phillips)

Plans: Work on the PCD method will continue with emphasis on the establishment of its multi-operator, single-laboratory precision. Further intercomparisons of PCD measurements with SPV measurements will be made.

The review of the MOS capacitance technique will be completed and areas will be selected for detailed investigation. The analysis of the SPV results will be studied to determine the relationship between the value of the intercept and the diffusion length for various ratios of diffusion length to epitaxial layer thickness.

To provide a firmer basis for understanding the various diode recovery techniques, emphasis will be shifted toward additional study of *p-n* step-junction diodes. Voltage decay measurements will be made on the junction diodes now being fabricated. Further understanding will be sought of the dependence of the voltage response on the amplitude of the forward pulse to the injection level. The experiment to study the reproducibility of the reverse recovery technique will be concluded and the results analyzed. The dependence of the measured reverse recovery time on the frequency of the input square wave will also be studied, and efforts to modify the reverse recovery circuitry to permit continuous variation of the ratio of forward to reverse current will continue.

The study of the literature on the measurement of carrier lifetime in transistor structures will also continue.

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1. Method for Measuring the Minority-Carrier Lifetime in Bulk Germanium and Silicon (ASTM Designation F28-66), *1969 Book of ASTM Standards*, Part 8, November, 1969.
2. J. Nishizawa, Y. Yamoguchi, N. Shoji, and Y. Tominaga, "Application of the 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.
3. C. Jund and R. Poirier, "Carrier Concentration and Minority Carrier Lifetime Measurement in Semiconductor Epitaxial Layers by the MOS Capacitance Method", *Solid-State Electronics* 9, 315-319 (1966); F. P. Heiman, "On the Determination of Minority Carrier Lifetime From the Transient Response of an MOS Capacitor", *IEEE Trans. Electron Devices* ED-14, 781-784 (1967); S. R. Hofstein, "Minority Carrier Lifetime Determination from Inversion Layer Transient Response", *IEEE Trans. Electron Devices* ED-14, 785-786 (1967).
4. F. Bergman, C. Fritzsche, and H. D. Riccius, "Bestimmung der Diffusionslänge in Halbleiten Mit Hilfe des Oberflächenphotoeffektes," *Telefunken Ztg.* 37, 186-193 (1964).

4. INHOMOGENEITIES

Objective: To develop improved methods for measuring inhomogeneities responsible for reducing performance and reliability of germanium and silicon devices and, in particular, to evaluate a photovoltaic method as a means of measuring radial resistivity variations in germanium and silicon circular wafers.

Progress: The present method [1] commonly used to detect radial resistivity variations in germanium and silicon circular wafers involves six four-probe measurements of resistivity: two at the center of the wafer and four, 3.2 mm from the edge, 90 deg apart. Several problems are encountered with this method. First, there are basic limitations in the application of the four-probe method to the measurement of resistivity variations, as has been discussed by Swartzendruber [2]. More recently, Vieweg-Gutberlet and Schönhofer [3] have examined this limitation in more detail and have recommended that to obtain more exact information about the radial resistivity profile along a diameter of a wafer, two-probe measurements should be made on a bar cut along that diameter of the wafer. They have shown in two cases that the discrepancy between profiles obtained by the two- and four-probe methods is as large as 10 to 20 percent. Second, the off-center correction factor [4] is very sensitive to the location of the probe near the wafer edge. As a result, small uncertainties in the probe placement produce large errors in the apparent resistivity (see Section 2). Finally, information about resistivity variations between the center and the measurement points near the edge cannot be obtained with this method.

Because of limitations in the four-probe method, the measurement of radial resistivity variation has caused considerable confusion between silicon suppliers and users. Preliminary evaluation of the photovoltaic method has indicated that it may in principle be used for this determination. Some interest in the use of this method as a replacement for the four-probe method has been indicated. Thus, the immediate objective of the task has been reoriented toward establishment of the suitability of applying the photovoltaic method to the measurement of radial resistivity variation on a practical scale.

Resistivity is a factor in determining the equipment required for the photovoltaic method. For a given light probe intensity, the voltages that must be measured are larger for higher resistivity specimens and so the measurement requirements are less severe. Several measurement techniques useful at different resistivity levels have been tested. The resistivity range investigated extends from about 100 Ω-cm to 0.1 Ω-cm.

For the higher resistivity specimens a 150-W incandescent lamp can serve as the light source for the light probe and a low-noise (≈0.5-μV peak-to-peak input noise) d-c amplifier can be used as the detector. The specimen surface need only be lapped with 5-μm alumina; no other surface preparation is needed. Specimens with a nominal resistivity as high as 100 Ω-cm and as low as about 40 Ω-cm have been measured in this way.

For lower resistivity specimens, to about 10 Ω-cm, use of this apparatus requires that the specimen surface be specially treated to reduce the surface recombination velocity and thereby increase the signals to a level measurable by the d-c amplifier. With the use of a more sensitive detecting scheme, measurements can be made on wafers with still smaller resistivities, to about 1.0 Ω-cm, without treatment of the specimen surface other than lapping with 5-μm alumina. In this case a chopped light probe and phase-sensitive lock-in detector are used. The longer integrating time required to detect the smaller signals lengthens the scanning time. As much as several minutes, depending on the signal level, may be required for a single scan compared with the 20 s which is typical when signals are large enough to use the d-c amplifier.

To make measurements on lapped specimens with resistivities less than about 1.0 Ω-cm requires a more intense light source for the light probe. A cw He-Ne laser with an output of 20 mW at a wavelength of 1.15 μm was used to make measurements on a silicon specimen with a nominal resistivity of 0.1 Ω-cm. The intensity of the light probe required results in thermal gradients in the specimen. However, by chopping the light and using a lock-in detector it is possible to extract the photovoltage signals from the thermally induced voltages.

The surface preparations to reduce the surface recombination velocity, s, in germanium and silicon have been inconvenient to use and inconsistent in their ability to reduce s. Further use of these preparations was abandoned and the use of the light chopper and the lock-in detector was extended to the higher resistivity range so that lapped specimens may be used throughout. At present, the incandescent lamp is being used as the primary source. Consideration is being given to incorporation of a laser source permanently into the system because of its advantages in the reduction of scanning time, in the extension of the resistivity range to at least 0.1 Ω-cm, and the assurance that the distribution of the photo-induced electron-hole pairs is uniform in the specimen bulk intercepted by the light probe. For the uniform distribution condition to be satisfied to a good approximation when the incandescent lamp is used, the specimen thickness must be less than the carrier diffusion length, a boundary condition which is not always convenient to satisfy.

Two problem areas were encountered while continuing to evaluate the relationship between photovoltage and the resistivity gradient (NBS Tech. Note 488, p. 9) by comparing the resistivity profile obtained using this equation with the resistivity profile obtained from four-probe measurements. They have to do with making contact to the specimen and the precision of the four-probe method and are discussed below.

This equation was derived with the assumption that the electrical contacts to the wafer are ohmic and semicircular in shape. To avoid the inconvenience of making semicircular notches in the wafer, small flats were made at the edge of the wafer and contacts made to them. It was felt that sufficiently small, flat contacts (1.0 to 1.5 mm) would provide a good approximation to the semicircular contact specified in the theory. Experiments have indicated this to be the case when measuring the photovoltage and photo-induced change in resistance of the specimen, but not when measuring the total specimen resistance. This resistance enters as the square in the equation for the resistivity gradient. Means for preparing wafers with semicircular contacts were developed for use in further evaluating the equation.

Discrepancies between the resistivity profile obtained by the photovoltaic method and that obtained by the four-probe method were observed especially near the edge of the wafer. Part of this difficulty may lie both with the basic limitation of the four-probe method to measure resistivity variations and the sensitivity of the off-center correction factor to location of the probe near the wafer edge as noted above. Both these limitations are absent if the two-probe method is used to make the profile. However, since this requires cutting a rectangular bar from the wafer along the measurement diameter, it cannot be undertaken until all photovoltaic studies of the wafer have been completed.

Plans: The evaluation of the relationship between photovoltage and radial resistivity gradient in germanium and silicon circular wafers will be completed. Profiles determined by both the four-probe and two-probe methods will be compared with the photovoltaic resistivity profile obtained on selected wafers. To help establish the priority of continuing work on this measurement method, the interest of additional suppliers of silicon wafers in the photovoltaic method for detecting radial resistivity gradients will be sought in telephone contacts.

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1. Methods of Test for Bulk Semiconductor Radial Resistivity Variation (ASTM Designation F81-67T), *1969 Book of ASTM Standards*, Part 8, November, 1969.
2. L. J. Swartzendruber, "Four-Point Probe Measurement of Non-Uniformities in Semiconductor Sheet Resistivity," *Solid-State Electronics* 7, 413-422 (1964).
3. F. Vieweg-Gutberlet and F. X. Schönhofer, "Grenzen der Anwendbarkeit des 4-Spitzen-Gleichstrom-Messverfahrens an Silicium-Proben, Teil I. u. II," *Archiv für techn. Messen*, No. 369, 237-240 (October, 1966); No. 370, 259-262 (November, 1966).
4. L. J. Swartzendruber, "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples," NBS Tech. Note 199, April, 1964.

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: Photoconductivity measurements as a function of wavelength have been made on copper-doped germanium specimens cooled to about 10 K. Specimens doped with 10^{15} copper atoms cm^{-3} were prepared from both *n*- and *p*-type starting material. Photoconductivity was observed at wavelengths less than 30 μm, the long wavelength limit associated with the copper impurity level at 0.04 eV above the valence band. The apparatus is now available for photoconductivity measurements on germanium specimens (see Section 9).

Work on the summary report describing the determination of oxygen in silicon and germanium by infrared absorption methods was postponed to next quarter. (W. R. Thurber)

An editorial and technical review was completed of four ASTM draft documents concerning determination of impurity concentrations by infrared absorption. Calculations were made to establish the conditions under which the present and revised methods for determining oxygen content of silicon yield a similar value for absorption coefficient. These results are particularly important in view of the fact that the calibration relating oxygen concentration to absorption coefficient is different in the new method. (W. M. Bullis)

Plans: The literature will be reviewed and an informal summary prepared on the relevance of photoconductivity measurements for determining deep impurities and their concentrations in silicon and germanium.

The summary report describing the determination of oxygen in silicon and germanium by infrared absorption methods is expected to be completed next quarter.

6. HALL EFFECT

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

Progress: The Hall effect apparatus was modified to permit automatic measurement with the digital voltmeter on high-resistivity materials. A new specimen holder was fabricated and the necessary wiring completed.

The report on the use of a time-shared computer to control the Hall experiment has been completed [1]. (W. R. Thurber and W. M. Bullis)

Plans: The new Hall apparatus will be tested and then used for measurements on high-purity germanium (see Section 9) and gold-doped silicon (see Section 8).

Work will continue on the report concerning Hall measurements and their interpretation.

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1. W. M. Bullis, W. R. Thurber, T. N. Pyke, Jr., F. H. Ulmer, and A. L. Koenig, "Use of a Time-Shared Computer System to Control a Hall Effect Experiment," NBS Tech. Note 510, October, 1969.

7. DEEP-LEVEL STUDIES

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

Progress: Although it has been concluded from comparisons of the results of resistivity, Hall coefficient, and carrier lifetime measurements on high-resistivity indium antimonide before and after the introduction of lithium that the residual deep-lying energy level is associated with a lattice vacancy, some ambiguity remained in the interpretation of the lifetime data (NBS Tech. Note 495, pp. 15, 16). Consequently, the photomagnetoelectric-photoconductivity apparatus was improved and additional lifetime measurements on both untreated and lithium-treated specimens were made. Because lithium tends to precipitate from indium antimonide crystals, Hall coefficient and resistivity measurements were repeated on a treated specimen to establish that the lithium concentration had not changed during the time between these and earlier measurements. Hall coefficient and resistivity measurements were also made on a previously unmeasured, untreated specimen to establish the characteristics of the wafer prior to introduction of lithium.

Analysis of the data obtained in these measurements was begun.
(J. L. Scales)

Plans: Analysis of the data will continue with emphasis on resolving the previously noted ambiguity in the interpretation of the lifetime data. On completion of the analysis, a final report will be prepared for publication.

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.

Progress: Specimens for initial electrical and activation analysis studies were prepared by diffusing gold into 10 Ω-cm boron-doped silicon wafers at temperatures of 850, 950, 1050, 1150, and 1250°C. The wafers were lapped with 5-μm abrasive to a depth of 25 μm on each side to remove the surface layer of gold which remains after diffusion. Diffusion conditions were selected so that each wafer was intended to be saturated with gold. Since the solid solubility is an exponential function of reciprocal temperature, gold concentrations were expected to vary from about 10^{15} to 10^{17} cm^{-3} [1]. The wafers were cut and a portion sent to the Analytical Chemistry Division for determination of the actual gold concentration by neutron activation analysis. Hall bars were cut ultrasonically from the remainder, and a 0.5-μm layer of aluminum was evaporated onto the contact areas of the Hall bars. Measurements of the resistivity and Hall coefficient at room temperature were begun to determine the effect of the gold on the free carrier concentration and the hole mobility.

A program to solve the charge balance equation for various models [1] which describe the energy level structure of gold in silicon was prepared for use on the NBS computer. It had been observed previously that the resistivity increases until the gold concentration of about 10^{16} cm^{-3} is reached and then decreases as still more gold is added. It was thought that the higher values for the degeneracy factor of the lower, or donor, level might explain this maximum in the room temperature resistivity against gold concentration curve for *p*-type silicon. A series of computations was made in which the degeneracy factor for this level was varied between 2 and 16. The computed curves did not show a maximum even for the highest values of degeneracy factor; it was concluded that some other mechanism must be responsible for the observation.

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

Plans: The initial electrical measurements will be completed and analyzed. The results of the neutron activation analysis will be used to verify that diffusion procedures such as those employed in the initial run provide the expected quantity of gold in the wafer. After establishment of satisfactory preparation procedures, the work will continue with studies of wafers cut from crystals of both *n*- and *p*-type silicon crystals with a room temperature resistivity of about 1 Ω-cm.

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1. W. M. Bullis, "Properties of Gold in Silicon," *Solid-State Electronics* 9, 143-168 (1966).

