NATIONAL BUREAU OF STANDARDS REPORT

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STANDARD MEASUREMENTS OF THE RESISTIVITY OF SILICON BY THE FOUR-PROBE METHOD

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STANDARD MEASUREMENTS OF THE RESISTIVITY OF SILICON BY THE FOUR-PROBE METHOD

by

W. Murray Bullis
Electron Devices Section
Electronic Instrumentation Division
Institute for Applied Technology

Final Report to Component Standards Branch
Electronics Research Center
National Aeronautics and Space Administration

ERC Project ER-6576
(3 April 1967 to 30 November 1967)

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FOREWORD

This work represents the concluding phase of a long-range program designed to establish the technical basis for a method of measuring the resistivity of silicon slices with an interlaboratory precision such that the relative standard deviation is 1 per cent or less. This phase was supported by the Electronics Research Center of the National Aeronautics and Space Administration. Mr. L. M. Pauplis of the Qualifications and Standards Laboratory, Component Standards Branch, Electronics Research Center, was project manager for NASA. Significant contributions to the project were made by J. C. French, F. H. Brewer, L. J. Swartzendruber, and W. M. Bullis (project leader). The participation and cooperation of many industry members of the Resistivity Task Force, Subcommittee VI, ASTM Committee F-1 was instrumental in the successful completion of the work, and their contributions of time, materials, and suggestions are greatfully acknowledged.

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STANDARD MEASUREMENTS OF THE RESISTIVITY OF SILICON BY THE FOUR-PROBE METHOD

by

W. Murray Bullis

ABSTRACT

An improved standard procedure for measurement of circular silicon slices with four in-line point probes has been developed in cooperation with the Resistivity Task Force of ASTM Committee F-1. Detailed analysis of a series of round-robin experiments showed that the procedure can attain a precision of ± 2 per cent (three standard deviations) for interlaboratory comparisons of slices with room temperature resistivity between 0.005 and 120 ohm-cm. Resistivity non-uniformity in the test slices was shown to be a significant factor in limiting the precision which could be achieved. The importance of including correction factors for temperature, finite thickness, finite diameter, and unequal probe separations was demonstrated. The results of the round-robin experiments also emphasized that the precision quoted can only be achieved if the measurements are carefully and correctly made on a well maintained, accurately calibrated test system which meets the requirements imposed by the test method. Determination of the precision to be expected from the method in non-referee applications such as routine production and quality control will require additional study of such factors as surface conditions, probe force, current levels, etc. Nevertheless, use of the various procedures of the method, in particular the sections on probe and measuring circuit evaluations and on thermal sinking of the wafer, would be expected to yield significantly improved precision in such applications. Use of these procedures on a regular and widespread basis should be encouraged.

1. INTRODUCTION AND STATEMENT OF PROBLEM

The resistivity of a semiconducting material is controlled principally by the density of free carriers which exists in the material. In commercially useful semiconductors, such as silicon and germanium, the number of free carriers is tailored and controlled for particular applications by the addition of specific small quantities of impurity dopants. The electrical characteristics of semiconductor devices depend in a critical way on the free carrier density in the various regions of the device structure. Because of this basic importance of resistivity, and of the relative simplicity with which a resistance measurement may be made by means of the four-probe method on a surface, this parameter is the one most widely used in design and production control of semiconductor devices.

For similar reasons, resistivity is the parameter which is most widely used in specifying semiconducting materials from which devices are to be fabricated. Although the standard four-probe method is quite simple in principle, the precision and reproducibility which were obtained in actual practice have been inadequate for some time. A substantial expense to the industry, and ultimately to the users of devices, arises from disagreements between vendors and users. In addition disagreements among different test sets within the same organization are frequently found when comparisons are made. Need for improved precision in measuring resistivity of silicon for high quality, high reliability devices led the industry, through the Committee F-1 on Materials for Electron Devices and Microelectronics of the American Society for Testing and Materials, to request the Electron Devices Section of the National Bureau of Standards to assist in the development of new standards with the aim of achieving a precision of 1 per cent or better (one standard deviation). The necessity for determining variations in resistivity along a slice radius which developed in connection with power devices and integrated circuits further emphasized the need for this precision.

When the project began, it was thought that sufficiently precise measurements could only be made by using the two-probe method. In this method, current of uniform density is passed through a rectangular bar with metallic contacts completely covering the ends of the bar and the potential drop is measured between two pointed or wedge shaped probes applied to the side of the bar a known distance apart as shown in Fig. 1. Careful comparisons of this method were made with the four-probe method. In this latter method, the current is passed through the outer two of four pointed probes in a linear array placed on a flat semiconductor surface and the potential drop is measured between the inner pair as shown in Fig. 1. Detailed investigations of the effects of variations in specimen surface preparation, probe force, probe diameter, and probe material were carried out. The importance of allowing for the variation of resistivity with temperature was demonstrated

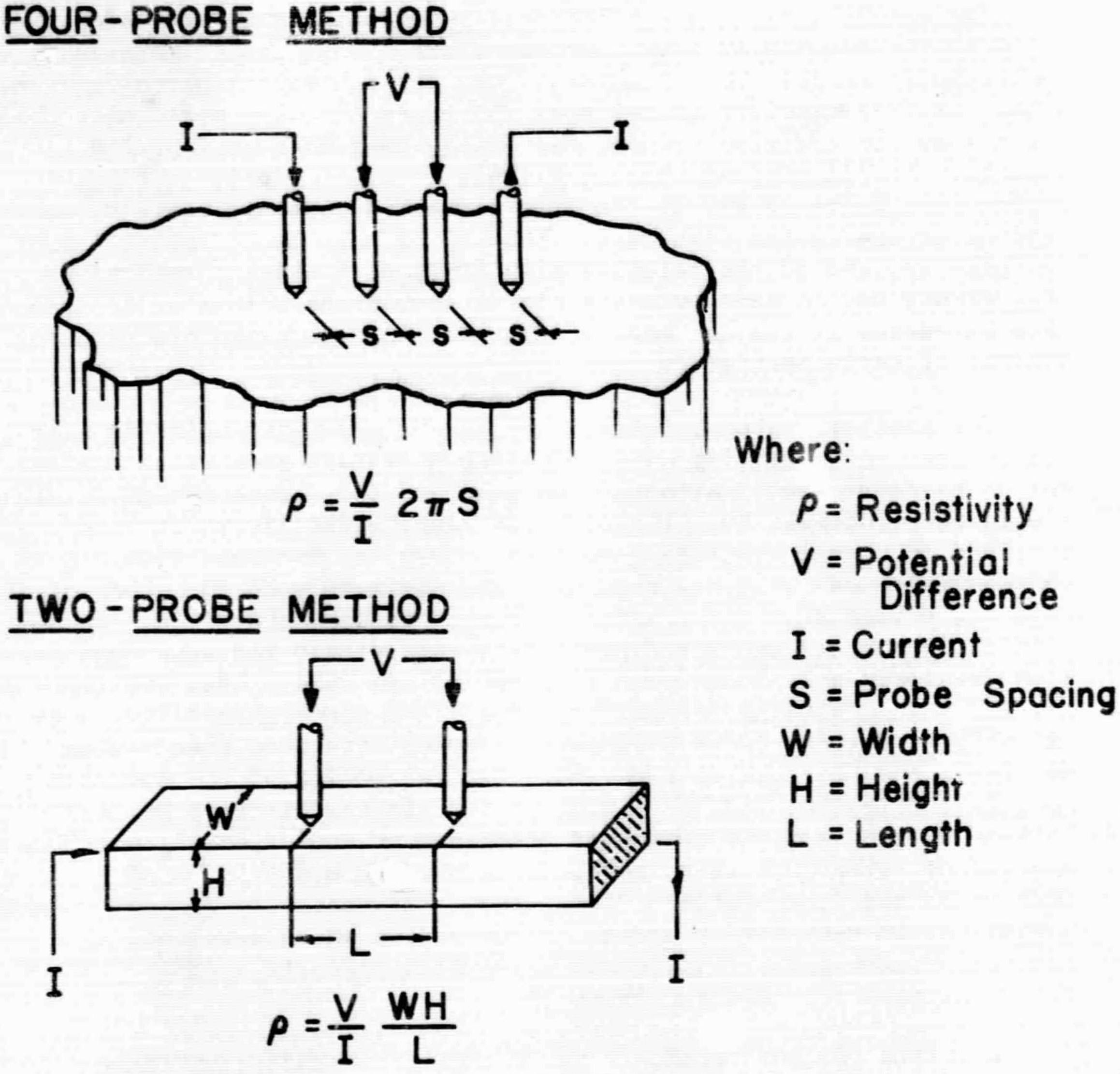


Figure 1. Probe Methods of Measurement of Resistivity.

and geometrical correction factors appropriate to both circular and rectangular wafers were computed.

These studies resulted in the development of an improved four-probe method for measuring resistivity of semiconductor slices. A round-robin experiment on several silicon slices in the 5 to 20 Ω-cm range indicated that a precision over an order of magnitude better than that obtained using the earlier techniques could be achieved. Thus, it was demonstrated that the use of the expensive and destructive two-probe method was not necessary to achieve the desired precision. The correction factor tables which were published as a part of the program have been widely used. These factors also enabled a significant improvement in off-center resistivity measurements to be made. This improvement together with the increased precision of the method enabled the accuracy of the determination of radial variations of resistivity to be improved significantly.

The present project was undertaken in order to complete the development, writing, and publication of a standard method for the measurement of the resistivity of silicon wafers suitable for use throughout the electronics industry in cooperation with the ASTM and to provide the additional effort which was necessary to extend and refine the method for maximum usefulness. These added efforts involved: (1) extension of the application of the four-probe method to the most widely used resistivity ranges of silicon, (2) establishment of the precision of the method in the various resistivity ranges, (3) more precise establishment of the environmental control and geometrical requirements of the method, and (4) participation with ASTM in the writing of an industry standard for the measurement.

To accomplish these objectives the following tasks were performed:

1) Results of two round-robin experiments being carried out by the Resistivity Task Force of Subcommittee VI of ASTM Committee F-1 were analyzed. These experiments were in progress at the inception of this project and were completed in June 1967.

2) Experimental studies were carried out to establish the environmental control and geometrical requirements of the method and the relative influence of these factors on the precision of the method.

3) The procedure for making four-probe resistivity measurements on silicon slices was extended to include the entire resistivity range between 0.0005 and 2,000 Ω-cm.

4) A new draft of the resistivity standard based on the results of the above study was written and submitted to the Resistivity Task Force for review comment. It

is expected that this draft will appear as an ASTM Tentative Method in the 1968 Book of Standards.

Work on these various tasks is reported in detail in the following sections.

2. ANALYSIS OF THE ROUND-ROBIN EXPERIMENTS

2.1 INTRODUCTION

Two round-robin experiments covering a wide spectrum of resistivity were carried out between November 1966 and June 1967 by the Resistivity Task Force of Subcommittee VI, Committee F-1 on Materials for Electron Devices and Microelectronics of the American Society for Testing and Materials to determine the limits of precision for a Method of Test for Resistivity of Silicon Slices Using Four Point Probes. Earlier, a preliminary round-robin experiment consisting only of three specimens about 10 Ω-cm had been carried out to establish the feasibility of the method. In the near future a fourth round-robin experiment is planned in order to extend the method to lower resistivity (∿ 0.001 Ω-cm) and to recheck the precision in the 1000 Ω-cm range.

This section summarizes the results of the two wide-spectrum experiments. For completeness, the report on the preliminary experiment which was originally presented to the Task Force at the Chicago meeting June 1966 is included as Appendix A. Laboratories which had participated in the preliminary experiment participated in one of the new experiments:

Bell Telephone Labs., Allentown, Pa.,
Dow Corning Corp., Electronic Products Div., Hemlock, Mich.,
IBM Corp., Components Div., Hopewell Jcn., N. Y.
Monsanto Co., Inorganic Chemicals Div., St. Louis, Mo., and
National Bureau of Standards, Washington, D. C.

Except for NBS, laboratories in the second had not participated in earlier experiments on resistivity:

Autonetics, Anaheim, Calif.,
Fairchild Semiconductor, Mountain View, Calif.,
General Electric Co., Syracuse, N. Y.,
NBS, Washington, D. C.
Western Electric Co., Allentown, Pa., and
Westinghouse Electric Co., Youngwood, Pa.

The original plan was to have 5 n-type and 5 p-type slices in each experiment. Each series was to include specimens with resistivity about 0.01, 0.1, 10, 100, and 1000 Ω-cm. Except for the fact that the 10 Ω-cm n-type slices were improperly typed and turned out to be p-type, this plan was carried out. Three slices were broken during the tests; two were replaced so that data could be obtained. The 0.01 Ω-cm p-type slice in the second test was not replaced. Slices were prepared according to the test method (see Appendix F - ¶7 Preparation of the Test Specimen) by the laboratory which supplied them. Analog circuits of resistance 0.001, 0.01, 0.1, 10, 100, and 1000 Ω were furnished for both tests. The 10, 100, and 1000 Ω analog circuits contained, as the standard, commercial precision (±0.05 per cent) resistors and, as the large series resistors, ordinary carbon composition (±10 per cent) resistors. The other standard resistors were fabricated from various wire of appropriate diameter and length.

2.2 EXPERIMENT 1.

In this experiment, the ten silicon wafers and six analog circuits were furnished to each of the participants in turn together with suitable data sheets. Wafers from crystals 605603, 601333, 71983, and 71166 were n-type; the remainder were p-type. The procedure governing the tests was "Proposed Method of Test for Resistivity of Silicon Slices Using Four Point Probes", Third Draft, December 1, 1966.[1] Since this procedure pertained only to specimens in the 10 to 20 Ω-cm range a separate schedule of currents to be used in the test was also supplied:

Range (Ω-cm or Ω)	0.001	0.01	0.1	10	100	1000
Current (mA)	50	50	30	0.3	0.1	0.02

2.2.1 Results. The method specifies tests to evaluate both the condition of the probe assembly and the accuracy of the electrical measuring equipment in addition to the resistivity measurement itself. All these facets of the method were studied as part of the round-robin experiments. The results are summarized in Tables I through X. The resistivity measurements themselves are considered first (Tables I through V), followed by the probe separation measurements (Table VI) and the electrical analog circuit measurements (Table VII). Results presented in Tables VIII through X are used in the error analysis presented in paragraph 2.2.2.

2.2.1.1 Resistivity Tests. Table I lists the average resistivity of each wafer (based on ten measurements in both the forward and reverse directions of current) reported by each laboratory. The grand average (Avg.), the sample standard deviation (s) (of the averages), and the relative sample standard deviation (s(%)) (of the averages) were calculated for each wafer. Results of the preliminary round-robin had suggested that computation errors occur frequently. Hence, the reported raw data were used to recompute the averages with the use of an electronic desktop calculator programmed to yield average, sample standard deviation, and relative sample standard deviation. Results are shown in Table II, from which it can be seen that the relative sample standard deviation is less than 0.7 per cent in eight of the eleven cases. This would suggest that a precision of ±2 per cent (R3S%) for these cases could be expected most of the time if the experiment were repeated with the same care as exercised in this test. Measurements on the 100 Ω-cm and 1000 Ω-cm p-type wafers and one of the 1000 Ω-cm n-type wafers had larger sample standard deviations. Only the 1000 Ω-cm p-type wafer significantly exceeded 1 per cent. Without additional experiments it is not possible to include the 1000 Ω-cm range in the ±2 per cent precision statement above.

TABLE I - Average Resistivity (Ω-cm) at 23°C (as Reported)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s	s(%)
605603-3	0.008393	0.008378	0.008403	0.00834	0.0083343	0.008370	0.000031	0.37
601333-2	0.08540	0.08569	0.085058	0.08502	0.085577	0.08535	0.00030	0.35
71983-2	100.32	101.19	95.8052	101.725	100.65	99.94	2.37	2.37
71166-2a	836.4	842.53			835.0	838.0	4.0	0.48
71166-2b			1136.27	1044.13	932.	1037.5	102.3	9.86
600200-2	0.007763	0.007824	0.007778	0.00777	0.0077426	0.007776	0.000030	0.39
607075-2	0.10927	0.10958	0.109189	0.10883	0.10881	0.10914	0.00032	0.30
70877-3	7.916	7.937	8.0403	8.0410	7.909	7.969	0.067	0.83
49445-2	11.857	11.97	12.0045	11.735	11.877	11.889	0.106	0.89
66969-1	111.91	114.03	113.150	112.735	112.59	112.88	0.78	0.69
16603-2	940.3	979.83	981.740	967.31	941.6	962.2	20.1	2.09

TABLE II - Average Resistivity (Ω-cm) at 23°C (Recomputed)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s	s(%)
605603-3	0.008389	0.008452	0.008361	0.00830	0.0083343	0.008367	0.000058	0.69
601333-2	0.08540	0.08538	0.084723	0.08462	0.085577	0.08514	0.00044	0.51
71983-2	100.33	101.11	101.48	101.9	100.6	101.08	0.64	0.63
71166-2a	836.4	842.61			835.0	838.0	4.0	0.48
71166-2b			1122.	1040.	932.	1031.3	95.3	9.24
600200-2	0.007761	0.007818	0.007743	0.007752	0.0077426	0.007763	0.000031	0.41
607075-2	0.10928	0.10964	0.10873	0.10901	0.10881	0.10909	0.00037	0.34
70877-3	7.915	7.930	7.991	8.0267	7.909	7.954	0.052	0.65
49445-2	11.859	11.94	11.92	11.747	11.877	11.869	0.075	0.63
66969-1	111.90	114.09	112.5	114.20	112.59	113.06	1.03	0.91
16603-2	939.0	951.19	947.8	964.9	944.8	949.5	9.7	1.02