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 study [1] which shows that the effective Fano factor, F', in germanium at 77 K is less than the previously determined value of 0.13 [2-4] has been completed with the analysis of gamma-ray pulse-height data obtained with the collimated gamma-ray beam on Ge(Li) detector 213 in the manner previously described for Ge(Li) detector 83-2 (NBS Tech. Note 495, pp. 18-20). Trapping of charge carriers in detector 213, a 6-cm^3 detector fabricated for use as a reference detector in the continuing program of evaluation of materials and devices for gamma-ray spectroscopy, was observed to be less than in previous detectors. Excellent collection of charge over a compensated region of 8.5 mm appears to occur at about 1400 V/cm as compared to the previous test detector, 83-2, where a field of 2400 V/cm was required to obtain good charge collection over a compensated region of only 5 mm. Consistent with the observation of less electron trapping in detector 213 than in detector 83-2, F' is about equal to 0.10 over a larger region, approximately 4-mm wide, in the former device. This region was shifted towards the center of the compensated region which is also consistent with the observation of more nearly equal collection of electrons and holes in detector 213. The value of F' = 0.107 found in the present study is considered to be the upper limit of the intrinsic Fano factor, F. The measurement, however, appears to be limited by the lack of knowledge of the width of the secondary ionization sheath in the compensated region created by the collimated gamma-ray beam. The data also suggest that the use of lower noise electronic components, such as an FET preamplifier with the input FET operated below room temperature, would help in improving the quality of the measurement. (A. H. Sher and W. J. Keery)

The Germanium Section of ASTM Committee F-1 is examining methods of measuring the quality of lithium-drifted germanium gamma-ray detectors and the single crystal, *p*-type germanium from which these detectors are fabricated. Round-robins are currently being conducted on the measurements of lithium-ion drift mobility in germanium and trapping times in detectors. As part of the lithium-ion drift mobility round robin, measurements of lithium-ion drift mobility on specimens of germanium were completed according to the test procedure being considered by this Section. A computer program was written to analyze this data. NBS was requested to analyze with this program all subsequent data collected by the participants in the round robin. Measurements of trapping lifetimes in Ge(Li) detectors were also performed for specimens distributed in the second round robin. (A. H. Sher)

Additional nomographs are being prepared which relate to fabrication and testing procedures of Ge(Li) detectors. These nomographs permit

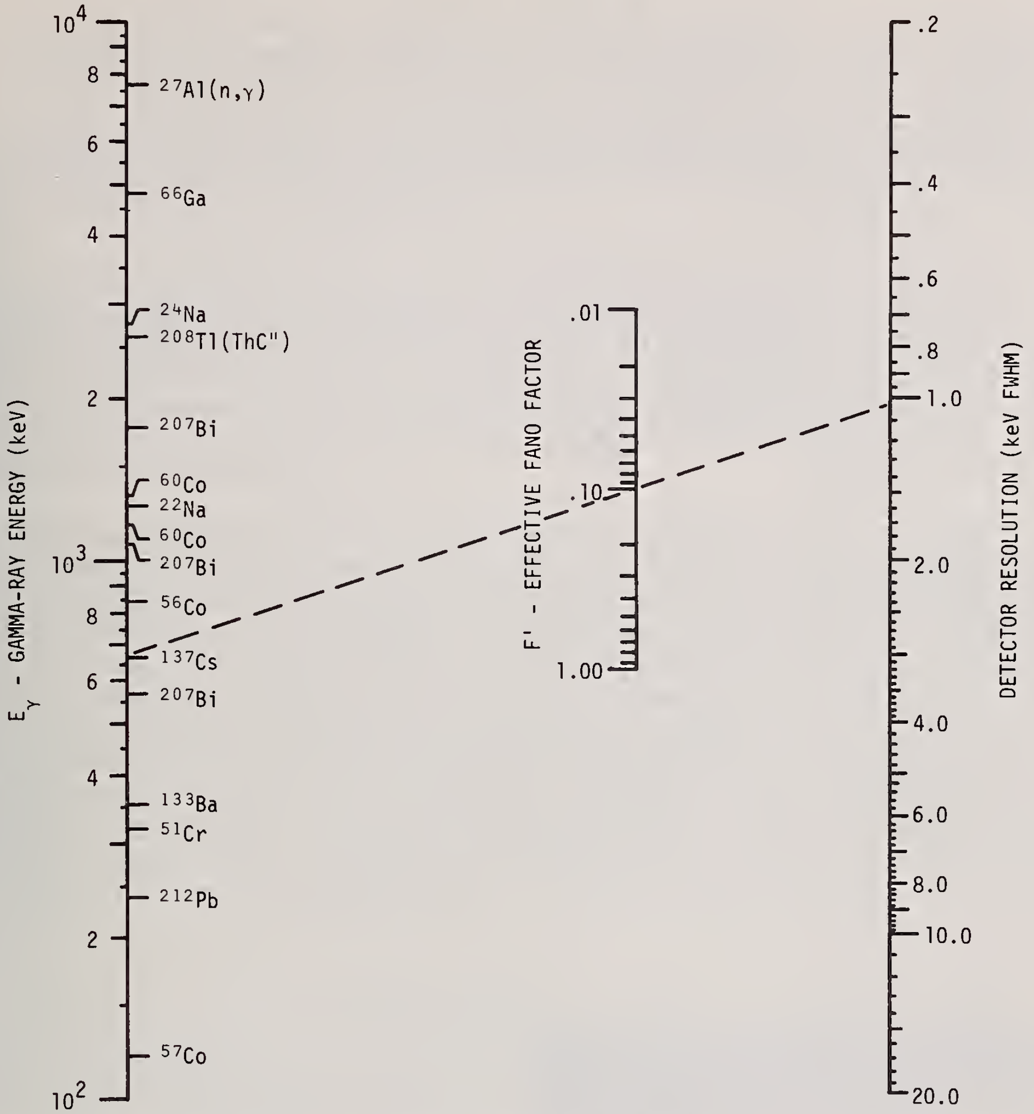

Fig. 3. Nomograph relating gamma-ray energy (E_γ), effective Fano factor (F'), and detector resolution. The dashed line shows an example in which a detector resolution of 1.0 keV at an E_γ of 662 keV yields an F' of approximately 0.1.

rapid evaluation of various parameters used during the fabrication. and testing of lithium-drifted germanium. Nomographs completed thus far include those relating drifted depth, time, temperature, and voltage during drift [NBS Tech. Note 488, p. 15]; junction area, capacitance, and drifted depth for planar detectors; detector resolution, total system resolution, and pulser resolution; and gamma-ray energy, effective Fano factor, and detector resolution. The last of these is shown in Fig. 3. A straight line between a value of the detector contribution to line-width and the gamma-ray energy, E_γ (keV), at which the measurement was performed will intersect the center scale at the value for the effective

Fano factor, F'. Also, by connecting values of E_γ with F = 1, the statistical width, Δ_E (keV), for the particular gamma-ray energy will be obtained on the right-hand scale. Nomographs in preparation are those relating oxygen concentration, lithium mobility, and acceptor concentration; and, capacitance, area, and drifted depth for coaxial Ge(Li) detectors. The publication of these nomographs in the form of a Technical Note is anticipated. (A. H. Sher)

Infrared response measurements (IRR) on Ge(Li) detector structures are being investigated as a possible procedure for screening *p*-type, single-crystal germanium to be used in fabricating Ge(Li) detectors as suggested by Armantrout [5]. A prototype of the low-noise preamplifier to be used in these measurements was assembled. The operating characteristics of this preamplifier unit are not yet suitable for use in the measurement system. (R. T. Dalton and W. J. Keery)

Plans: Lithium mobility studies, detector performance measurements (with emphasis on the use of a collimated gamma-ray beam to study carrier trapping phenomena), and the treatment of gamma-ray pulse-height data by computer techniques will be continued. Construction and testing of a cooled FET preamplifier for use in the spectroscopy system and the preamplifier for the IRR measurements will also proceed. An investigation of the relationship between the distribution of etch pits on crystals of *p*-type germanium and the characteristics of Ge(Li) detectors fabricated from the crystals will be begun. Impurity photoconductivity measurements (see Section 5) will be made on germanium crystals which show a high degree of charge-trapping. Measurements of IRR will also be made at 77 K on Ge(Li) structures fabricated from this material.

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

1. A. H. Sher and W. J. Keery, "Variation of the Effective Fano Factor in a Ge(Li) Detector," presented at Nuclear Science Symposium, San Francisco, October, 1969; to be published in *IEEE Trans. Nucl. Sci.*, February, 1970.
2. H. R. Bilger, "Fano Factor in Germanium at 77°K," *Phys. Rev.* 193, 283-253 (1967).
3. J. M. Palms, P. Venugopalo Rao, and R. E. Wood, "The Characteristics of an Ultra-High Resolution Ge(Li) Spectrometer for Singles and Coincidence X-Ray and Gamma-Ray Studies," *IEEE Trans. Nucl. Sci* NS-16, 34-46 (January, 1969).
4. A. H. Sher and B. D. Pate, "Determination of the Fano Factor in Germanium at 77°K," *Nucl. Instr. and Meth.* 71, 251-255 (1969).
5. G. A. Armantrout, "A Highly Sensitive Photoresponse Technique for Determining Impurity and Defect Energies and Concentrations in Germanium," presented at Third International Conference on Photoconductivity, Palo Alto, August, 1969; available from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Va. 22151, UCRL-71625.

10. METALLIZATION EVALUATION

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

Progress: A scratch test for characterizing the adhesion of metal films to non-metallic substrates based on the work of Karnowsky and Estill [1] is being evaluated. This study is being coordinated with a proposed procedure for the method which is presently under consideration by ASTM Committee F-1. The film-substrate system being used for the preliminary work is aluminum on quartz because of the importance of this system to microelectronics.

Experiments were begun on controlled scratching of aluminum films with a rudimentary apparatus consisting of a polished diamond stylus mounted on the arm of a dynamometer. Preliminary work was directed toward investigation of criteria for film failure, precision of a crude scratch test apparatus, and ways to determine the tip radius. Test films were prepared by evaporating about 0.5 μm of aluminum onto polished fused-quartz plates. The stylus was lowered onto the film to be scratched until the desired load was attained as determined by the dynamometer. The substrate was moved for a distance which gave a linear scratch path length of at least 1 cm. The nominal radius of the styli used for these familiarization studies varied from 18 to 127 μm. The total load on the stylus was varied on successive scratches in the range from 5 to 14 grams.

Both reflected light from the scratched aluminum film and light transmitted through the regions where the film was removed were used to observe the scratches. The critical load W_c at which the film fails is defined as that load for which light can be observed at 40X magnification to be transmitted through a scratch. As shown in Fig. 4, the aluminum film was removed by the stylus intermittently along the scratch path in all cases. As the stylus load was increased, removal of the metal was more complete. However, in no case was a length of film longer than about 8 mm removed.

Fig. 4. Typical scratch test plate showing failure points of a 0.56-μm thick vacuum evaporated aluminum film on a quartz substrate viewed by transmitted light. Region A has noticeably higher adhesion than the adjacent areas. The scratch test path is 1 cm long.

The intermittent failure is believed to result from variations in both film adhesion and substrate hardness.

The repeatability of the determination of the critical load W_c for film failure was also studied. A series of scratches was made with a 76-μm radius tip in which tip loads were varied by 0.1 g between adjacent scratches. This procedure was repeated several times in different regions on an aluminum-coated quartz plate. It was found that in areas of a few millimeters in width, the critical load for film failure was constant within ±0.1 g of its mean value of 1.9 g. This observation illustrates the sensitivity of the scratch test and indicates its usefulness as a quantitative method for comparing the relative adhesion of aluminum films on quartz substrates.

The difference in the critical load for two styli with different radii used to scratch the same aluminum film was determined. According to a derivation by Benjamin and Weaver [2], the shear force, F (g/cm^2), required to strip a metal film from a non-metallic substrate may be expressed as

$$F = P/[\frac{\pi r^2 P}{W_c} - 1]^{\frac{1}{2}}, \qquad (1)$$

where r is the radius of the stylus tip (cm), W_c is the critical load (g), and P is the indentation hardness of the non-metallic substrate, (g/cm^2). If the film adhesion to a given substrate is assumed to be constant, the force, F, should be constant and the critical load, W_c, should vary as the square of the tip radius, r. From measurements with two styli, it was found as shown by the data in Table 4 that this relationship was not followed if r is taken as the nominal tip radius. This anomalous result has not yet been explained.

Table 4 - Critical Loads for Failure of Aluminum Film

Tip radius, r (μm)	Critical load, W_c (g)
18	5.0 to 5.5
76	2.0 to 2.5

Attempts were made to use photomicrography to determine the radius of curvature of the stylus profile in the region of the stylus tip which comes in contact with the aluminum film. Shadowgraph techniques previously used had not provided enough detailed information about the tip radius. Photomicrography was found to be tedious, time consuming, and without any advantage over the shadowgraph techniques. Neither of these methods provides information about the critical portion of the tip.

A normal pull test for determining the adhesion of aluminum films is also being considered. A hot-melt resin glue was used to attach a dozen steel "common" pins to a 0.5-μm thick film of aluminum on a glass slide. When the pins were pulled off, the fracture always occurred at the interface between the glue and the top of the pin head and not at the aluminum-substrate interface. This failure is thought to be associated with improper positioning of the pin during gluing which can be minimized with proper jigging.

The literature search on adhesion testing of metallic films is continuing. (W. K. Croll and J. Oroshnik)

<u>Plans</u>: Evaluation of the scratch test method will continue using a form of balanced lever system in order to achieve better versatility, sensitivity, and a uniform stylus force on the film while the scratch is being made. A draft method for determining the adhesion of metal films to hard translucent substrates by the scratch test now being considered by ASTM Committee F-1 will be evaluated. Determination of stylus tip radius and topography will be attempted using interferometric methods. A test jig will be designed for use in the normal pull test method to hold the pins during their attachment to the metal film with a hot-melt resin glue. The literature search will be continued but limited to adhesion testing of thin metallic films on non-metallic substrates.