TABLE III - Sample Standard Deviation in Per Cent (Recomputed)

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.
605603-3	0.008367	0.49	0.91	1.24	0.97	0.71	0.69
601333-2	0.08514	0.10	0.19	0.30	0.29	0.32	0.51
71983-2	101.08	0.13	0.33	1.41	2.12	0.38	0.63
71166-2a	838.0	0.27	0.33			1.53	0.48
71166-2b	1031.3			8.82	0.30	1.24	9.24
600200-2	0.007763	0.15	0.08	0.18	0.08	0.20	0.41
607075-2	0.10909	0.16	0.25	0.16	0.02	0.16	0.34
70877-3	7.954	0.11	0.32	1.30	0.32	0.34	0.65
49445-2	11.869	0.14	0.12	1.08	0.21	0.19	0.63
66969-1	113.06	0.10	0.59	0.43	0.33	0.25	0.91
16603-2	949.5	0.23	0.87	0.82	0.68	1.15	1.02

2.2.1.2 Relative Single Laboratory Deviation. The relative sample standard deviations obtained by each of the laboratories for the sequence of ten measurements for each wafer are shown in Table III together with the sample standard deviation of the grand average for each wafer repeated from Table II. There appears to be little correlation between the deviation reported for a wafer by a particular laboratory and the per cent difference between that laboratory's average resistivity value and the grand average. However, one of the limiting factors in obtaining reproducible resistivity measurements is the uniformity of the wafer being measured. Although the method requires that the measurements be made with the center of the probe array located within ±0.25 mm of the center of the wafer, there is no way to verify from the data reported that this was actually done in every case. If uniform wafers were used in the experiment, this source of variation would not be present. That not all wafers used in the test were as uniform as would be desirable was shown by resistivity profiles of each wafer which were made at the end of the round-robin series. These were made at NBS with the use of a four-point probe which had the probe separation recommended in ¶4.3.4 of the method and which met the requirements of ¶8.1 of the method (see Appendix F). Measurements were taken at intervals of about 1 mm along two perpendicular diameters. Comparison of the single laboratory deviations in Table III with these profiles which are shown in Fig. 2 suggests that more uniform wafers show generally smaller deviations. Differences in deviation between laboratories may be due as much to differences in locating the center of the wafer as to other errors.

2.2.1.3 Single Readings. The procedure being tested by this round robin calls for ten readings to be taken on each wafer measured. Although this procedure is acceptable for referee and other comparative measurements, single readings are much more practical in production control and inspection applications. Hence several single readings were analyzed to determine how the precision is affected in this case. The result of the analysis of the sixth, first, and tenth readings are shown in Table IV. In this table the per cent difference between individual resistivity values and the overall grand average value for that wafer are listed. The per cent difference between the value of resistivity of a wafer as determined by averaging the individual values reported by the various labs and the overall average value is listed in the column headed "Avg". The relative sample standard deviation determined for each wafer is listed in the last column. If the 1000 Ω-cm p-type wafer is excluded from the discussion it can be seen that over three-fourths of the values fall within 1 per cent of the appropriate grand average value. Less than 5 per cent of the values differ by 2 per cent or more. Comparison of the sample standard deviations with those in Table II shows that the reproducibility is only moderately degraded.

2.2.1.4 Median of Three Readings. Sometimes it is possible to improve the precision of a determination over that of a single

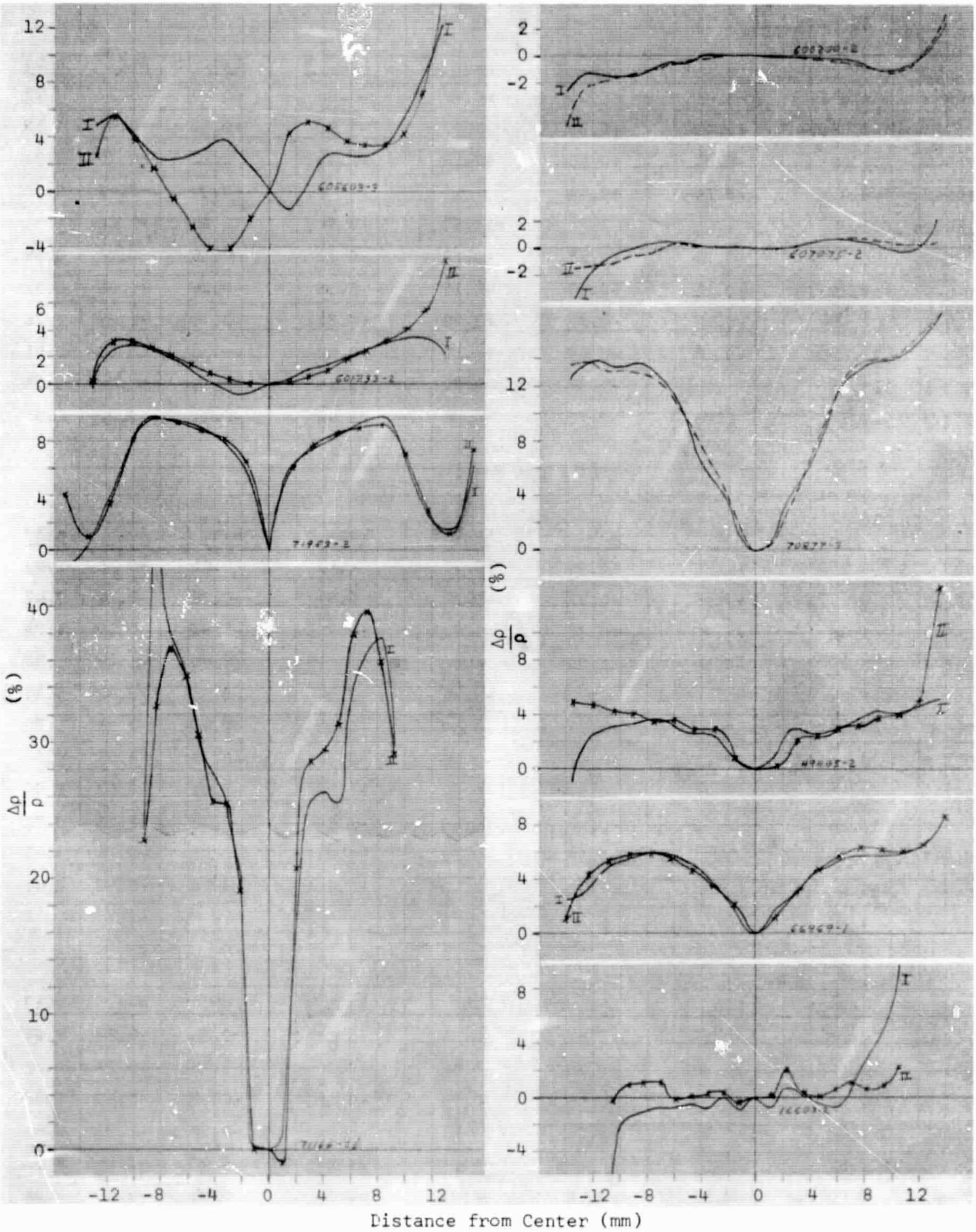


Figure 2. Specimen Resistivity Profiles for the Slices Used in Experiment 1. (No profile was made on Slice 71166-2a.)

TABLE IV - Per Cent Difference of One Reading from Overall Average Resistivity

a) Sixth Reading

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s(%)
605603-3	0.008367	-0.41	-0.55	+0.42	-0.80	+0.49	-0.17	0.49
601333-2	0.08514	+0.45	+0.36	-0.07	-0.80	+0.41	+0.07	0.53
71983-2	101.08	-0.62	+0.40	+2.15	+2.00	-0.42	+0.70	1.30
71166-2a	838.0	-0.14	+0.43			+0.99	+0.43	0.56
71166-2b	1031.3			+12.65	+0.94	-10.60	+1.00	11.51
600200-2	0.007763	-0.21	+0.63	-0.12	-0.13	-0.16	0.00	0.35
607075-2	0.10909	+0.11	+0.56	-0.11	-0.08	-0.24	+0.05	0.31
70877-3	7.954	-0.57	-0.11	+2.29	+0.83	-0.74	+0.34	1.25
49445-2	11.869	+0.08	+0.68	+1.52	-0.99	+0.38	+0.33	0.91
66969-1	113.06	-1.11	+1.67	-0.72	+1.14	-0.53	+0.09	1.23
16603-2	949.5	-1.03	-0.70	-0.36	+1.40	-0.41	-0.22	0.95