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1. M. M. Karnowsky and W. B. Estill, "Scratch Test for Measuring Adherence of Thin Films to Oxide Substrates," *Rev. Sci. Instrum.* 35, 1324-1326 (1964).
2. P. Benjamin and C. Weaver, "Measurement of Adhesion of Thin Films," *Proc. Roy. Soc.* (London) 254 A, 163-176 (1960).

11. PROCESSING FACILITY

Objective: To establish a microelectronic fabrication laboratory capable of producing specialized monolithic silicon devices for use in research on measurement methods.

Progress: Photomasking, computer-aided device design, and fabrication of test structures have been emphasized. The present status of the remainder of the processing facility may be described by considering each of the operations necessary to produce a monolithic device. At present diffusions may be produced with characteristics suitable for transistor fabrication. Boron-diffused layers have sheet resistances of 100 Ω/square with a Gaussian distribution of boron and surface concentrations of approximately 3×10^{18} cm^{-3}. Phosphorus-diffused layers, suitable for an emitter, may be produced with error function phosphorus distributions and sheet resistances of 10 to 15 Ω/square. Both types of diffusions have depths of approximately 2 μm in 1 Ω-cm silicon.

The steam oxidation process yields 0.5-μm thick oxides which are adequate for masking both phosphorus and boron diffusions. To demonstrate that these oxides are free of pinholes an aluminum film is evaporated over the oxide and raised to a temperature sufficient to alloy the aluminum into silicon. Alloying occurs where pinholes are present; triangular pits can be observed where alloying has occurred. Vacuum-evaporated aluminum metallizations 0.5-μm thick are being routinely produced. This thickness is sufficient to permit good ultrasonic aluminum wire bonding and yet allow easy photoengraving. No problem has been experienced with excessive pinholes or poor adhesion of the metal films. A photographic system for producing emulsion masks was developed. Mask sets suitable for making 1-mil (25.4-μm) line widths can be produced by means of a pinhole camera. The system has an overall reduction of the artwork of 200 times. The basic chip size used is 50-mils (1.27-mm) square. The camera efficiently produces masks with sufficient resolution to satisfy present needs for simple devices for test method development. Difficulties have been experienced in the adhesion of a positive-working photoresist on the oxidized wafers during etching. Poor photoresist adhesion and the attendant undercutting of the oxide is a common problem in the semiconductor industry. It is usually solved by careful attention to surface conditions during and after oxidation. Better control of these conditions is being attempted.

A set of "design rules" has been developed for device structures which can be produced by the fabrication facility. These rules define the technical objectives of the shallow-diffusion process which has been established and provide a starting point for the design of monolithic devices. To further assist in the design of circuit structures, a computer program, SNAP-II [1], has become operational on the NBS computer. The program has the capability of analyzing an electronic circuit and predicting its performance and has already been useful in designing a filter network for the wire bond study. In addition, a proposed silicon transistor structure was investigated to predict its feasibility as a test device. This structure was designed within the constraints of the design rules mentioned above. A preliminary analysis based on a simple hybrid-pi model [2] indicates that the bipolar structure will have an upper frequency limit (f_T) of approximately 2.5 MHz.

A large-area diode structure, 40-mils (1.0-mm) square, was designed and the mask set produced. After mounting on TO-5 headers these diodes are intended for use as heat-generating devices in the study of voids introduced into the die bonds (see Section 16).

Metallized arrays of square bonding pads have been produced for wire bond evaluation studies (see Section 12). The bonding pads are typical of those found on actual devices using aluminum metallization. Their size is nominally 5-mils (127-μm) square over 0.5-μm thick silicon oxide. The aluminum film was approximately 0.5-μm thick. Various heat treating techniques have been employed to optimize their suitability for wire bonding. (T. F. Leedy and J. Krawczyk)

A new reflector for the substrate heater has been completed for use in the evaporator. This quartz reflector is rectangular in cross-section rather than cylindrical. The rectangular design is expected to provide a more uniform substrate temperature distribution than the cylindrical type. A modified version of the platinum-bonded silicon sandwich substrate temperature sensor (NBS Tech. Note 475, p. 21) is presently under construction. (W. K. Croll)

Plans: Diodes for die attachment evaluation studies will be produced. Small planar diodes (area about 10 mil^2) will be produced for both epitaxial resistivity and lifetime studies. Work will continue on the electronic circuit analysis program ECAP [3] for use in evaluating devices and circuits under transient conditions. A contact printer will be installed in the photomask area to allow production of emulsion photomasks with greater contrast and better edge definition.

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1. T. D. Price, C. M. Kimme, and J. R. Gliessman, "User's Manual for SNAP-II Computer Program," prepared for U. S. Naval Weapons Center, China Lake, Calif. 93555, by ARINC Research Corporation, Western Division, Santa Ana, California. Publication 474-01-1-909. (Available from Naval Weapons Center.)
2. P. E. Gray, D. DeWitt, A. R. Boothroyd, and J. F. Gibbons, *Physical Electronics and Circuit Models of Transistors*, John Wiley & Sons, Inc., New York, 1964, p. 168.
3. IBM Application Program, "1620 Electronic Circuit Analysis Program (ECAP) (1620-EE-02X) User's Manual," (Available from either IBM, Technical Publications Department, 112 East Post Road, White Plains, N. Y. 10601 or UNIVAC Division of Sperry Rand Corporation, P. O. Box 8100, Philadelphia, Pa. 19101.)

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: Experimental efforts during this period were directed primarily toward further characterization of ultrasonic bonding systems by means of the capacitor microphone described last quarter (NBS Tech. Note 495, pp. 25-30). Preliminary work on the high-current pulse method for non-destructive wire bond testing was begun and the study of aluminum wire ball formation continued. The investigation of the tensile strength of both round and ribbon aluminum wire after burn-in at various temperatures was resumed. A scanning electron microscope has become available to the project; initial efforts were devoted to operator training. Evaluation of factors affecting the pull test for wire bond stregnth has begun. Improvements are being made to the wire bond puller to increase the speed and convenience of operation. Work on the comprehensive bibliography and critical review is continuing.

Characterization of Ultrasonic Bonding Systems — Extensive studies of the motion of ultrasonic bonding tools have been made with a capacitor microphone. Both equipment and techniques have been refined; the improved 0.007-in. diameter resolution makes it possible to measure the relative vibrational amplitude along the entire length of the tool.

The apparatus shown in Fig. 5 was assembled in order to position the microphone precisely with respect to the transducer and tool. In addition to micrometer-controlled translational movement along three perpendicular axes, the microphone can be rotated ±135 deg. around the tool in order to observe sideways and other modes of vibration. Details of the design of the high resolution microphone extension tip and the various measurement techniques are discussed in Appendix E.

The effects of mount design on the vibration amplitude of the bonding tool tip were examined. As can be seen in Fig. 5, the transducer-tool assembly is suspended from a fixed mount. It is essential that this be done in such a way that absorption of ultrasonic energy into the mount is minimized. Transducer manufacturers have provided tables [1] of design dimensions which should be avoided for various materials because of the possibility of resonant absorption.

Different transducer mounts were made and tested with several commercial transducer-tool assemblies. In one poor mounting condition, the wave form shown in Fig. 6 was observed. This wave form apparently results when some of the transducer energy is absorbed into the mount at a subharmonic rate. Under another condition, hash was observed at the peak of an otherwise normal sine wave. It was also observed that an improperly mounted transducer can have different resonance frequencies as the

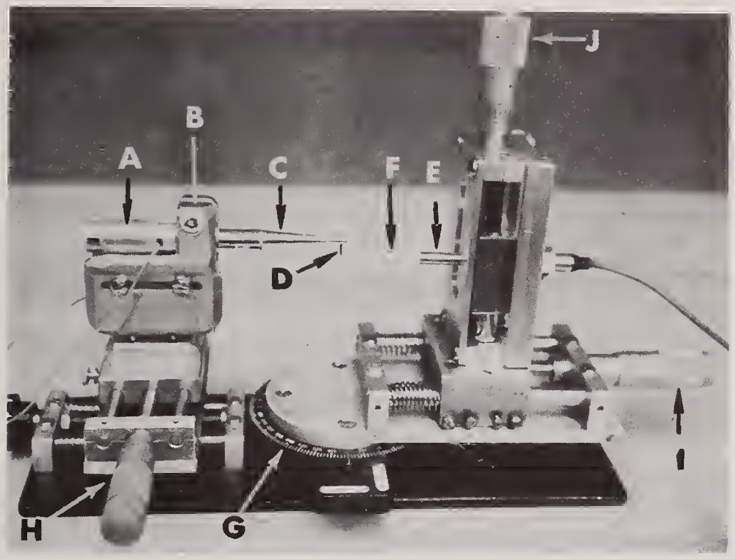


Fig. 5. Precision ultrasonic transducer-microphone mounting apparatus.

A: Ultrasonic transducer
B: Transducer mount
C: Tapered horn
D: Bonding tool
E: Capacitor microphone
F: Tapered tip
G: Graduated scale for rotation
H: Y-axis micrometer
I: X-axis micrometer
J: Z-axis micrometer

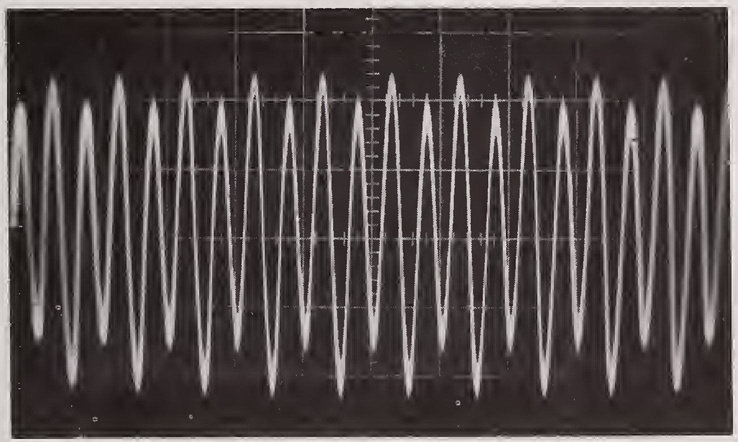


Fig. 6. Unusual wave form obtained with the ultrasonic microphone. This wave form results from a transducer mount resonance that absorbs power at half the excitation rate (30 kHz rate when the transducer is driven at 60 kHz).

applied power is changed. It was noted that energy can be absorbed by screws on the mount, near but not necessarily contacting the transducer. Since polytetrafluoroethylene (PTFE) is an excellent ultrasonic damping material, this absorption can be reduced by wrapping each screw with PTFE plumbing pipe tape before insertion.

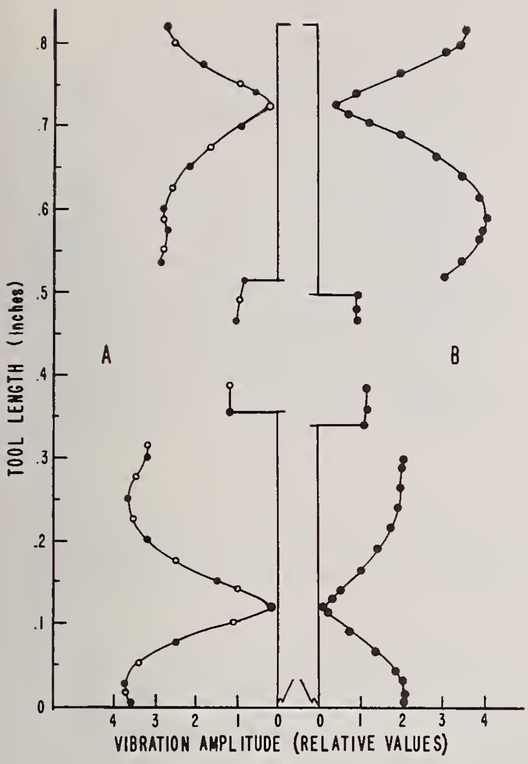
These problems do not depend solely on the mount characteristics, but rather on the mount-transducer combination. In a particular transducer mount that was carefully designed to avoid resonant dimensions, one commercial transducer lost energy by subharmonic absorption, but another transducer from a different commercial source functioned satisfactorily. It was also noted that most of the above characteristics are power sensitive. Below some threshold there is usually no evidence of absorption into the mount. As the power control setting is increased, more power may be absorbed into the mount instead of driving the tool. At still higher levels this power absorption may decrease. As a result, it cannot always be assumed that the vibration amplitude of the tool tip will always increase at the same rate as the power control setting.

It has also been verified that these effects can exist on commercial ultrasonic bonding machines. Direct observation of the vibration amplitude of the tool tip by means of an ultrasonic microphone has been shown to be an effective means of establishing whether or not power is being transmitted to the tool [2].

The amplitude of vibration along the length of the bonding tool was studied on many different tools inserted to various extensions. Several different transducers were used to drive the tools. In Figs. 7 to 9 are shown typical examples of the relative amplitude (peak-to-peak) of the tool vibration as a function of distance along the tool. The relative amplitude (peak-to-peak) of the vibration of the end of the transducer horn is also shown. The horn tip vibration amplitude, which is typically less than the tool motion, is not constant across the face of the horn tip. The reason for the variation is not understood.

The standing wave patterns in Fig. 7 demonstrate the changes which may occur when the same tool is removed from the horn and reinserted to a slightly shorter tip extension. The relative amplitude of the tip vibration changes substantially as does the relative amplitude of the standing wave pattern above and below the horn tip. The same power setting was used for both measurements, and the driving frequency required to maximize the tip vibration amplitude changed only slightly (about 50 Hz).

As shown in Fig. 8 similar changes occur when the tool is removed and reinserted to within 0.002 in. of the same extension. The results of a similar experiment carried out on a short tool driven by a different transducer and type of power supply are shown in Fig. 9. In this case



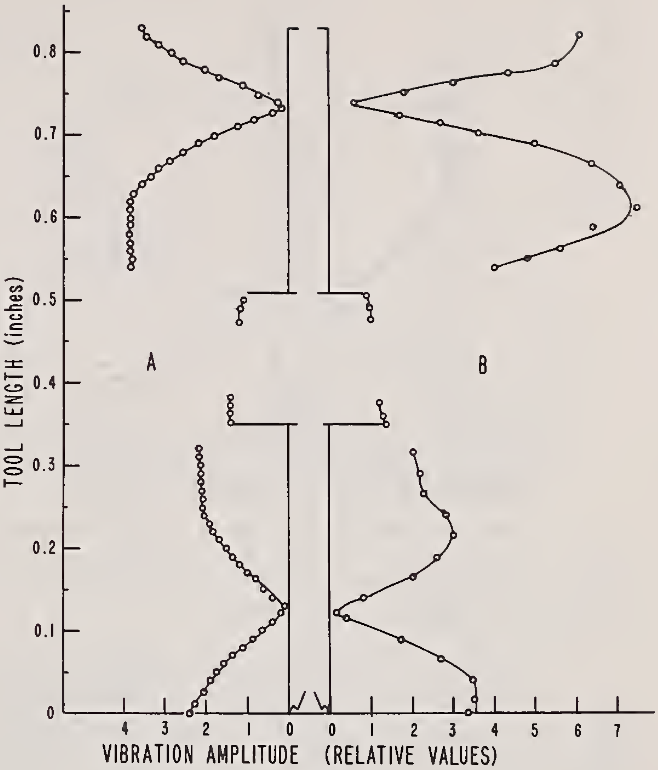


Fig. 7. Vibrational amplitude of a long ultrasonic bonding tool.

A: A bonding tool extended 0.350 in. below the horn.

B: The same bonding tool extended 0.335 in. below the horn.

The vibrational amplitude of the horn, which is in general less than that of the tool, is shown in the center part of the figure. Data for both extensions were taken with the same power setting on the ultrasonic supply, although the frequency was retuned slightly for maximum amplitude in each case. Amplitudes are relative values consistent within a given figure; they cannot be compared directly with other figures. The amplitudes and position of the horn are measured at its tip. Since the horn is tapered, the tool extension cannot be measured from the figure. Rounding of the amplitude curve at the tool extremities can be attributed to edge effects.

Fig. 8. Vibrational amplitude of a long ultrasonic bonding tool. The tool was removed after taking the data for A and then replaced to the same extension for taking the data for B. The vibrational amplitude of the horn, which is in general less than that of the tool, is shown in the center part of the figure. Data for both extensions were taken with the same power setting on the ultrasonic supply, although the frequency was retuned slightly for maximum amplitude in each case. Amplitudes are relative values consistent within a given figure; they cannot be compared directly with other figures. The amplitudes and position of the horn are measured at its tip. Since the horn is tapered, the tool extension cannot be measured from the figure. Rounding of the amplitude at the tool extremities can be attributed to edge effects.

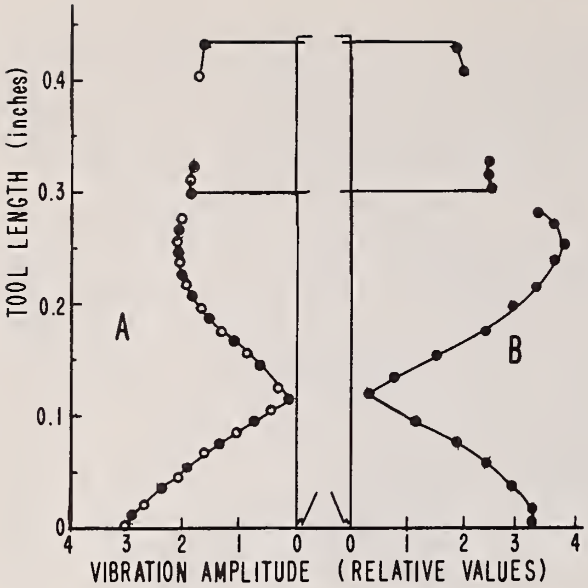


Fig. 9. Vibration amplitude of a short ultrasonic bonding tool. The tool was removed after taking the data A and then replaced to the same extension for taking the data B. The vibrational amplitude of the horn, which is in general less than that of the tool, is shown in the center part of the figure. Data for both extensions were taken with the same power setting on the ultrasonic supply, although the frequency was retuned slightly for maximum amplitude in each case. Amplitudes are relative values consistent within a given figure; they cannot be compared directly with other figures.

the tip amplitude remains essentially the same; however the standing wave pattern changes markedly.

A dramatic indication of the effect of tool extension can be seen from the frequency dependence of the tip vibration amplitude as shown in Fig. 10. For the 0.375-in. tool extension there is a double resonance peak with very large amplitudes in the resonance. After removal and reinsertion of the tool to an extension of 0.325 in., the curve with a single peak which has amplitude, resonant frequency, and shape typical of normal bonding is observed. The standing wave pattern observed with the bonder tuned to the 59 kHz resonance peak of Fig. 10 is shown in Fig. 11. In this case the relative vibration amplitude at the tip of the tool is the largest of any amplitude measured during this series of experiments, but the vibration amplitude of the upper portion of the tool is smaller than usually observed. The power control setting was that normally used for bonding 0.001-in. aluminum wire.

The results of these studies further confirm that the normally stated, unloaded electrical Q of a transducer has no significance insofar as the vibration characteristics of the bonding tool are concerned. Not only does the mechanical (acoustical) Q of a particular transducer-tool combination depend on the method of electrical drive (NBS Tech. Note 495, p. 28), but is also depends on the length and extension of the bonding tool. Mechanical Q can be changed as much as a factor of 2 by varying the tool extension within the range specified by the manufacturer. It was found to be relatively difficult to tune-up the system if the length of the tool is greatly different from that specified by the manufacturer of the transducer. In particular, a long (0.828-in.) tool employed in a

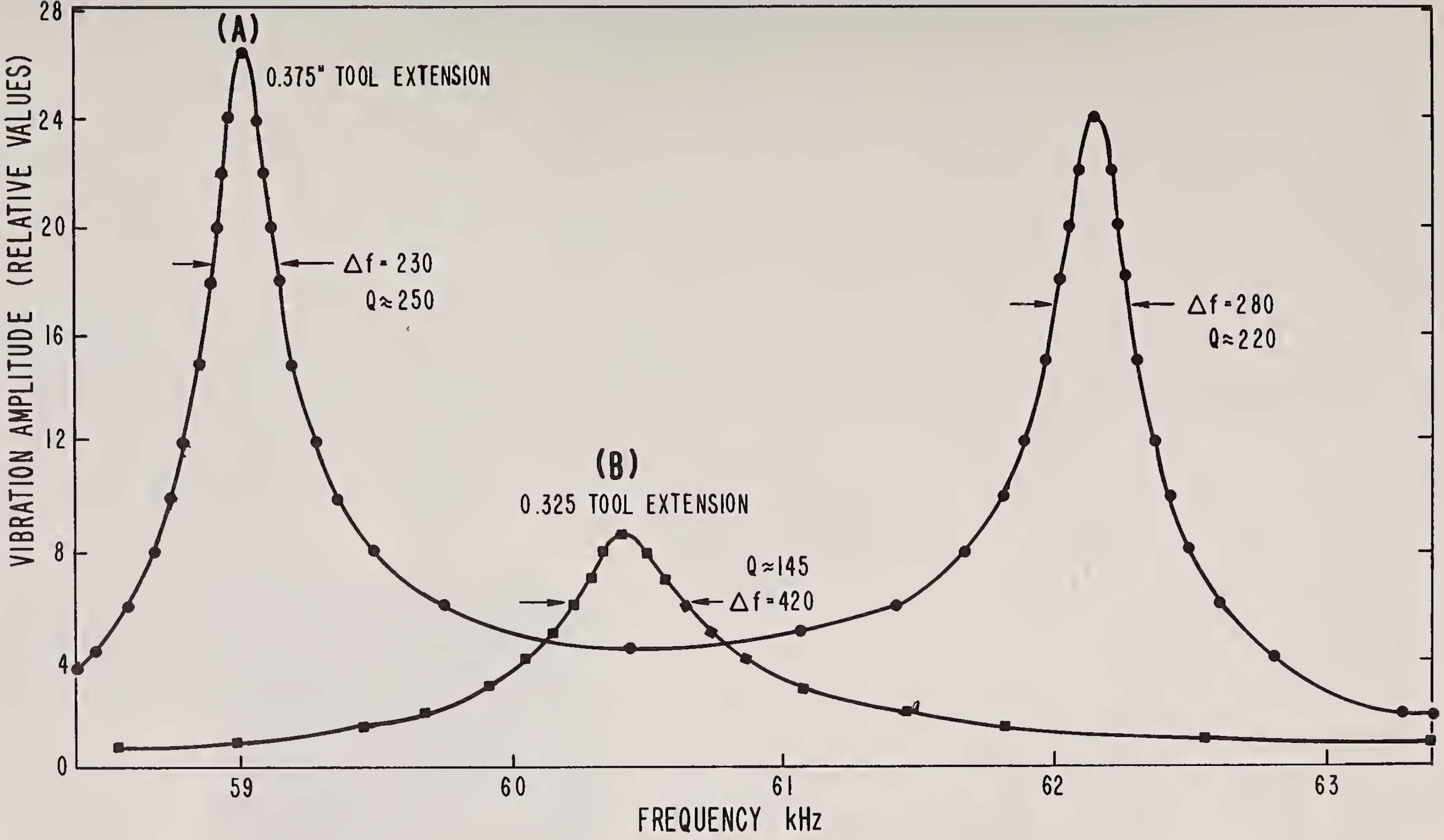


Fig. 10 Mechanical resonance curves for the same tool set at two different extensions below the horn. The control setting of the ultrasonic power supply was identical for both curves. The maximum amplitude in curve B is typical of conditions used for bonding 0.001-in. aluminum wire.

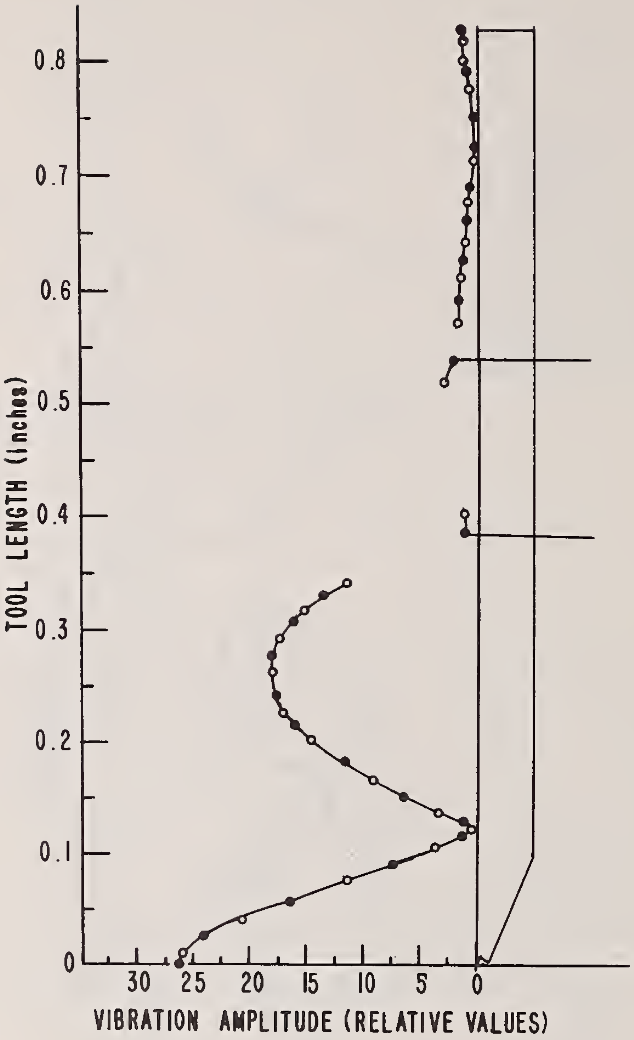


Fig. 11. Vibration amplitude of a long ultrasonic bonding tool which demonstrates unusually large resonance effects. These measurements were made with the ultrasonic system tuned to the 59 kHz resonance peak of Fig. 10, curve A. Amplitudes are relative values consistent within a given figure; they cannot be compared directly with other figures. The amplitudes and position of the horn are measured at its tip. Since the horn is tapered, the tool extension cannot be measured from the figure.

transducer designed for a short (0.437-in.) tool often results in a multiple resonance curve similar to that shown in Fig. 10.

It is to be expected that the vibration characteristics of the tool tip will be modified somewhat during bonding because of the loading on the tool; the mechanical Q of the loaded transducer-tool combination will be lowered. Preliminary measurements of the standing wave pattern of tools under a simulated bonding load of 24 grams show that the position of the lowest node shifts only a small amount (∿0.007 in.). In general the shift was found to be in a direction away from the end of the tool but for some conditions of tool length and extension the node moved toward the tool tip. The observed shift was sufficiently small that it alone does not appear to affect the maximum vibration amplitude at the tip. This shift may or may not be mirrored in the node near the top of a long tool. Under this load condition, the resonance curve broadens and the effective Q is reduced. Thus the ability to maintain the tool tip amplitude constant under load conditions depends to some degree on feedback in the transducer power supply. Further studies are necessary to establish more firmly the effects of loading on the tool tip motion.

Efforts are now under way to establish the origin of the variability in the vibration amplitude of the tool tip. A series of experiments was conducted to ensure that the variability is not due to faulty measurement equipment or techniques. Measurements on tools which have not been moved can be repeated to within about 5 percent. At the present time, it is felt that the principal difficulty rests with irregularities in the shape of the hole in the horn through which the bonding tool is inserted and with irregularities in the set screw which clamps the tool in place. A typical hole in a relatively new transducer horn is shown in Fig. 12. Irregular ridges with a height about 0.001 in. can be readily seen, and there is evidence of metal bent outward during the threading of the set screw hole. Because of these irregularities, the tool would not necessarily be driven from the same point on its length each time it was reinserted and clamped. This is equivalent to inserting the tool to a different depth each time even though the gage block measurement between horn and tool tip remains unchanged. Furthermore, the set screw wears with use and probably will not always clamp the tool in the same position. Some improvement could

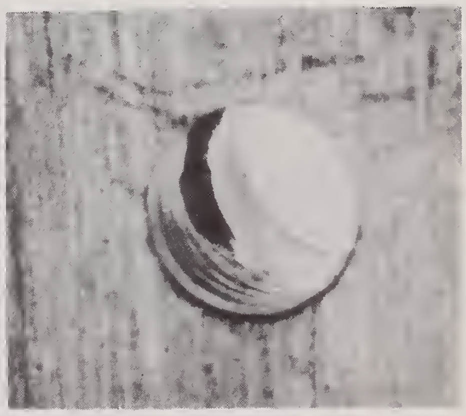


Fig. 12. Hole in an ultrasonic transducer horn through which the bonding tool is inserted. The dark area on the left is a part of the thread for the set screw. The photograph clearly shows irregularities on the wall of the hole. Magnification is approximately 18 times.

probably be obtained if the tool holes were drilled, reamed, and polished. Even under such conditions, a tolerance of ∿0.0002 in. is about the best that could be expected, and this, combined with a similar tolerance which is customarily specified on the radius of tungsten carbide bonding tools, can result in a misfit many times the vibration amplitude. Wear of the edge of the hole by the tool after prolonged use may also increase the problem.