b) First Reading

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s(%)
605603-3	0.008367	+0.80	+1.96	+0.72	-2.12	-0.61	+0.15	1.60
601333-2	0.08514	+0.15	-0.21	-0.27	-0.94	+0.26	-0.20	0.47
71983-2	101.08	-0.90	+0.33	-1.22	+1.70	-0.87	-0.19	1.21
71166-2a	838.0	-0.56	+1.20			+3.45	+1.36	1.98
71166-2b	1031.3			-3.46	+0.94	-8.95	-3.82	5.15
600200-2	0.007763	-0.08	+0.71	-0.59	-0.03	-0.40	-0.08	0.50
607075-2	0.10909	-0.05	+0.47	-0.49	-0.06	-0.33	-0.09	0.37
70877-3	7.954	-0.41	-0.57	-0.02	+1.58	-1.01	-0.09	1.00
49445-2	11.869	-0.23	+0.43	+0.68	-1.60	-0.26	-0.20	0.75
66969-1	113.06	-1.21	+0.58	+0.22	+1.07	+0.05	+0.14	0.85
16603-2	949.5	-0.98	+1.23	0.00	+2.60	-0.48	+0.47	1.44

c) Tenth Reading

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s(%)
605603-3	0.008367	-0.13	+1.64	+0.78	-0.56	+0.94	+0.54	0.87
601333-2	0.08514	+0.36	+0.35	-0.41	-0.26	+1.40	+0.29	0.71
71983-2	101.08	-0.75	+0.08	-0.19	-3.05	-0.10	-0.80	1.30
71166-2a	838.0	-0.45	+0.58			-0.50	-0.12	0.61
71166-2b	1031.3			+1.20	+0.65	-7.40	-1.85	4.90
600200-2	0.007763	-0.12	+0.61	-0.01	-0.15	-0.25	+0.02	0.34
607075-2	0.10909	0.00	-0.47	-0.38	-0.07	-0.26	-0.24	0.30
70877-3	7.954	-0.58	-0.68	-0.38	+0.92	-0.19	-0.18	0.65
49445-2	11.869	-0.20	+0.43	+0.35	-0.99	+0.25	-0.03	0.59
66969-1	113.06	-1.00	+0.80	-1.20	+1.03	-0.73	-0.22	1.05
16603-2	949.5	-1.16	+0.78	+0.26	+1.66	-0.36	+0.24	1.07

measurement by taking the median value of three measurements. This involves no arithmetic and only a small amount or extra measurement time while enabling isolated "wild" readings to be avoided. To test the usefulness of this approach, the median value of the sixth, seventh, and eighth readings was analyzed with the results shown in Table V. It can be seen by comparison with Table IV that the improvement is not consistent enough over the single reading case to justify the extra labor involved.

2.2.1.5 Probe Separation Measurement. In the preliminary experiment the test for probe quality was shown to be adequate. In that experiment each laboratory measured the separations on the same probe. As a result of this experiment, it was concluded that a single laboratory relative sample standard deviation greater than 0.25 per cent in any measurement of probe separation would be considered grounds for rejection of the probe. It was also concluded that the three separations must be equal within 2 per cent for the probe to be acceptable.

In the present experiment, each laboratory furnished its own probe. The results of the probe separation measurements on the five probes used are given in Table VI. The separation (S_i), the sample standard deviation (s_i), and the relative sample standard deviation (s_i (%)) are given for each of the three separations followed by the average separation ($\bar{S}$) and the probe separation correction factor (F_{sp}). Two probes did not meet the requirements of the method. The probe used by lab 2 had separations which differed by more than 2 per cent in addition to slightly greater than acceptable deviation in two of the three separations. The probe used by lab 4 had one separation with slightly greater than acceptable deviation. Note that the probe used by lab 2 had the probe separation correction factor nearest to unity of all the probes used. No increase in the measurement spread could be attributed definitely to either of these conditions. In neither case were the requirements missed by a large amount.

Details of the derivation of F_{sp} are given in Appendix C. The 2 per cent requirement on probe separation difference is necessitated by the use of the approximate formula (C-7) for F_{sp}. Unless the exact formula (C-5) is used to calculate F_{sp}, or (as in the case of the probe used by lab 2), F_{sp} is within 0.1 per cent of unity, this requirement may not be relaxed. However, the results of this experiment suggest that the allowed relative sample standard deviation for probe separation can probably be increased to 0.30 per cent without producing an observable increase in the overall sample standard deviation.

2.2.1.6 Electrical Equipment Tests. The electrical equipment test in the preliminary round robin was successful in identifying one inadequate measuring system. The test circuit consisted of a precision resistor and four other resistors arranged as shown in Fig. 3 of the test method (Appendix F). The value of the other resistors, 300

TABLE V - Per Cent Difference of Median of Three Readings from Overall Average Resistivity

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s(%)
605603-3	0.008367	+0.33	+0.96	+0.42	-0.68	-0.83	+0.04	0.77
601333-2	0.08514	+0.40	+0.35	-0.50	-0.52	+0.41	+0.03	0.49
71983-2	101.08	-0.62	+0.23	+0.48	+1.90	-0.40	+0.32	0.99
71166-2a	838.0	-0.14	+0.43			-0.67	-0.13	0.55
71166-2b	1031.3			+12.65	+0.94	-9.82	+1.26	11.10
600200-2	0.007763	-0.18	+0.70	-0.12	-0.06	-0.36	-0.01	0.41
607075-2	0.10909	+0.18	+0.56	-0.37	-0.06	-0.33	0	0.39
70877-3	7.954	-0.54	-0.11	+2.29	+0.78	-0.54	+0.38	1.19
49445-2	11.869	+0.08	+0.68	+1.52	-0.99	+0.15	+0.29	0.91
56969-1	113.06	-1.03	+1.67	-0.66	+1.14	-0.60	+0.10	1.21
16603-2	949.5	-1.03	+0.18	+0.99	+1.90	-0.41	+0.32	1.14

TABLE VI - Probe Separation Measurement in Millimeters

	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5
S_1	1.5933	1.5657	1.5926	1.5903	1.5890
s_1	0.00053	0.00145	0.00262	0.0010	0.0010
$s_1(\%)$	0.03	0.09	0.16	0.06	0.06
S_2	1.5885	1.5850	1.5989	1.5936	1.5964
s_2	0.00043	0.00409	0.00160	0.0033	0.0008
$s_2(\%)$	0.03	0.26	0.10	0.21	0.05
S_3	1.5936	1.6096	1.5941	1.5870	1.5865
s_3	0.00069	0.00447	0.00180	0.0041	0.0008
$s_3(\%)$	0.04	0.28	0.11	0.26	0.05
$\bar{S}$	1.5918	1.5867	1.5951	1.5903	1.5905
r_{sp}	1.0022	1.00107	0.99747	0.9976	0.99608

times that of the precision resistors, is based on the work of Logan[2] who estimated that if the contact force is 0.25 N (25 gf) the spreading resistance at an osmium probe point is about 300 times the resistivity of the specimen. Present conditions of tungsten carbide probe points and a contact force of 1.75 N would be expected to reduce this ratio by about a factor of two. The larger value was selected for the test in order to allow for uncertainties in the estimate.

The results of measurements are shown in Table VII. There were no problems encountered in measuring either the 10 or 100 Ω resistors. Values reported by two labs (3 and 5) for the 1000 Ω resistor fell outside the allowed band while three labs (1, 2, and 3) reported relative sample standard deviations in excess of the allowed 0.3 per cent. Comparison of the measurements on the 1000 Ω resistor and measurements on specimens 71166-2 and 16603-2 shows the following interesting but unexplained facts:

1) In measurements on the resistor lab 3 was low and lab 5 was high; in measurements on the wafers the reverse is true. (A possible explanation of this inversion is that the measurements were made by lab 5 closer to the center of the wafers.)
2) In measurements on the wafers, labs 1, 2, and 3 did not show significantly larger relative sample standard deviations than labs 4 and 5 except in one instance.

Problems of reproducibility were encountered in measuring the three smaller resistors. Since the scatter in the resistor measurements much exceeded that in the wafer measurements it is suspected that these analog test circuits were an inadequate test of the electrical measuring equipment in the 0.01 and 0.1 Ω ranges because of unstable standard resistors. Further work will be required to eliminate this problem which probably arises from either thermally generated voltages or from temperature dependence of resistance or both. In addition to the larger deviation of average values reported for the 0.001 Ω resistor, all labs had relative sample standard deviations in excess of 0.3 per cent. No specimens in this resistivity range were included in this experiment. A separate round-robin to test this range will be started as soon as an improved standard resistor in this range can be assembled and tested.