Several possible solutions to these problems are being considered. A simple remedy for set screw wear is to replace it every time the bonding tool is changed. It was also found that more reproducible contact between such screw and tool can be achieved if the end of the set screw is filled with a soft material such as lead. Such a screw, or at least the lead tip, would still have to be replaced periodically. Various methods of rigidly holding the tools, such as epoxying or brazing, appear to be impractical. The misfit problem might be reduced by filling the empty regions with a glue-like material that transmits 60 kHz vibration and that can be put in and removed easily. It is also possible that a replaceable metal-foil gasket could be used for this purpose or that the geometry of the hole could be modified by enlarging it everywhere except directly behind the set screw so that all motion originates from a much smaller area.

Another problem associated with tolerance is the length of bonding tools which is specified to ±0.015 in. This means that two supposedly identical tools could differ in total length by 0.03 in. If the motion of the top portion of a long tool is coupled to that of the bottom of the tool then the tip motion can be seriously affected when a tool is replaced by another of different but supposedly identical length. All except one of the tools used in this study were well within the specification limits. This suggests that tool length tolerances could be tightened considerably.

It appears that once a tool is firmly clamped in position, it will maintain the same tip motion for given drive and temperature conditions until removed. This suggests that it would be feasible to establish the bonding schedule from measurements of tip vibration amplitude after each tool change or adjustment. Either an ultrasonic microphone or a simple laser interferometer could be used for this purpose.

The temperature dependence of the resonant frequency and tip vibration amplitude of tools driven by transducers with both ferrite and nickel magnetostrictive ultrasonic elements was measured to determine if a change in ambient temperature could detune a system. A linear temperature coefficient of 17 to 20 Hz/deg C over a range of 18 to 30°C was measured for both types of transducers. This value is consistent with the expansion characteristics of the stainless steel parts and is satisfactorily low for normal bonding machine use. However, some modern bonding machines have multiple high intensity lamps that illuminate an extended area around the work. Since these lamps are capable of heating the transducer by 5 or 10 deg C, it is possible that changes in the illumination could cause measurable changes in the operating characteristics of the transducer.

To date this work has been a laboratory characterization of the ultrasonic systems employed in bonding wire leads to silicon devices. These measurements and techniques are to be applied to studies of bonding machines during the bonding operation in order to establish the significance of individual findings with respect to the bonding process.

Laser Calibration of the Capacitor Microphone — Although much useful information can be obtained by means of the ultrasonic microphone system, its major limitation is that absolute amplitude measurements cannot be made without independent calibration of the system. The only known accurate method for this calibration is a laser interferometer system such as that described by Martin [2]. Figures 13 and 14 show typical oscilloscope traces obtained by laser interferometer measurement [3] of the vibration amplitude of the tip of a highly polished bonding tool. Each fringe represents 0.316-μm (12.5-μin.) displacement. The end or turn-around points are clearly broader than the intermediate motion fringes; this defines one cycle of motion. Because the measurement system was not refined, it was difficult to measure accurately the fraction of a fringe represented at the end points. Although the signal is relatively weak, the trace in Fig. 13 represents a normal type of interferometer curve. For the trace in Fig. 14 the signal-to-noise ratio was improved by adjusting the interferometer. The turn-around points in this trace appear to be unusual, but because of the preliminary nature of the measurement, no interpretation has been attempted.

The power setting required to produce the tool tip motion shown in Fig. 14 (about 60 μin. peak-to-peak) is on the low side of the range normally employed for bonding 0.001-in. aluminum wire. At slightly higher power levels still within the normal bonding range, the turn-around point showed high frequency modulation which could not be explained unless some harmonic motion was present which was not observable by the ultrasonic microphone which has a maximum response of 80 kHz. Additional measurements are required to complete the calibration of the ultrasonic microphone and to explain the nature of the traces.

(G. G. Harman)

High-Current Pulse Test Method — During the quarter, work began on a high-current pulse method of testing finished bonds and interconnections on integrated circuits. It had been noted on a previous project that during high-current pulsing of laser diodes, weak wire bonds would separate at the bond-device interface or give an irregular current wave shape pattern during the pulse. Based on this experience, this bond

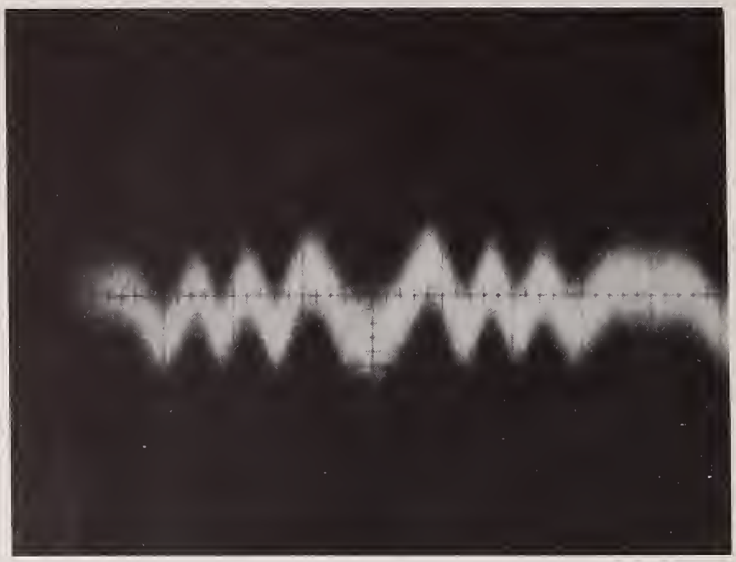


Fig. 13. Oscilloscope trace of a laser interferometer fringe measurement of bonding tool tip motion. The turn-around points are broader than the intermediate fringes. Each complete fringe represents a 12.5-μin. displacement of the tool.

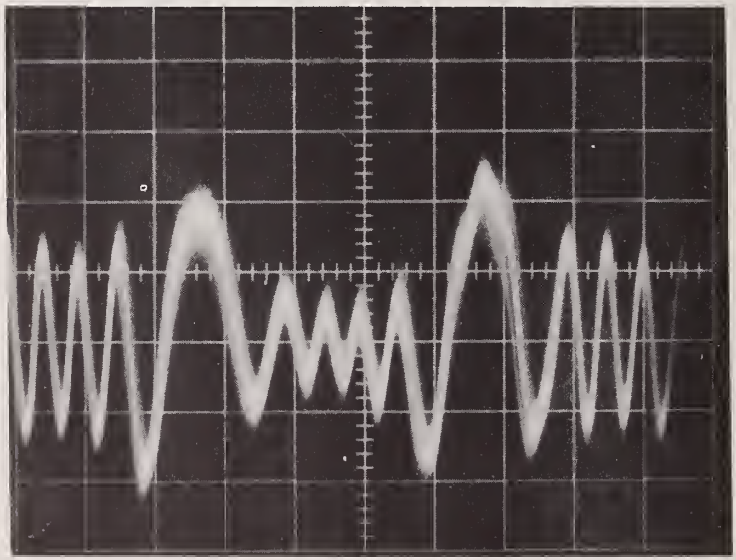


Fig. 14. Oscilloscope trace of a laser interferometer fringe measurement of bonding tool tip motion with improved signal-to-noise ratio. The turn-around points are more difficult to interpret than those of Fig. 13.

evaluation method is being adapted to test wire bonds on silicon devices and integrated circuits.

Various probes and micromanipulators were acquired and modified to be used in the test. It was established that the direct current capability of typical aluminum metallization stripes, 0.001 to 0.002 in. wide and 0.5 μm thick, on silicon exceeds that of 0.001-in. diameter aluminum wire which can carry about 1 A. Presumably this is because of the better heat sinking of the aluminum stripes. A four-terminal method has been used to determine contact resistance between probe and metallization under pulsed current conditions. Because of burnout of the metallization immediately under the probes, tungsten probes with relatively flat tips are being made. These new probes have been designed to have a very low inductance in order to avoid ringing in making the low impedance measurements.

Aluminum Wire Ball Formation — Some aluminum balls made by the spark-discharge method previously described (NBS Tech. Note 495, p. 30) were examined under a scanning electron microscope. They were found to be sufficiently uniform in size and sphericity to be acceptable for bonding purposes. An improved method for making aluminum balls involves the passing of a tiny jet of hot inert gas (∿1000°C) across the wire. An apparatus for making aluminum balls in this way is being assembled.
(H. K. Kessler)

Tensile Strength After Burn-In — To help in determining how much of the bonding strength decrease after burn-in is due to a decrease in the strength of the wire, a comparison was made between the breaking strength of aluminum (with 1 percent silicon) ultrasonic bonding wire before and after heat treatment at 150°C for 168 hours. Both 0.001-in. diameter round wire and 0.0015-in. by 0.00055-in. ribbon wire were tested. The wires were cut to approximately bond length and mounted vertically in epoxy. They were pulled to breaking with the hot-melt-glue bond puller. The results of this test, which are summarized in Table 5, suggest that the ribbon wire is degraded less by the heat treatment than the round wire.

Scanning Electron Microscopy — Scanning electron microscopes have been widely used in studying wire bonds and other parts of semiconductor devices because of the high resolution and large depth of focus. Near the end of this quarter, NBS acquired a new scanning electron microscope which is available for use by this and other tasks in the program. Initial activity was devoted principally to operator training; a number of bonds were scanned in addition to the study of aluminum balls reported above.

Pull Test Evaluation — A series of experiments was begun to determine the effect of pull angle on the strength of wire bonds as measured

Table 5 - Pull Strength of Aluminum - 1 Percent Silicon Wire

Spool	No heat treatment			After heat treatment[a]			Percent
	No.	Avg.[b]	s[c]	No.	Avg.[b]	s[c]	difference[d]
#1 - Round	19	11.2 g	0.95 g	23	8.2 g	0.66 g	26.8
#2 - Round	26	11.7	0.80	16	7.7	0.42	34.2
#1 - Ribbon	15	10.7	0.60	15	8.8	0.70	17.7
#1 - Ribbon	35	11.2	0.72	43	8.9	0.70	19.9

[a] Heat treated at 150°C for 168 hours

[b] Average force required to break wire

[c] Sample standard deviation

[d] Percent difference of averages

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by the pull test. A group of similar loop-type bonds is prepared by first gently straightening the wire with a regular hook-type puller and then cutting each loop at its highest point. Each bond is then pulled to destruction at the desired angle with the hot-melt-glue puller. The initial tests involve pulling at angles of 30, 45, 60, and 90 deg from the plane of the wafer surface. (K. O. Leedy)

Improvements in the bond pulling apparatus are being made in order to speed up both the process of bond pulling and the recording of data. These improvements include a means of recording on a graph the force at which the bond fails. (H. K. Kessler)

Bibliography and Critical Review — The collection of papers for the bibliography has taken longer than anticipated. The search for reports of government-sponsored work is essentially complete except for scanning *Scientific and Technical Aerospace Reports* for NASA reports that may not have been included in the list previously provided by DDC. Research of journal literature is about 95 percent complete. Almost 70 articles and reports that are considered significant have been assigned keywords for their inclusion in the bibliography.

A detailed outline of the critical review survey paper has been completed, and the writing of the paper itself has begun. The paper is expected to include the following sections:

I. Introduction

II. Types of Wire Bond Systems and their Fabrication (thermocompression, ultrasonic)

III. Factors in the Fabrication of Highly Reliable Wire Bond Systems (wire bond system components and fabrication processes, associated assembly processes)
IV. Stress-Induced Degradation and Failure (functional, temperature, electrical, radiation stresses)

V. Evaluation of Wire Bond Systems (purpose, description, evaluation, and correlation of observational, mechanical, in-process, electrical, thermal, and radiation tests)

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

Plans: Methods for studying bonding tool motion in the laboratory will be extended to studies of the actual bonding process on bonding machines. A simple, inexpensive milliwatt laser interferometer will be constructed and used in conjunction with the microphone for studying tool and transducer motion. Additional methods of ultrasonic bond monitoring will be studied. A linear sweep frequency drive will be introduced to permit monitoring of the resonance Q spoiling during the actual bond formation.

Investigations of the effect of burn-in at various temperatures on the tensile strength of wire and wire bond systems will continue. Experiments will be designed statistically to evaluate the importance of various factors in the pull test. Work on the modifications to the bond puller will continue.

The scanning electron microscope and microprobe will be used for studying sections of bonding wire including some with which good bonds could not be made in an effort to evaluate inhomogeneities which might be responsible for its poor performance.

The study of the effect of metallization sintering time and temperature on ultimate wire bond strength will resume. Work on ribbon wire will resume when new, harder wire is received. A search for damage to the silicon substrate by this hard wire will be made; damage will be detected by means of preferential etching of the silicon after removal of the bond and aluminum metallization pad.

Work on the bibliography and critical review will continue. Both are expected to be completed by next summer.

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1. P. M. Uthe, *UTI Tech. Newsletter* 1, No. 5 (August, 1968).
2. Other methods such as the laser interferometer system described by B. D. Martin at the New York State Technical Services Symposium on Lasers and their Application in Local Industries (Binghamton, N. Y., April 14, 1969) may also be used to observe the tool tip vibration.
3. These measurements were carried out with a laser interferometer of the NBS Vibration Measurements Section temporarily adapted to this use with the cooperation and assistance of L. D. Ballard.

13. DIE ATTACHMENT EVALUATION

Objective: To evaluate methods of detecting 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 for techniques utilized in evaluating the uniformity and quality of semiconductor device die attachment has provided general guidelines for the progress of the program to date. Due to the small number of relevant papers, continued emphasis on this area does not appear justified other than to maintain an awareness of current progress. Therefore, no comprehensive bibliography or review paper is planned at this time.

To determine the sensitivity of radiographic techniques for detecting voids in the die attachment of the devices to be studied, radiographs were taken of TO-5 headers with holes drilled through the 3.0-μm thick gold layer that is plated over the header shell. The TO-5 headers used in this study consist of a gold plated iron-nickel-cobalt alloy shell with a compatible borosilicate glass seal around the leads. This type of header was chosen for its compatibility with the power rating of the diodes being fabricated on chips 50-mils square. Initial results indicate that radiographic techniques will be of use in determining the size and location of the voids that are deliberately incorporated into the die attachment system.

The processing of the diode chips to be used in the die attachment studies was initiated (see Section 11). (F. F. Oettinger)

Plans: The processing of diode chips to be used in the void detection studies will be continued and die-bonding equipment will be assembled. Following these, bonding of diode chips to headers with controlled voids will begin.

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: Additional assistance was given ASTM Committee F-1 in the development of standard procedures for leak testing. The procedure for determining the hermeticity of electron devices with a helium mass spectrometer leak detector was extensively revised. (W. M. Bullis)

Plans: Assistance to ASTM Committee F-1 in the development of standard procedures for leak testing will continue. Further revisions will be made in the helium mass spectrometer procedure; the bubble test procedure will also be modified. Further review of NASA test methods [1] will be undertaken with emphasis on the correlation of the precision needed to meet the requirements for microcircuit line certification [2] and the precision to be expected from these test methods.

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1. "Test Standards for Microcircuits," Draft of NASA-STD-XX-3, October 1, 1969.
2. "Microcircuit Line Certification," Draft of NASA-STD-XX-6, December, 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: Assistance was given to Task Group 1 of the JEDEC Committee JS-6, on Power Transistors, in the revision of the initial drafts of the chapter, "Users Guide for Power Transistors" which is to be part of a JEDEC suggested standard titled "Recommended Standards for Power Transistors." (H. A. Schafft)

Plans: Assistance to the JS-6 task group in the writing of "Users Guide to Power Transistors" will be continued. The section on failure modes will be revised to include suggestions made during the task group and committee meetings.

16. THERMAL PROPERTIES OF DEVICES

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

Progress: The literature search and the review of the methods of measurement of thermal resistance and transient thermal response of semiconductor devices was continued. The writing of the first draft of the review paper was initiated. (M. Sigman and F. F. Oettinger)

Previously reported, preliminary measurements of thermal resistance, R_θ, and d-c current gain, h_{FE}, made on a limited number of transistors (NBS Tech. Note 495, pp. 33-36) indicated that a correlation exists between the onset of lateral thermal instability and the abrupt changes in R_θ and h_{FE} which occur at a particular value of collector-emitter voltage, V_{CE}. This result suggested the possibility that h_{FE} measurements can be used as a screen to detect the onset of thermal instabilities. To facilitate the study of the use of h_{FE} as a screening technique it was decided to design and build an h_{FE} measuring system that would allow greater speed and flexibility in the measurements.

A block diagram of this apparatus, which generates and plots base current, I_B, as a function of collector-emitter voltage V_{CE}, for constant collector current, is shown in Fig. 15. A constant-voltage power supply with a small (2-Ω) current-sensing resistor in its negative lead is connected across the collector-emitter terminals of the device under test, DUT. This 300-mA, 500-V power supply has been modified so that the output voltage can be swept at rates which are limited to 5 Hz or less by the mechanical response of the x-y recorder. The I_B - V_{CE} characteristics showed no noticeable difference between manual operation which required 5 minutes to complete the sweep, and electrically generated sweep rates of up to 5 Hz. The x-axis of the x-y recorder is made proportional to the collector-emitter voltage. The operational amplifier (I_B SERVO) compares the potential across the current sensing resistor with that of a negative reference voltage and adjusts I_B to keep I_C constant. A portion of the voltage drop across the 100-Ω resistor in series with the base of the DUT is applied to the y-input of the x-y recorder. Since I_C is held constant, I_B is proportional to the reciprocal of h_{FE}. The case temperature of the DUT is kept constant at 25°C by means of a servo controller not shown in the figure.

A representative I_B - V_{CE} characteristic is depicted in Fig. 16. As V_{CE} is increased from V_A to V_B the transistor operates in a normal mode. If V_{CE} exceeds V_B, a hot spot forms and I_B increases abruptly. If V_{CE} is increased further, I_B increases until second breakdown occurs. If, on the other hand, V_{CE} is varied between V_B and the lower voltage V_C, the transistor operates in a stable, current-constricted mode. If V_{CE} is further reduced, the transistor returns to the normal non-constricted mode at V_C and remains in this mode for lower values of V_{CE}. If V_{CE}

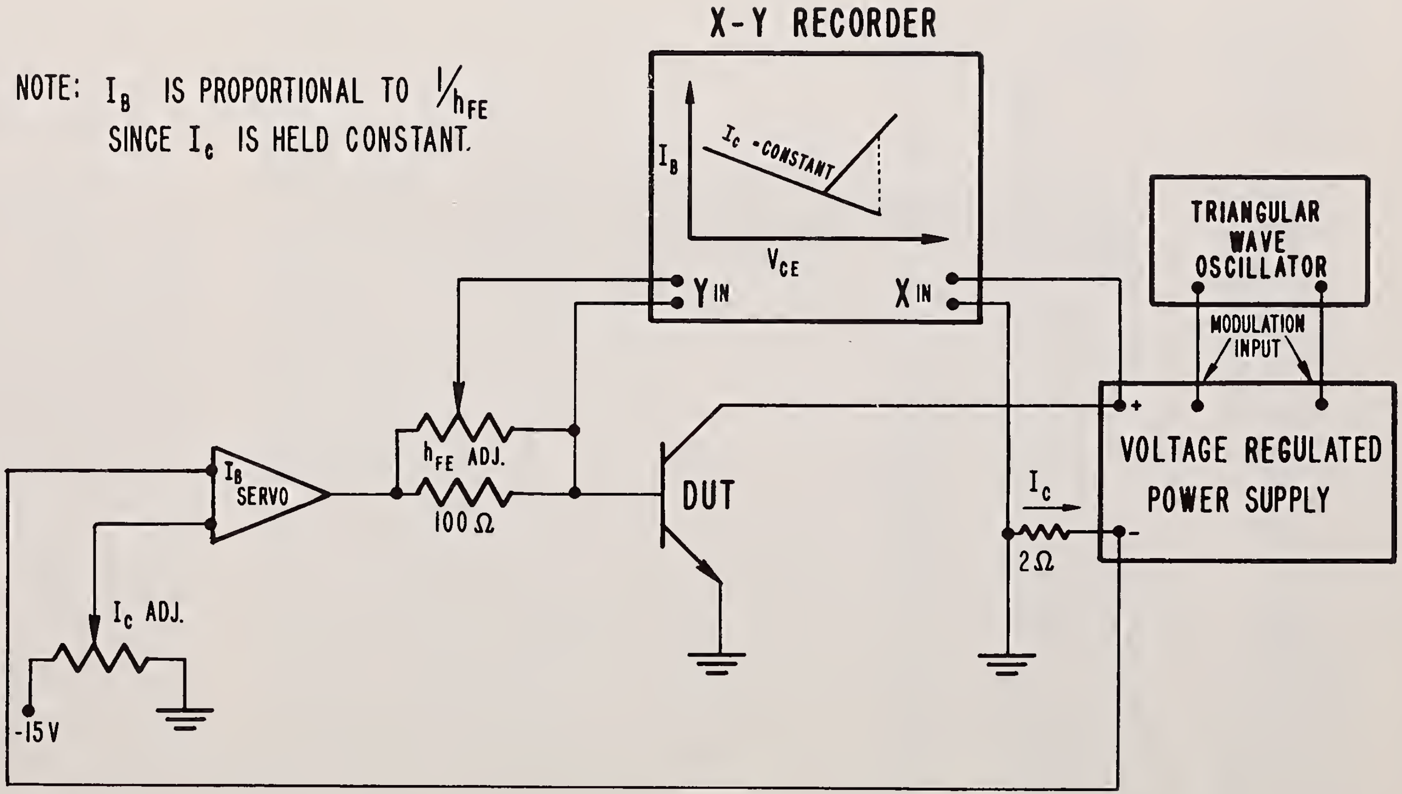

Fig. 15. Diagram of apparatus used to plot automatically curves of transistor base current as a function of the collector-emitter voltage with the collector current held constant.

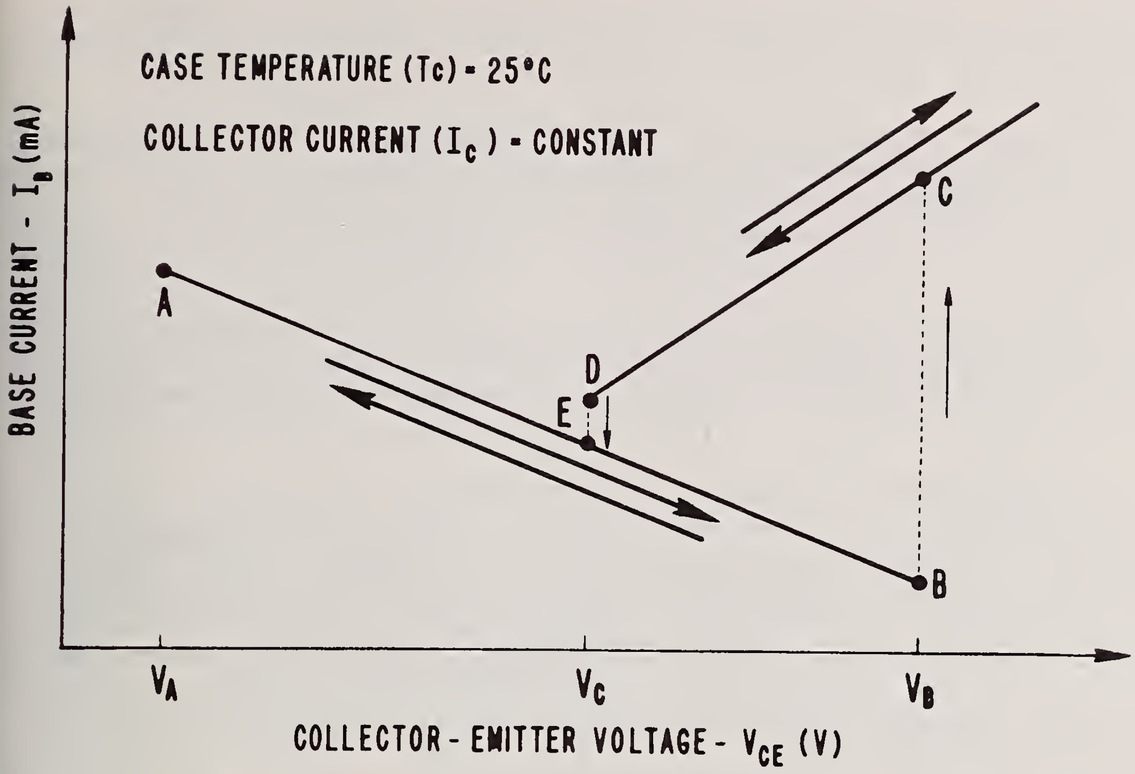

Fig. 16. Automatically plotted curve of base current versus collector-emitter voltage for a planar, diffused, power transistor.

is then increased, the transistor operates along the line AEB unless V_B is again exceeded.

To interpret the characteristics traced in Fig. 16 consider the following: Initially, there is an increase of h_{FE} with temperature due to the increased power dissipation [1] as V_{CE} is increased from V_A to V_B. At $V_{CE} = V_B$, a hot spot develops, and h_{FE} abruptly decreases. This may be explained by making use of the observation of Whittier and Tremere [2] that in thin-base, diffused, planar transistors, the base transport factor drops abruptly when a critical current density is exceeded. Since hot-spot formation is caused by a rapid current constriction, the associated increase in current density can account for the rapid decrease in the base transport factor which in turn causes h_{FE} to decrease. After the formation of the current constriction, a decrease in V_{CE} causes an increase in h_{FE} as indicated by that portion of the curve between C and D in Fig. 16. The reason for this hysteresis effect is not wholly understood, but effects being considered as possible causes are the reversal of the current gain-temperature dependence at high current density and temperature levels [1], as well as the dependence of h_{FE} on current density [2]. At point D, the hot spot is sufficiently cool that the current density in the constriction is not maintained above the critical value noted above and h_{FE} increases abruptly to its value prior to the onset of the current constriction (point E). Initial studies in which thermographic phosphors are used to display visually and measure transistor surface temperature distributions (see Section 17) substantiate the existence of the hot spots postulated in this interpretation of the h_{FE} - V_{CE} traces. (S. Rubin, R. L. Gladhill, and F. F. Oettinger)

Plans: The literature search will continue. The format and key words for the bibliography will be established. The first draft of the review paper on methods for measurement of thermal resistance and transient thermal response is expected to be completed in the next quarter.

Construction of an apparatus to automate the measurement of R_θ by the emitter-base voltage technique will be studied, and if warranted, undertaken. Further measurements of h_{FE} as a function of V_{CE} will be made on transistors representing different production techniques and geometries with the automated h_{FE} apparatus. The results of these measurements will be compared with thermographic phosphor measurements (see Section 17) to determine the suitability of the h_{FE} measurement to detect the onset of thermal instabilities and to study further the physical basis for the noted curves.

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1. D. Buhanan, "Investigation of Current-Gain Temperature Dependence in Silicon Transistors," *IEEE Trans. Electron Devices* ED-16, 117-124 (1969).
2. R. J. Whittier and D. A. Tremere, "Current Gain and Cutoff Frequency Falloff at High Currents," *IEEE Trans. Electron Devices* ED-16, 39-57 (1969).

17. THERMOGRAPHIC MEASUREMENTS

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

Progress: The photometric equipment for recording the temperature distribution on the surface of a transistor chip has been used to confirm that the sudden change in h_{FE} illustrated in Fig. 16 (see Section 16) coincides with the formation of a hot spot on the transistor. Furthermore, once the hot spot has formed, the collector-emitter voltage must be lowered considerably before the hot spot disappears, again following the pattern indicated in Fig. 16. Work in under way to measure changes in the area of the hot spot as collector-emitter voltage is reduced from the voltage at which the hot spot occurs.