2.2.2 Error Analysis. The following quantities are measured in the experiment:

Voltage (V)
Current (I)
Temperature (T)
Wafer Diameter (D)
Wafer Thickness (w)
Probe Spacing (S)

TABLE VII - Average Resistance (Recomputed)

a) Measured Values (Ω)

Analog Circuit	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s	s(%)
No. 1	0.000939	0.000943	0.000920	0.00096	0.0009280	0.000938	0.000015	1.63
No. 2	0.010279	0.010254	0.00998	0.01027	0.010262	0.010209	0.000128	1.26
No. 3	0.10061	0.10056	0.0979	0.10048	0.10051	0.10001	0.00118	1.18
No. 5	10.015	10.012	10.00	10.000	10.013	10.008	0.007	0.07
No. 6	100.04	100.052	100.0	100.0	100.04	100.03	0.02	0.02
No. 7	1000.7	999.17	996.	1000.	1005.	1000.2	3.2	0.32

b) Sample Standard Deviation (Per Cent)

Analog Circuit	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.
No. 1	1.02	0.60	1.09	1.05	0.36	1.63
No. 2	0.02	0.14	0.20	0.00	0.01	1.26
No. 3	0.02	0.02	0.16	0.00	0.01	1.18
No. 5	0.04	0.01	0.00	0.00	0.01	0.07
No. 6	0.03	0.12	0.00	0.00	0.02	0.02
No. 7	0.53	2.70	0.57	0.00	0.11	0.32

TABLE VIII - V/I (Ω) Corrected to 23°C

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s	s(%)
605603-3	0.01665	0.01671	0.016654	0.01650	0.016650	0.016633	0.000785	0.47
601333-2	0.16630	0.1658	0.165594	0.16515	0.16766	0.16610	0.000965	0.58
71983-2	191.85	193.32	194.924	195.825	194.0	193.984	1.5221	0.78
71166-2a	1701.3	1711.			1718.8	1710.4	8.77	0.51
71166-2b			2323.89	2123.63	1915.	2120.84	204.459	9.64
600200-2	0.01483	0.01490	0.01485	0.014819	0.014888	0.014857	0.000354	0.24
607075-2	0.21202	0.2115	0.211751	0.21207	0.21273	0.21201	0.00046	0.22
70877-3	15.452	15.54	15.6199	15.7433	15.556	15.582	0.1082	0.69
49445-2	23.538	23.72	23.7791	23.734	23.752	23.705	0.0957	0.40
66969-1	221.31	225.07	223.169	226.628	224.03	224.04	1.998	0.89
16603-2	1809.	1831.	1837.56	1862.62	1833.9	1834.82	19.121	1.04

With the use of the experience gained in the round-robin experiment and the limits of error specified in the measurement method, it is possible to estimate the contribution of error in each of these quantities to the overall measurement error.

The resistivity of a thin, homogenous semiconductor slice at a reference temperature T_o is given by[3]

$$\rho_o = \frac{V}{I} w F_2 F(w/\bar{S}) F_{sp} F_T$$

where the ratio of voltage V to current I is measured at a temperature T, w is the slice thickness, F_2 is a correction factor which accounts for finite slice diameter and which decreased from $\pi/\ln 2$ as the ratio $\bar{S}/D$ increases from 0, $F(w/\bar{S})$ is a correction factor which accounts for finite thickness and which decreases from 1.0 as the ratio $w/\bar{S}$ increases, F_{sp} is a correction factor which accounts for unequal probe separations, and F_T is a temperature correction factor. The assumption that the first three independent correction factors may be multiplied together to obtain the total geometrical correction factor is valid only when the deviations from the factors in their limiting cases ($D \to \infty$, $w \to 0$, and $S_1 \to S_2 \to S_3 \to \bar{S}$) are small. This is the reason that certain geometrical restrictions ($D \geq 10\bar{S}$, $w \leq \bar{S}$ and S_1, S_2, S_3 equal to 2 per cent) are employed in the method. Smits[4] has considered the factors F_2 and $F(w/\bar{S})$. More detailed tables of F_2 have appeared in the literature.[2,5] The factor F_{sp} is discussed in Appendix C. The temperature correction factor is discussed in Section 3 of this report. Tables or formulas for the four factors are given in the method (see Appendix F).

For small deviations from equilibrium values:

$$\frac{d\rho}{\rho} = \frac{dV}{V} - \frac{dI}{I} + \frac{dw}{w} + \frac{dF_2}{F_2} + \frac{dF(w/\bar{S})}{F(w/\bar{S})} + \frac{dF_{sp}}{F_{sp}} + \frac{dF_T}{F_T} \qquad (1)$$

Since $F_T = 1 - C_T(T - T_o) \approx 1$, one may write $(dF_T/F_T) = - C_T dT$. Then, with the use of (D-1) and (D-2) of Appendix D and (C-9) of Appendix C (1) becomes:

$$\frac{d\rho}{\rho} = \frac{dV}{V} - \frac{dI}{I} - C_T dT - (a - b - c)\frac{d\bar{S}}{\bar{S}} - c\,\frac{dS_2}{\bar{S}} + (1 - b)\frac{dw}{w} + a\,\frac{dD}{D},$$

where the coefficients a, b, and c are defined in Appendixes D and C. If all these factors were independent, the analysis could procede in a straightforward manner. Uncertainty in the measured values of $\bar{S}$, S_2, w, and D can be considered separately. However probe wander (resulting in changes in $\bar{S}$ and S_2) and uncertainty in temperature both affect the uncertainty in voltage.

2.2.2.1 Errors in V, I, and T. These three quantities are lumped together since it is necessary to account for the temperature variation of resistivity when voltage readings at different temperatures are compared. Three factors contribute to the error in the V/I ratio corrected to the reference temperature:

1) direct measurement error of V/I ratio
2) effect of probe wander on V, and
3) uncertainty of T.

The direct measurement error of the V/I ratio can be estimated from the measurement of the resistor in the analog test circuit. The relative standard deviation in this measurement is limited to 0.15 per cent. Under good conditions, it is considerably smaller than this limit as can be seen from Table VII.

Probe wander will affect the V/I ratio as discussed in Appendix C. Since the probe is raised and lowered between each of the ten independent readings of the ratio, the effect of probe wander on the uncertainty in the average value of the ratio is reduced to a negligible amount. Probe wander will be an important factor in the single reading procedure (cf. ¶2.2.1.3) but, as will be seen below, it is likely to be obscured by other effects.

Errors in temperature enter through uncertainties in the appropriate correction factor. If the maximum linear temperature coefficient is taken as 0.01 per deg uncertainties of temperature of ±0.2°C will be reflected as an ±0.2 per cent error in temperature correction factor. In many cases the temperature coefficient is smaller so this error will also be smaller. Uncertainties in linear temperature coefficient (C_T) of ±0.0001 are reflected as errors of about ±0.05 per cent at the extremes of the allowed temperature interval [(23±5)°C]. This error is independent of and much smaller than the error due to uncertainty in temperature so it can be neglected. With these assumptions and the assumption that the three sources of error are random and independent, $\delta V/V$ becomes:

$$\frac{\delta V}{V} = \sqrt{(0.15)^2 + (0.2)^2} = 0.25 \text{ per cent}$$

The data in Table VIII demonstrates that this small a deviation is seldom obtained even in those cases where the resistors in the analog test circuits were measured very accurately. The descrepancy probably arises from the inhomogeneity of the wafers. Note that the two wafers with the flattest resistivity profiles (Fig. 2) have the smallest V/I stardard deviations. The average observed value of $\delta V/V$ was 0.53 per cent if the 1000 Ω-cm slices are excluded.

2.2.2.2 Errors in D. These errors enter into the calculation of resistivity only through the correction factor F_2. The diameter of the wafer is required to be constant to $\pm D/5\bar{S}$ per cent of D. The average diameter was determined with an average relative standard deviation of less than 0.2 per cent as shown in Table IX. For the usual diameter of 25 mm ($16\bar{S}$ for the recommended probe separation) $\delta F_2/F$ is only 0.013 per cent. The maximum uncertainty in the proper diameter correction factor to be used can be estimated by considering inscribed and circumscribed circles. In this case $\Delta D/D = \pm D/5\bar{S}$ per cent so the maximum $\Delta F/F_2$ becomes ± 0.21 per cent for $D = 16\bar{S}$. Larger deviations will occur in smaller diameter wafers and smaller deviations in larger diameter wafers as discussed in Appendix D. The importance of using the diameter correction factor on wafers with $D \lesssim 25\bar{S}$ is also demonstrated in Appendix D.

2.2.2.3 Errors in w. These errors enter into the calculation of resistivity in two ways:

1) directly and
2) in the thickness correction factor $[F(w/\bar{S})]$.

The second of these, $\delta F/F$, is negative and has a value of $-0.27\ \delta w/w$ when $w = \bar{S}$ (the maximum thickness allowed by the method) and decreases in magnitude to zero as the thickness decreases. Some intermediate values are listed in Appendix D. The permitted deviation on w in the round-robin experiment was 0.16 per cent. From Table X it can be seen that this was not achieved which suggests that instruments with the required accuracy were not used. The value achieved was on the average about 0.3 per cent so that the total contribution to the error arising from this source is between 0.3 per cent (for thin wafers) and 0.22 per cent (for the thickest wafer permitted). The wafers used in the round-robin had a $w/\bar{S}$ ratio of about 0.75 so that the appropriate value for the deviation due to thickness measurement errors is $(1 - 0.12)\ (\delta w/s) = 0.26$ per cent.

2.2.2.4 Errors in S. There are two forms of this error. First, there is an uncertainty in the measured values of the probe separations which will depend on both probe wander on the polished test wafer and the error in measuring the position of the impressions. Second, there is the effect of probe wander on the measured V/I ratio. The first of these will enter into the resistivity calculation through the three correction factors F_2, $F(w/\bar{S})$, and F_{sp} as discussed in Appendixes C and D. For typical slices ($D = 16\bar{S}$, $w = 0.75\bar{S}$) the contribution from F_2 and $F(w/\bar{S})$ can be neglected so the appropriate value is $1.14(s/\bar{S}) = 0.34$ per cent. The effect of probe wander on the voltage measurement has been considered in ¶2.2.2.1 and Appendix C.

2.2.2.5 Summary. The total deviation may be found if it is assumed that each type of error discussed above is random and independent. With this assumption, the total deviation is the square root of the sum of the square of the individual deviations:

TABLE IX - Specimen Diameter Measurement (Centimeters)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s	s(%)
605603-3	2.832	2.832	2.834	2.832	2.835	2.833	0.0014	0.05
601333-2	2.908	2.895	2.902	2.891	2.888	2.897	0.0082	0.28
71983-2	3.343	3.360	3.3437	3.350	3.347	3.349	0.0069	0.21
71166-2a	2.047	2.051			2.06	2.053	0.0067	0.32
71166-2b			2.0713	2.070	2.071	2.071	0.0007	0.03
600200-2	3.099	3.096	3.104	3.099	3.103	3.100	0.0033	0.11
607075-2	2.972	2.969	2.9696	2.967	2.971	2.970	0.0019	0.06
70877-3	3.048	3.045	3.062	3.058	3.051	3.053	0.0070	0.23
49445-2	2.997	3.000	3.003	3.000	3.006	3.001	0.0034	0.11
66969-1	3.086	3.0911	3.10	3.086	3.094	3.091	0.0059	0.19
16603-2	2.337	2.344	2.349	2.347	2.343	2.344	0.0046	0.20

TABLE X - Specimen Thickness Measurement (Centimeters)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Avg.	s	s(%)
605603-3	0.1168	0.1173	0.1168	0.1171	0.11662	0.1169	0.0003	0.23
601333-2	0.1191	0.1197	0.1191	0.1194	0.11909	0.1193	0.0003	0.23
71983-2	0.1207	0.1209	0.1207	0.1206	0.12043	0.1207	0.0002	0.14
71166-2a	0.1166	0.11696			0.11581	0.1165	0.0006	0.50
71166-2b			0.1158	0.1166	0.11597	0.1161	0.0004	0.36
600200-2	0.1214	0.1218	0.1214	0.1219	0.12121	0.1215	0.0003	0.24
607075-2	0.1194	0.1203	0.1194	0.1196	0.11920	0.1196	0.0004	0.36
70877-3	0.1184	0.1181	0.1188	0.1184	0.11821	0.1184	0.0003	0.22
49445-2	0.1163	0.1163	0.1163	0.1145	0.11610	0.1159	0.0008	0.68
66969-1	0.1166	0.11709	0.1168	0.1167	0.11659	0.1168	0.0002	0.18
16603-2	0.1224	0.1227	0.1222	0.1227	0.12222	0.1224	0.0002	0.20

$$\frac{\delta\rho}{\rho} = \sqrt{(0.53)^2 + (0.013)^2 + (0.26)^2 + (0.34)^2}$$

$$= \sqrt{0.481} = 0.69 \text{ per cent.}$$

This value is remarkably close to the value frequently found in the round-robin experiment. It would appear to indicate that the errors in the experiment can be accounted for by the various factors above. Since the dominant error occurs in the V/I measurement, and since much of this can be attributed to wafer non-uniformity, more uniform slices must be available if increased precision is to be obtained.

2.3 EXPERIMENT 2.

This experiment was modeled after the preliminary experiment described in Appendix A. The procedure given in Appendix B was used with the addition of the table of currents described in connection with Experiment 1. In addition to the ten silicon wafers and six analog circuits the following equipment was furnished to each participant in turn: (1) four-point probe and holder, (2) micrometer stage with copper heat sink, mica insulator, and silicone heat-sink compound, (3) calibrated thermometer, and (4) polished silicon blanks for the probe separation measurement.

The analog circuits were similar to those used in Experiment 1. Resistivity profiles made on the wafers at the end of the test are shown in Fig. 3. In most cases each is similar to the profiles of the equivalent wafer used in Experiment 1.

2.3.1 Results. The results of the test are summarized in Table XI through XX. These tables are arranged in the same order as Tables I through X and present the data in a similar fashion. Much of the discussion related to Experiment 1 can be carried over to the present case. However, it is immediately obvious that the precision of the measurement is considerably less (i.e., has a higher numerical value) in Experiment 2. Examination of Tables XVII and XVIII shows that significant difficulties with the electrical measuring apparatus were encountered in several of the labs. Unfortunately, these difficulties render a quantitative analysis of the experiment meaningless.

It can be noted that geometrical measurements on the wafers (Tables XIX and XX) were made with nearly the precision attained in Experiment 1. Since the same probe was supplied to all participants, the data (Table XVI) yields an indication of the precision of the measurement of probe separation. Labs 1, 2, and 3 appeared to have problems in this area. The same probe was used in the preliminary experiment. Comparison of Table XVI with Tables III and IV of Appendix A shows that much of the spread in the present experiment is due to measurement problems rather than probe problems.

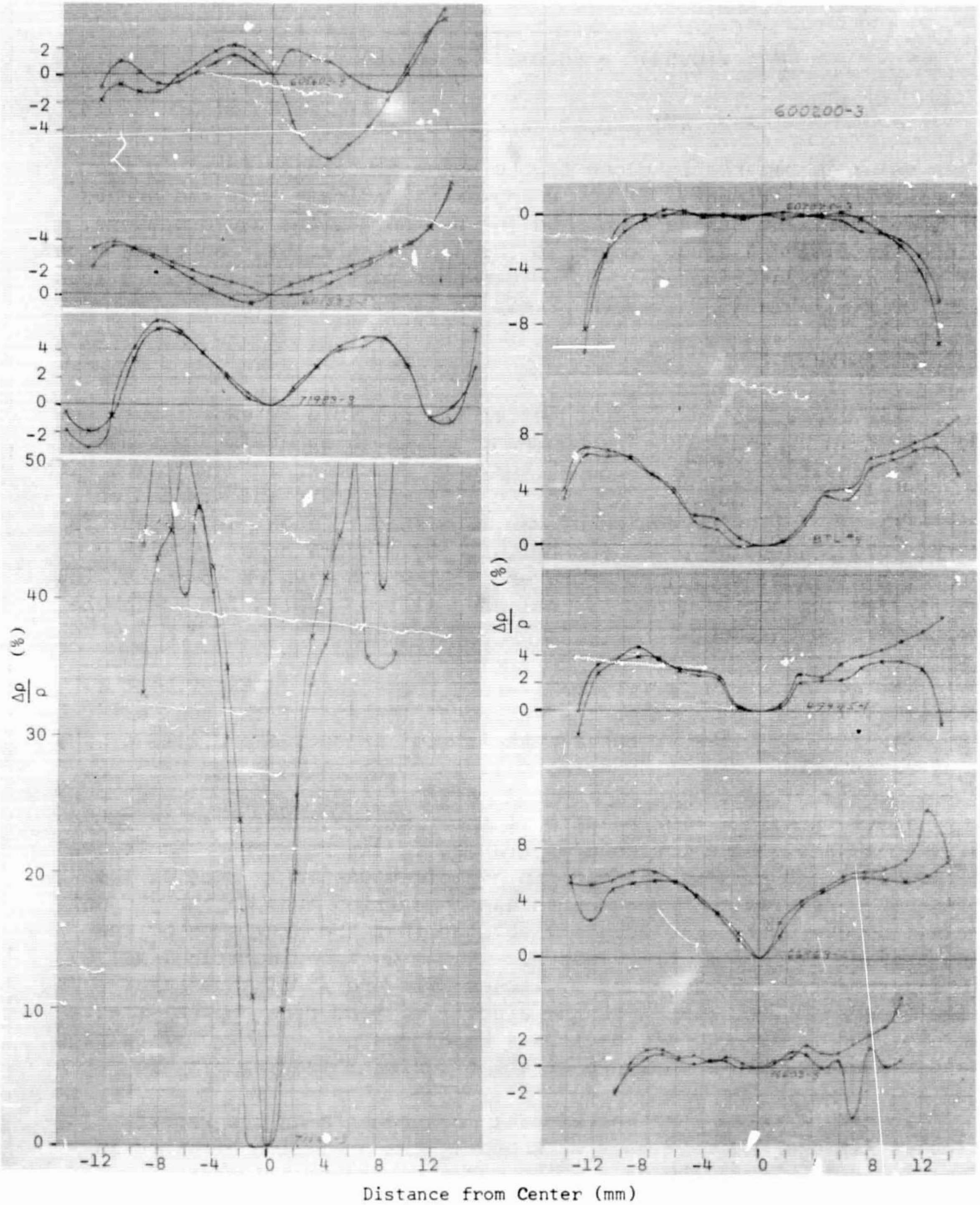


Figure 3. Specimen Resistivity Profiles for the Slices Used in Experiment 2. (No profile was made on Slice 600200-3.)