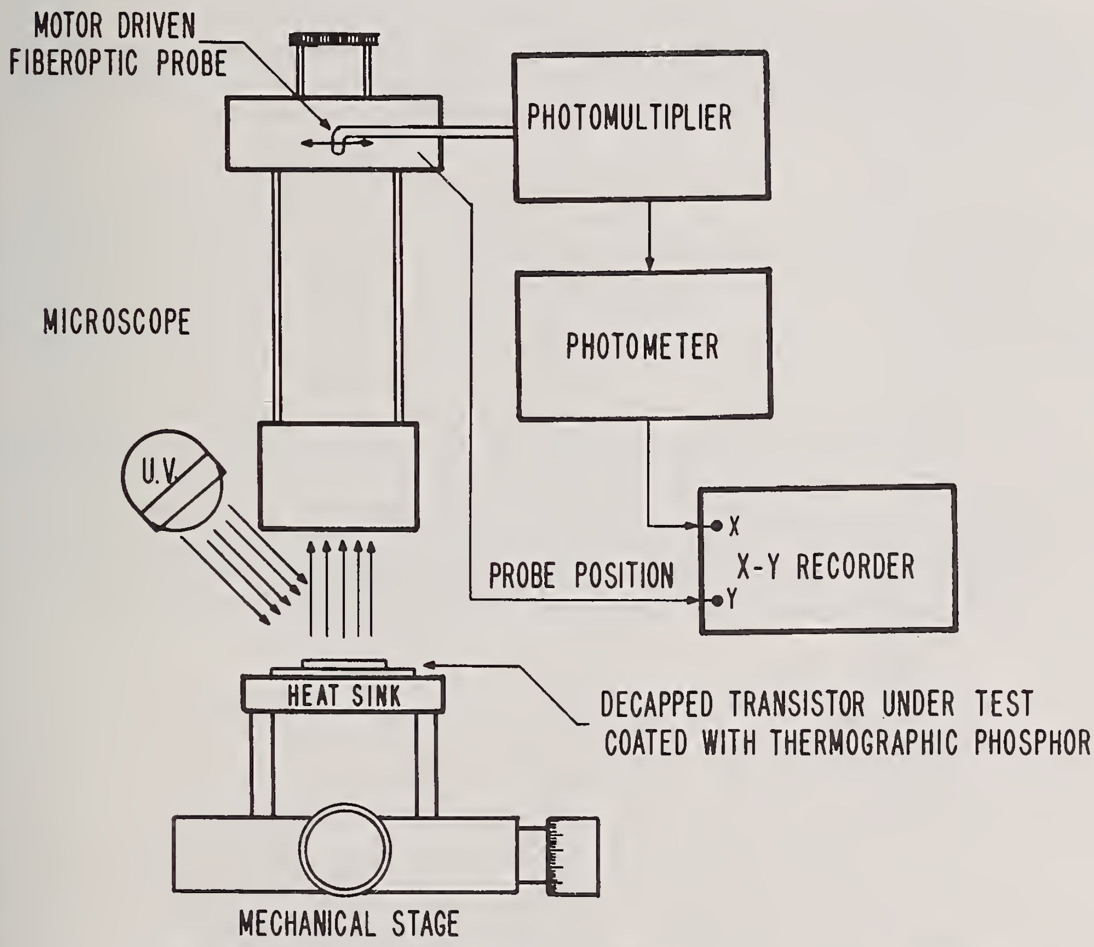

Fig. 17. Test equipment for observation of hot spots on the surface of a decapped transistor.

The equipment used in this study is shown schematically in Fig. 17. The fiber optic probe is located effectively in the image plane of the microscope and views an area approximately 60 μm in diameter on the surface of the transistor. The transistor can be moved by means of the mechanical stage so that any desired area on the surface can be brought under observation of the probe. The probe can also be driven along a diameter of the image plane to scan a path across the surface of the transistor. An electrical output coupled to the probe drive provides an input to the x-axis of the recorder that is indicative of the position of the probe. The input of the y-axis of the recorder is obtained from the output of the photometer. Because of the thermographic phosphor coating on the surface of the transistor, the light radiated from the transistor is proportional to temperature, so that the photometer output is proportional to temperature, and the recorder can be used to plot temperature as a function of position.

(G. J. Rogers, F. F. Oettinger, and L. R. Williams, Jr.)

Plans: Studies of hot-spot formation in transistors will be continued to determine the extent and temperature of the hot spot as collector-emitter voltage is varied. Studies of the spatial resolution and temperature resolution of the phosphors will be resumed.

18. MICROWAVE DEVICE MEASUREMENTS

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

Progress: Continuing discussions have been held with representatives of the Electronic Industries Association, the IEEE Microwave Theory and Techniques Group, the Naval Electronic Systems Command, the Naval Applied Science Laboratory, and others to determine further the nature and priorities of the work which is to be undertaken on microwave diodes and transistors. Work was started on error analysis for transistor scattering parameter measurements in cooperation with the JEDEC Committee JS-9, on Low Power Transistors, Task Group on Transistor "S" Parameter Measurement Standards which held its initial meeting at the National Bureau of Standards on September 30.

Radio frequency bridge network apparatus has been assembled for measurement of microwave transistor and diode impedance as a function of bias from 0.05 to 1 GHz. Slotted line and reflectometer or network analysis equipment is being assembled. The primary purpose of this type of measurement is to determine and compare errors associated with the most common techniques for determining transistor scattering parameters.

An X-band sweep-frequency generator has been incorporated into equipment for measuring reflection and transmission of two-port waveguide devices from 8 to 12.4 GHz. The primary purpose of this set up is to determine the r-f characteristics of diode mounts and therefore the impedances associated with microwave mixer diodes. This is necessary for evaluating mixer-diode, noise-measurement techniques. (R. C. Powell)

Plans: Detailed studies are expected to begin next quarter. Apparatus for measurement of mixer diode noise will be assembled to the extent that equipment is available. Evaluation of errors associated with microwave transistor scattering parameters will continue. The equipment for measuring reflection and transmission at X-band will be refined by the addition of such elements as precision attenuators and calibrated power meters to obtain the improved accuracy which will be needed.

19. 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: As part of the continuing effort to evaluate large-diameter silicon crystals for Si(Li) detectors, two dislocation-free crystals were characterized. The nominal values of the parameters measured on these crystals are given in Table 6 together with results of measurements on two 67-mm diameter crystals reported earlier (NBS Tech. Note 475, p. 30). The long-time component of carrier lifetime given in column 3 of Table 6 is an indication of single or multiple trapping of charge carriers. Note the interesting fact that only crystal 2 which had no long lifetime component has a low oxygen concentration. All crystals exhibited excellent junction rectification properties after the diffusion of lithium. During the compensation process, the lithium drift-rate was very low on the three crystals which contained large amounts of oxygen.

The principal problem with large-diameter silicon crystals appears to be the relatively high concentration of oxygen found in most crystals not grown by the vacuum float-zone process. The oxygen impedes the process of charge compensation by reducing the lithium-ion mobility. Efforts to develop silicon crystals with diameters greater than 50 mm are being made at several laboratories in this country. However, the principle objective of these efforts is to provide larger silicon crystals which would increase the yield of semiconductor devices and circuits on a single wafer rather than to develop high-resistivity, low-oxygen, detector-grade material.

The investigation of charge carrier lifetime in detector-grade silicon has continued with the collection of eleven silicon crystals with differing values of resistivity and lifetime. The nominal range of resistivity is 150 to 3000 Ω-cm as determined by a two-probe measurement made on the long dimension of the crystals; the range of PCD lifetime is 12 to 12000 μs. Methods to be used in this study for measuring the lifetime in diodes made from these crystals are being developed (see Section 3).

Switching circuits were developed for the detector test system which will automatically monitor the current and noise of several hundred detectors both on bias and in storage. It was found that the contribution of noise from the switching network was not a significant portion of the total detector system noise. This system is expected to be in operation within four months. (B. H. Audet)

Silicon avalanche-type radiation detectors [1] are being evaluated for possible use in space radiation experiments for the high-rate counting of low-energy electrons or protons. Two detrimental characteristics

Table 6 - Nominal Values of Several Parameters of Silicon Crystals for Si(Li) Detectors

Crystal	Diameter	Resistivity	Lifetime[a]	Oxygen[b]	Carbon[b]
	mm	Ω-cm	μs	cm^{-3}	cm^{-3}
1	50[c]	54	225, 6250	1.3×10^{18}	$<1.2 \times 10^{16}$
2	31[c]	500	470, none	$<2.4 \times 10^{15}$	$<1.2 \times 10^{16}$
3	67	77	210, 1950	1.8×10^{18}	$<1.2 \times 10^{16}$
4	67	66	180, 2350	1.7×10^{18}	$<1.2 \times 10^{16}$

[a] photoconductive decay lifetime, short-time and long-time components

[b] determined from infrared absorption measurements

[c] dislocation-free crystals

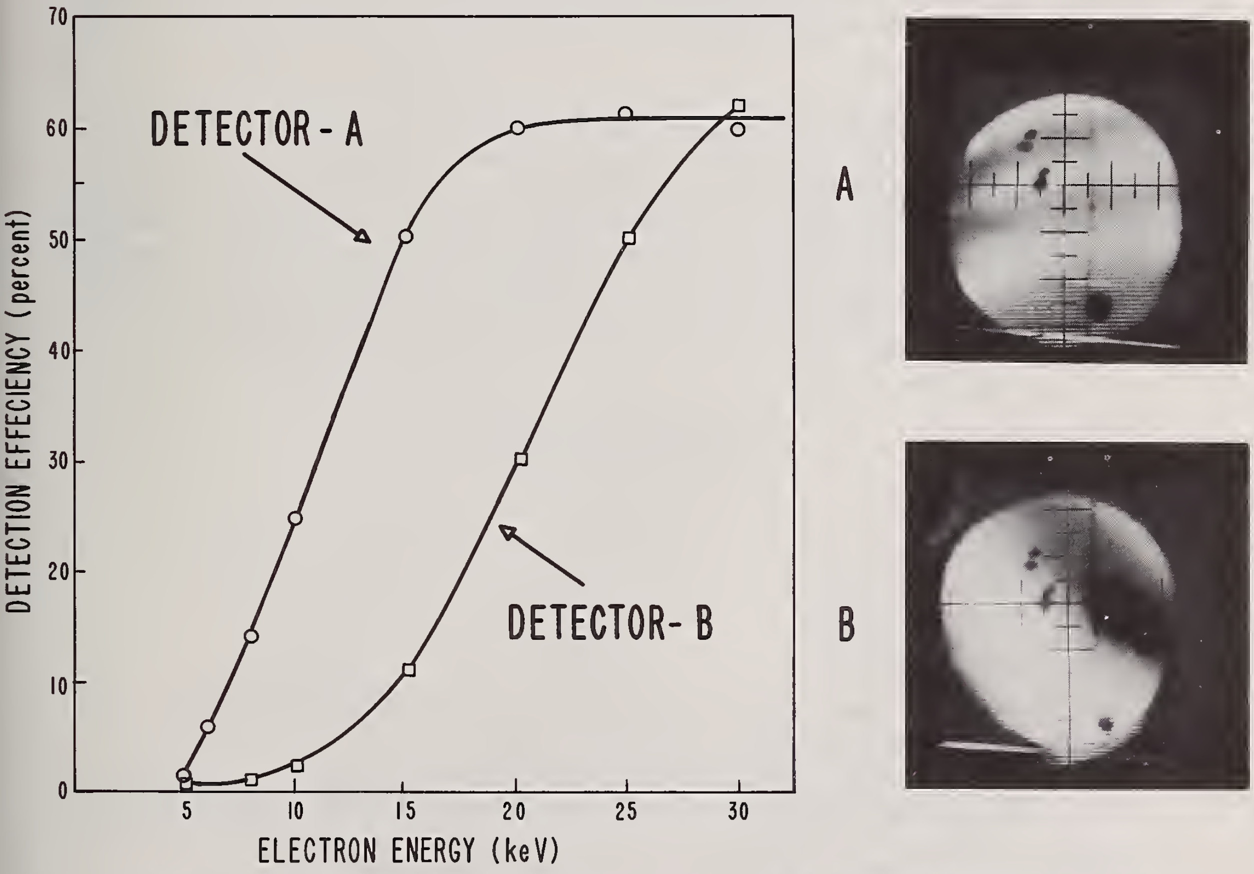

Fig. 18. Relative counting efficiencies of two silicon avalanche detectors as a function of electron energy. Optical scans of each detector at the right show that the bright areas, which indicate a high electric field, are more extensive in A than in B, thus accounting in part for the higher counting efficiency of A at low energies. (Photographs provided through the courtesy of G. C. Huth.)

of avalanche detectors which reduce the counting efficiency of low-energy electrons are the dead layer of about 1 μm at the sensitive surface, and the relatively large regions of significantly lower electric fields at the junction. The relative counting efficiencies for electrons as a function of electron energy are shown for two representative detectors in Fig. 18. Also shown are the areal scans of each detector using visible light with the detectors at their normal operating bias [2]. The regions of high electric field are represented by the bright areas while low-field regions appear darker. The largest avalanche multiplication takes place in the high-field regions of the junction. The lower counting efficiency of detector (B) for the lower electron energies can, in part, be explained by the smaller total area of high electric field than occurs in detector (A). (Y. M. Liu)

Plans: The study of electron damage in silicon surface-barrier detectors was not pursued in this reporting period because the Van de Graaff accelerator was not available. The effects on detector performance of 400-keV electrons, which have energies too small to displace a silicon atom, will be studied next. The concern here is with the very intense flux of low-energy electrons which occur in the radiation belts around the earth.

The current, noise, and counting response of detectors made from dislocation-free silicon crystals 1 and 2 (see Table 6) will be determined. Logic circuits for the automated system for monitoring detector performance during storage, in high-vacuum, or while undergoing temperature cycling will be developed. The counting characteristics of additional avalanche detectors will be evaluated, and efforts will be made to improve their performance for detecting low-energy particles.