TABLE XI - Average Resistivity (Ω-cm) at 23°C (as Reported)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s	s(%)
605603-2	0.0079680	0.008	0.0082	0.00808	0.00821	0.0081977	0.008109	0.000108	1.34
601333-1	0.084034	0.086	0.08436	0.08449	0.0851	0.084731	0.08479	0.000694	0.82
71983-3	96.693	136.8	100.76	106.1	86.64	96.78	103.96	17.31	16.65
71166-3	1264.3	3373.		1067.9	704.6	1152.	1512.	1061.	70.16
600200-3	0.0077248	0.008					0.00786	0.00019	2.48
607075-3	0.10800	0.103	0.1074	0.1059	0.107	0.10688	0.1064	0.00179	1.68
BTL-4	10.187	10.0	10.220	10.50	9.68	10.134	10.12	0.271	2.68
49445-1	11.822	11.8	11.7959	12.10	11.83	11.800	11.86	0.119	1.01
66969-2	110.53	102.3	109.02	119.2	108.3	110.47	110.0	5.4	4.95
16603-3	939.42			914.1	735.07	936.9	881.4	98.2	11.14

TABLE XII - Average Resistivity (Ω-cm) at 23°C (Recomputed)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s	s(%)
605603-2	0.0079895	0.0080	0.0082	0.00823	0.00822	0.0081977	0.008140	0.000113	1.39
601333-1	0.084201	0.086	0.0849	0.08501	0.0850	0.084731	0.08497	0.000586	0.69
71983-3	97.112	137.	101.2	107.6	86.6	96.78	104.38	17.39	16.66
71166-3	1268.3	3360.		1075.	704.	1161.	1514.	1054.	69.62
600200-3	0.0077600	0.0078					0.00778	0.000028	0.36
607075-3	0.10745	0.103	0.1079	0.1067	0.1070	0.10688	0.1065	0.00176	1.66
BTL-4	10.188	10.0	10.23	10.55	9.70	10.134	10.13	0.280	2.76
49445-1	11.855	11.8	11.85	12.26	11.83	11.811	11.90	0.177	1.49
66969-2	110.84	102.	110.8	119.6	107.9	110.47	110.2	5.5	4.99
16603-3	940.80			923.4	734.	936.9	883.8	100.1	11.33

TABLE XIII- Sample Standard Deviation in Per Cent (Recomputed)

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.
605603-2	0.008140	0.25	0.6	0.39	1.19	0.73	0.55	1.39
601333-1	0.08497	0.31	0.6	0.08	0.34	0.42	0.13	0.69
71983-3	104.38	0.38	2.6	0.89	1.22	4.31	0.24	16.66
71166-3	1514.	1.64	14.0		3.30	4.44	1.23	69.62
600200-3	0.00778	0.15	0.0					.36
607075-3	0.1065	0.15	0.0	1.17	0.50	0.32	0.08	1.66
BTL-4	10.13	0.34	1.2	0.88	0.89	5.05	0.30	2.76
49445-1	11.90	0.15	0.7	0.28	0.24	0.21	0.10	1.49
66969-2	110.2	0.14	2.5	0.25	1.74	0.69	0.18	4.99
16603-3	883.8	0.31			1.66	0.71	0.14	11.33

TABLE XIV - Per Cent Difference of One Reading from Overall Average Resistivity

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s(%)
605603-2	0.008140	-1.65	-1.72	+0.74	+1.35	+0.98	+0.18	0.00	1.34
601333-1	0.08497	-0.50	+0.04	+0.04	+0.39	+0.04	-0.11	-0.02	0.29
71983-3	104.38	-6.96	+8.26	-3.81	+5.19	-25.66	-7.14	-5.02	12.60
71166-3	1514.	-16.49	+162.88		-29.66	-54.76	-23.18	+7.73	81.59
600200-3	0.00778	-0.40	+0.26					-0.13	0.46
607075-3	0.1065	+0.90	-3.29	+0.56	+0.66	+0.19	+0.38	-0.09	1.58
BTL-4	10.13	+0.69	+0.69	+1.28	+3.95	-0.99	+0.51	+0.99	1.61
49445-1	11.90	-0.35	-1.68	-0.42	+3.28	-0.76	-0.83	-0.17	1.74
66969-2	110.2	+0.67	-4.72	-8.53	+7.89	-2.27	+0.19	-1.09	5.64
16603-3	883.8	+6.40			+4.66	-16.27	+5.83	+0.16	10.96

TABLE XV - Per Cent Difference of Median of Three Readings from Overall Average Resistivity

Specimen	Average Resistivity (Ω-cm)	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s(%)
605603-2	0.008140	-1.84	-1.72	+0.74	+0.98	+1.84	+0.54	+0.12	1.52
601333-1	0.08497	-0.84	+1.80	+0.04	-0.20	+0.04	-0.31	+0.08	0.90
71983-3	104.38	-7.05	+27.42	-3.81	+3.56	-15.88	-7.30	-0.56	15.13
71166-3	1514.	-16.06	+132.50		-29.66	-54.76	-23.65	+1.65	73.33
600200-3	0.00778	-0.29	+0.26					0.00	0.39
607075-3	0.1065	+0.89	-3.29	+0.56	+0.47	+0.47	+0.39	-0.09	1.58
BTL-4	10.13	+0.35	-0.30	+1.97	+3.95	-0.99	+0.08	+0.89	1.80
49445-1	11.90	-0.35	-1.68	-0.42	+3.03	-0.59	-0.80	-0.17	1.62
66969-2	110.2	+0.67	-5.63	+0.54	+7.71	-2.27	+0.33	+0.18	4.39
16603-3	883.8	+6.80			+4.66	-16.72	+5.83	+0.14	11.26

TABLE XVI - Probe Separation Measurement in Millimeters

	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.
S_1	1.59156	1.5951	1.5888	1.58966	1.5908	1.58775	1.5905
s_1	0.01387	0.0020	0.00729	0.00122	0.0015	0.001090	0.00259
s_1(%)	0.87	0.13	0.46	0.08	0.10	0.07	0.16
S_2	1.58692	1.5916	1.58534	1.56671	1.5867	1.58796	1.5875
s_2	0.01651	0.0046	0.00658	0.001168	0.0008	0.001123	0.00213
s_2(%)	1.04	0.29	0.41	0.07	0.05	0.07	0.13
S_3	1.59720	1.5961	1.60528	1.60099	1.6063	1.60210	1.6012
s_3	0.01852	0.0056	0.00267	0.00086	0.0023	0.001199	0.00411
s_3(%)	1.16	0.35	0.17	0.05	0.14	0.07	0.26
$\bar{S}$	1.59189	1.5944	1.59314	1.59245	1.5946	1.59261	1.59319
F_{sp}	1.0034	1.0018	1.0053	1.0039	1.0053	1.0031	1.0038

TABLE XVII - Average Resistance (Recomputed)

a) Measured Values (Ω)

Analog Circuit	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s	s(%)
No. 1	0.0010032	0.00107	0.00100	0.001025		0.0009799	0.001016	0.000034	3.38
No. 2	0.010102	0.0102	0.01001	0.00996	0.01001	0.0101023	0.01006	0.00009	0.87
No. 3	0.10025	0.101	0.1003	0.1017	0.0995	0.10022	0.1005	0.00076	0.75
No. 5	10.015	10.3	10.00	10.155	9.96	10.017	10.07	0.129	1.28
No. 6	99.853	100.	100.0	106.2	93.8	99.94	99.97	3.92	3.92
No. 7	990.75	880.	1001.	963.0		1003.5	967.6	51.6	5.33

b) Sample Standard Deviation (Per Cent)

Analog Circuit	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.
No. 1	0.11	4.7	0.80	0.32		0.08	3.38
No. 2	0.02	0.3	0.28	0.55	0.22	0.01	0.87
No. 3	0.00	0.0	0.2	0.26	0.13	0.00	0.75
No. 5	0.01	0.8	0.0	0.19	0.28	0.00	1.28
No. 6	0.05	0.0	0.0	0.12	0.04	0.03	3.92
No. 7	0.11	0.0	0.33	0.48		0.05	5.33

TABLE XVIII - V/I (Ω) Corrected to 23°C

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s	s(%)
605503-2	0.015798	0.0159	0.01629	0.0164	0.0163	0.016306	0.01617	0.000251	1.55
601333-1	0.16365	0.1669	0.1652	0.166	0.166	0.16533	0.1655	0.00110	0.66
71983-3	185.30	261.1	192.7	206.6	165.	185.2	199.3	33.13	16.62
71166-3	2583.0	6842.0		2194.	1438.	2370.	3085.	2144.	69.48
600200-3	0.014799	0.015					0.014810	0.000142	0.95
607075-3	0.20849	0.201	0.2095	0.208	0.208	0.20821	0.2072	0.00309	1.49
BTL-4	21.990	21.8	22.078	23.01	21.0	22.020	21.98	0.643	2.92
49445-1	23.432	23.5	23.563	24.52	23.5	23.54	23.68	0.416	1.76
66969-2	217.72	201.0	217.0	234.5	212.	217.34	216.6	10.83	5.00
16603-3	1808.3			1782.	1411.	1805.	1702.	194.1	11.40