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

1. G. C. Huth, "Recent Results Obtained with High Field, Internally Amplifying Semiconductor Radiation Detectors," *IEEE Trans. Nuc. Sci.* NS-13, No. 1, 36-42 (1966).
2. G. C. Huth, "A Flying Spot Scanner for Direct Measurement of Electric Field-Resistivity Profiles in Silicon *p-n* Junctions," presented at the New York meeting of the Electrochemical Society, May, 1969.

Appendix A

JOINT PROGRAM STAFF

Coordinator: J. C. French
Consultant: C. P. Marsden

Semiconductor Characterization Section

Dr. W. M. Bullis, Chief

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

Semiconductor Processing Section

Dr. J. A. Coleman, Chief

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

Electron Devices Section

J. C. French, Chief

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

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[*] Part Time
[†] Guest Worker
[§] Summer
[+] Secretary

Appendix B

COMMITTEE ACTIVITIES

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

Electronic Industries Association:
- MED 32, Active Digital Circuits: F. F. Oettinger, Associate Member
- MED 41, Physical Characterization Requirements: F. F. Oettinger, TG 41.6, Thermal Considerations

Joint Electron Device Engineering Council (EIA-NEMA):
- F. F. Oettinger, Technical Advisor on Thermal Resistance Measurements, JS-9, Low Power Transistors; Technical Advisor, JS-14, Thyristors.
- R. C. Powell, Microwave Diode Specification Problems, JS-3, UHF and Microwave Diodes; Task Group on Transistor Scattering Parameter Measurement Standards, JS-9, Low Power Transistors.
- H. A. Schafft, Consultant on Second Breakdown Specifications, JS-6, Power Transistors.

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

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. Radiation detectors — (A. H. Sher and B. H. Audet)
   - a. Assistance with mounting and testing a germanium gamma-ray detector was provided for the Nuclear Spectroscopy Section.
   - b. The assembly of special silicon detectors for nuclear radiation studies by the Photonuclear Physics Section was begun.

2. Special fabrication — (G. P. Spurlock)

   High-voltage, precision resistance cards were prepared for the High Voltage Section.

3. Quartz and glass fabrication — (E. I. Klein)
   - a. The absorption cell of a methane laser was modified and improved for the Engineering Metrology Section.
   - b. Improvements in ion and thermocouple gauges were made for the Vacuum Measurements Section.
   - c. Quartz cells used for studying the ultraviolet exposure characteristics of plastics and paints were constructed for the Materials Durability and Analysis Section.

4. Hall effect specimens — (H. K. Kessler)

   Ohmic contact dots were applied to several cross-shaped Hall effect specimens cut from both single and epitaxial gallium arsenide crystals for the Harry Diamond Laboratories.†

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† NBS Project 4251424

Appendix D

JOINT PROGRAM PUBLICATIONS

Prior Reports:

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, July 1 to September 30, 1968," NBS Tech. Note 472, December, 1968. (AD 681330)

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, October 1 to December 31, 1968," NBS Tech. Note 475, February, 1969. (AD 683808)

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, January 1 to March 31, 1969," NBS Tech. Note 488, July, 1969. (AD 692232)

"Methods of Measurement for Semiconductor Materials, Process Control, and Devices, Quarterly Report, April 1 to June 30, 1969." NBS Tech. Note 495, September, 1969. (AD 695820)

Current Publications:

W. M. Bullis and R. I. Scace,* "Measurement Standards for Integrated Circuit Processing," *Proc. IEEE* 57, 1639-1646 (September, 1969).

W. M. Bullis, W. R. Thurber, T. N. Pyke, Jr.,† F. H. Ulmer,† and A. L. Koenig,† "Use of a Time-Shared Computer to Control a Hall Effect Experiment," NBS Tech. Note 510, October, 1969.

W. M. Bullis, "Measurement Methods for the Semiconductor Device Industry — A Review of NBS Activity," NBS Tech. Note 511, December, 1969.

A. H. Sher and W. J. Keery, "Variation of the Effective Fano Factor in a Ge(Li) Detector," to appear in *IEEE Trans. Nucl. Sci.*, February, 1970.

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

† Information Processing Technology Division, NBS Center for Computer Sciences and Technology.

Appendix E

Notes on Procedures for Use of a Capacitor Microphone to Measure Vibration Amplitude of the Tool Tip of an Ultrasonic Wire Bonder

Introduction — Measurements of the amplitude of the motion of the ultrasonic bonding tool by means of a capacitor microphone sensitive to ultrasonic vibrations have been used to characterize ultrasonic bonding systems. Most of the experiments were carried out on the apparatus shown in Fig. 5 (see Section 12). In this apparatus the transducer-horn-tool assembly is mounted on a micrometer stage and can be moved horizontally. The capacitor microphone is mounted on a second micrometer stage which can be moved horizontally and vertically and rotated about an axis. The entire assembly is used inside a small, bench-top anechoic chamber lined with two layers of black darkroom felt.

Microphone Extension Tip Design — To improve the spatial resolution of the capacitor microphone system, a tapered extension tip is added to the microphone. Bonding tool motion is generally studied at some fixed frequency except for determinations of mechanical Q of the transducer. For fixed-frequency measurements, the microphone-taper system need not have a flat frequency response; a reinforcing standing wave at the test frequency would tend to enhance the sensitivity. Therefore, almost any hard, continuous, tapered material may be used for the tip. The plastic cone from a tube of silicone rubber has been found to be satisfactory.

To determine the mechanical Q of a typical transducer, the frequency must be changed over a range of 4 or 5 kHz. In this case standing waves in this frequency range must be eliminated. The simplest way to do this is to construct a tip that has the dimensions desired for making the measurements and insert appropriate material into the tip to damp any possible standing waves.

A cross section of a high resolution microphone taper is shown in Fig. 19. This 1¼-in. long taper has a pick-up area 0.007 in. in diameter and is suitable for studying the motion of the bonding tool over its entire length. The pick-up area is defined by the hollow needle, A. The stainless steel sleeve, E, must not protrude more than about 0.040 in. past the diaphragm of the microphone. To eliminate standing waves over the range of interest around 60 kHz, the damping material, D, should extend within 0.040 in. of the diaphragm. In this form the tip provides at least 90-percent rejection of all signals not entering through the hole. The rejection can be increased to more than 95 percent by wrapping a layer of ¼-in. thick urethene foam around the large end of the taper, overlapping the microphone to prevent sound from entering from the rear. A slight additional improvement can be obtained by spiral-wrapping the cone with polytetrafluoroethylene tape or increasing the thickness of the outer coating, B, with a different silicone rubber.

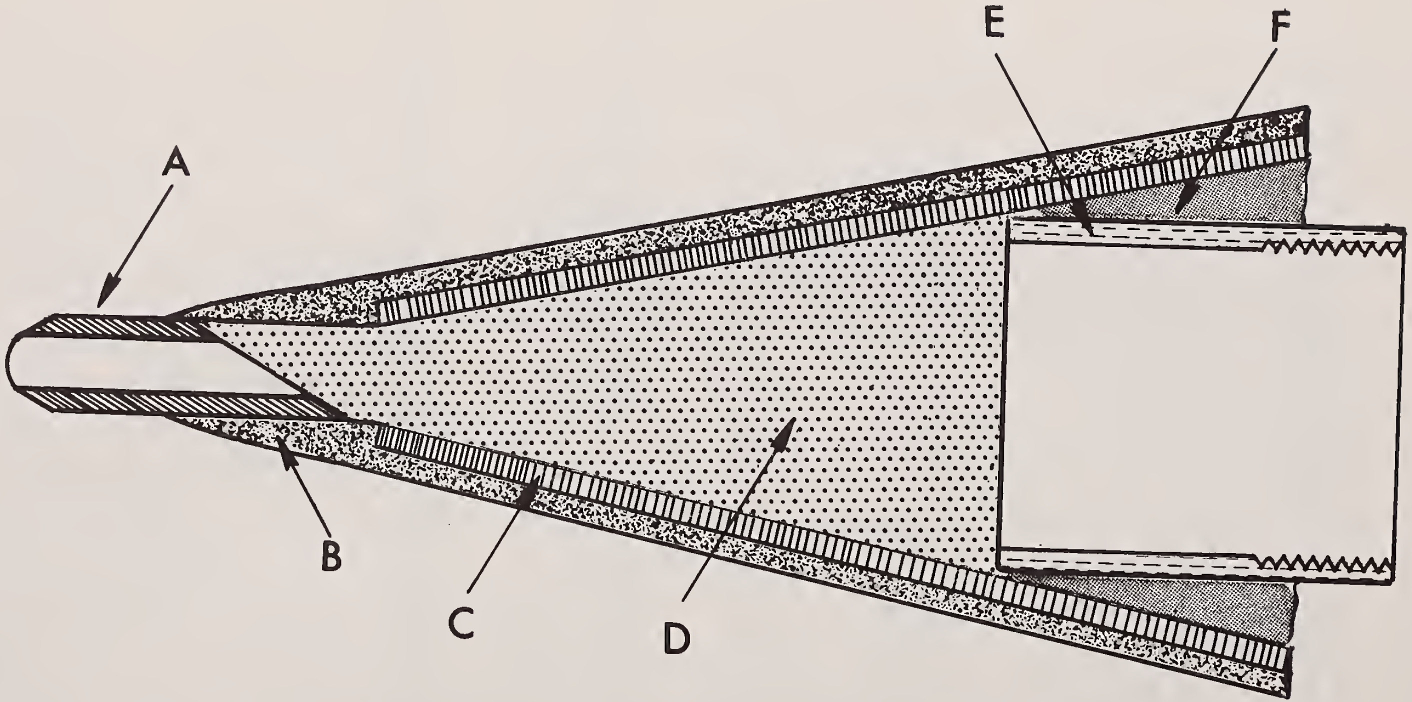

Fig. 19. Cross-sectional view of a high-resolution extension tip for the capacitor microphone.

A: Section of #27 hypodermic needle
B: Self-leveling silicone rubber coating
C: Aluminum foil (0.002-in. thick) wound as a cone
D: Cotton or open-pore plastic foam damping material with almost no reflection adjusted in length to have about 50 percent absorption
E: Stainless steel sleeve fitted tightly over the end of the microphone
F: Epoxy or silicone rubber bond between the sleeve, E. and the aluminum cone, C. (This bond must be tight or stray signals can enter the microphone.)

Many specialized cones and tips have been designed. In one case the steel tip was seated at right angles to the cone axis so that the microphone could be placed at the side of the bonding tool and horn. In another case a compression tip was made in which the entrance to the cone was sealed with a 0.002-in. thick layer of high-performance silicone rubber. Although this offered complete environmental freedom from standing waves and noise, the pick-up area could not be decreased below a 0.02-in. diameter circle. This was adequate for many measurements. In operation the tip was brought into direct contact with the tool or horn. It produced a good signal and did not mechanically load the system noticeably.

Acoustical Properties of Materials — Simple arrangements of the microphone and horn may be used to determine, with accuracy suitable for these studies, the reflectivity or absorption of plastic foams or other materials in the frequency range of interest. For reflectivity measurements, place the microphone (without the tapered tip) so that its axis is parallel to that of the transducer horn and both point in the same direction. The microphone and transducer should be about 3/8 in. apart with the microphone diaphragm placed in a standing wave node. The sample to be tested is placed with a flat area facing the microphone and transducer. For measurements of transmission through materials which are not highly reflective, place the microphone in front of and pointing toward the transducer horn, and place the sample between the two.

Most materials, such as paper, metal foil, and rubber plastic foams, are highly reflective. However darkroom felt, soft paper tissues, and a few open-pore plastic foams have almost no reflection. A single layer of black darkroom felt was found to absorb about half of the 60-kHz energy with almost no reflection. Two layers of this material used to line the bench-top anechoic chamber were sufficient to reduce reflections to a negligible level. One open-pore plastic foam which had almost no reflection was found to have relatively low absorption. This material was used for damping standing waves inside extension tips. The length of the foam was adjusted for about 50 percent absorption, the calculated absorption required to critically damp a standing wave in the tapered tip.

Measurement Techniques — The actual measurement technique depends on the quantity of interest. For recording a resonance curve to determine the mechanical Q of the system, for observing the motion of the bonding tool during swept-frequency operation, or for peak-tuning the system, the entrance hole to the tapered tip can be 0.025 in. or more in diameter. The center of the hole is placed in front of and about 0.001 in. away from the bonding tool and about 0.035 in. above the tip toward the horn. Under these conditions, a signal of several millivolts is obtained directly from a capacitor microphone with a ¼-in. diameter diaphragm. These measurements are relative and can be made with the microphone in one position. The amplitude reproducibility when the microphone was removed and then replaced carefully in the same position was found to be better than ±15 percent. This limits the precision of comparisons between different runs.

For studying the vibration along the entire length of a bonding tool, an amplitude reproducibility of about ±5 percent is essential to obtain meaningful results. This was achieved by means of the following procedure. The steel tip of the microphone is advanced until it just touches the vibrating tool. The point of contact is clearly evident because hash appears on the oscilloscope wave form of the microphone output. Then the tip is retracted, about 0.0001 in., until the hash just disappears. A large and reproducible signal is obtained with this technique.

Such measurements are tedious. The bonding machine power supply must be shut off during the time the microphone is raised to its next position. This prevents heating of the transducer which would eventually detune it. Nevertheless a complete measurement such as those in Figs. 7 to 11 (see Section 12) can be taken in about 20 min.

Information about sideways vibration modes of the bonding tool can also be obtained. In this case the system is tuned to resonance with the microphone in front of the tool. The microphone is then rotated 90 deg to the side of the tool, and the system is retuned to a new maximum if one exists. Sideways modes with amplitudes about one fourth that of the main resonance have been observed about 250 Hz from the main resonant frequency. These characteristics are dependent on the length of the tool extension. In order to observe weaker sideways modes it is necessary to compare resonance curves from the front of the tool to those taken at the side. For practical purposes such weak modes would appear to have little effect on bonding.

Calibration — For absolute measurements it is necessary to calibrate the microphone response by an independent means. A laser interferometer system can be used for this calibration. Although preliminary measurements have been made (see Section 12) detailed calibration procedures have not yet been worked out.

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