TABLE XIX - Specimen Diameter Measurement (Centimeters)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s	s(%)
605603-2	2.8313	2.81783		2.837	2.835	2.835	2.831	0.0078	0.27
601333-1	2.893	2.858		2.903	2.903	2.901	2.892	0.0192	0.67
71983-3	3.340	3.3338		3.338	3.340	3.331	3.337	0.0040	0.12
71166-3	2.065	2.064		2.068	2.064	2.068	2.066	0.0020	0.10
600200-3	3.0803	3.09562					3.088	0.0108	0.35
607075-3	2.9634	2.93687		2.967	2.965	2.965	2.959	0.0127	0.43
BTL-4	3.228	3.175		3.226	3.236	3.238	3.221	0.0260	0.81
49445-1	3.0015	3.0162		3.018	3.005	3.0038	3.009	0.0076	0.25
66909-2	3.081	3.01625		3.086	3.084	3.087	3.071	0.0306	1.00
16603-3	2.340			2.355	2.347	2.350	2.348	0.0063	0.27

TABLE XX - Specimen Thickness Measurement (Centimeters)

Specimen	Lab. 1	Lab. 2	Lab. 3	Lab. 4	Lab. 5	Lab. 6	Avg.	s	s(%)
605603-2	0.117057	0.1166		0.1158	0.1161	0.11625	0.11636	0.00048	0.42
601333-1	0.119168	0.1189		0.11836	0.1186	0.11865	0.11874	0.00031	0.26
71983-3	0.12082	0.1209		0.11989	0.1204	0.12051	0.12050	0.00040	0.33
71166-3	0.11618	0.1166		0.11582	0.1156	0.11589	0.11602	0.00039	0.33
600200-3	0.121376	0.1212					0.12129	0.00012	0.10
607075-3	0.119228	0.1191		0.11836	0.1184	0.11673	0.11876	0.00040	0.33
BTL-4	0.105537	0.1054		0.10439	0.1049	0.10480	0.10500	0.00047	0.44
49445-1	0.11668	0.1156		0.11506	0.1156	0.11561	0.11571	0.00059	0.51
66909-2	0.11734	0.1176		0.11684	0.1171	0.11716	0.11721	0.00028	0.24
16603-3	0.12257			0.12192	0.1222	0.12219	0.12222	0.00027	0.22

It should also be noted that some of the observed difficulties arose because of inadequate resolution in the measuring equipment. As a result, specific resolution requirements were added to the revision of the method.

2.4 CONCLUSIONS

From the results of these experiments, it can be concluded that resistivity measurements can be made according to the procedures of the method under test with a precision of ±2 per cent (3 standard deviations). Relaxation of the requirement of averaging 10 pairs of readings to permit a single pair to be used increases the 3 standard deviation interval to ±4 per cent. It was shown that errors in the six quantities measured during the test account for the overall deviation obtained if effects of wafer inhomogeneity are included. It was found that much of the error which entered in the determination of the potential difference between the inner probes arose from this source.

This precision was not achieved over the entire resistivity range. No very low (∿0.001 Ω-cm) resistivity wafers were included in the experiments. In addition gross inhomogeneity in the 1000 Ω-cm p-type wafers used in the tests prevented the acquisition of good data. Accordingly, additional tests at both extremes are still needed. The low resistivity test is scheduled to begin soon; in addition several high resistivity wafers will be included in this test.

The poor precision achieved in Experiment 2 serves to emphasize the need for adequate equipment and control procedures if precise measurements are desired. Although resistivity is probably the most widely measured semiconductor characteristic, the precise determination of resistivity can only be done with facilities which are well maintained, accurately calibrated, and properly used.

The control procedures outlined in the test method appear to be adequate to identify problems associated with the probe or electrical measuring equipment. Additional study of the low resistance analog circuits will be required before their usefulness can be fully documented. This will be done in connection with the forthcoming round-robin experiment.

The results of the analysis indicate that aside from the single measurement pair modification discussed above relaxation of the various requirements of the method will reduce the precision of the measurement to a value which is generally unacceptable. In particular if the permitted standard deviation were doubled the probe separation uncertainty would become the dominant factor contributing to the variation in measured resistivity. Furthermore, difficulties in thickness determination at the 1.1 mm level suggest that uncertainty in this deminsion will also become a dominant factor if slices 0.25 to 0.5 mm are measured. Where ±10 per cent measurements are sufficient, some relaxation in the geometrical requirements on the wafer would be feasible even if only a single pair of readings is taken. It is possible to incorporate the various geometrical correction factors into a direct reading instrument.[6] It is also possible to include circuits which incorporate the correction for temperature and unequal probe separations.

The method can be extended for use in sheet resistance measurements and for production control of slices. In order to determine the limits of validity and the precision which might be anticipated in such applications, additional studies of the effects of decreased probe pressure and different surface conditions must be carried out.

2.5 NOTES AND REFERENCES

1. This draft version of the method differs only slightly from the version published for information only in the back of Part 8 of the 1967 ASTM Book of Standards. A revision of this version is included as Appendix F.

2. M. A. Logan, "An AC Bridge for Semiconductor Resistivity Measurement Using a Four-Point Probe," Bell System Tech. J. 40, 885-919 (1961).

3. Note that this formula is appropriate to a thin slice while the formula shown in Fig. 1 is appropriate to a semi-infinite volume.

4. F. M. Smits, "Measurement of Sheet Resistivities with the Four-Point Probe," Bell System Tech. J. 37, 711-718 (1958).

5. L. J. Swartzendruber, "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin Circular Semiconductor Samples," NBS Technical Note 199, April 15, 1964.

6. L. J. Swartzendruber, F. H. Ulmer, and J. A. Coleman, "Direct-Reading Instrument for Silicon and Germanium Resistivity Measurement," (to be published).

3. EXPERIMENTAL STUDIES

3.1 INTRODUCTION

An experimental study of the temperature coefficient of resistivity of silicon and germanium was concluded during the project. A report describing this work has been prepared for publication. Its abstract is attached as Appendix E. In addition, initial experimental work was undertaken to establish thermal equilibration time, the effect of non-uniform thickness, and the effect of probe needle wobble.

3.2 THERMAL EQUILIBRATION TIME

Two sets of experiments were run. In the first, a 0.1 Ω-cm n-type germanium wafer cut into a "clover-leaf" shape for van der Pauw measurements was cooled below or heated above room temperature. After reaching a suitable temperature it was placed on the copper heat sink of the four-probe apparatus. The temperature of the wafer determined from its resistivity was monitored as a function of time. It was found that the wafer always approached a temperature somewhat greater than the heat sink temperature but that it was within 0.5°C of the heat sink temperature in less than 3 minutes when initially at -50°C and in less than 7 minutes when initially at +35°C.

These results emphasize the importance of the use of proper current levels when measuring resistivity. The current used (about 100 mA) was large enough to cause sufficient joule heating in the wafer to raise the temperature above the heat sink temperature. An auxiliary experiment, in which the wafer was not placed directly on the copper heat sink but instead, inside a plastic box at the heat sink temperature, showed that, in the absence of the heat sink, the wafer rises to a temperature nearly 9 deg above that of the heat sink in about 20 minutes. This current, which is larger than would normally be used on wafers of this resistivity, was selected in order to allow more rapid measurements to be made.

Even with the larger current, it was not possible to follow the initial stages of decay. Hence, a second series of measurements on a silicon wafer about 1.2 mm thick were made in which the temperature difference between the top of the wafer and the copper block was measured with a differential copper-constantan thermocouple. One junction of the thermocouple was attached to the wafer with gallium-indium eutectic; mechanical support was provided by gluing the wires just behind the junction to the wafer. The wafer was cooled or heated to the desired initial temperature. After the reference junction of the thermocouple was immersed in an oil-filled well in the copper heat sink and the leads were connected to a recorder with a maximum sensitivity of 1μV/mm, the wafer was placed on a 12 μm thick mica sheet on the heat sink and the probes were lowered. No current was passed through the probes. In all cases the wafer temperature had reached within 0.2 deg of the heat sink temperature in less than 30 s. Noise on the thermocouple leads prevented determination of smaller temperature differences. In an auxiliary experiment, the wafer was placed near but not on the heat sink; about 11 min. elapsed before the wafer reached within 0.3 deg of the heat sink.

3.3 EFFECT OF NON-UNIFORM THICKNESS

These experiments were carried out on an aluminum-doped silicon wafer of about 0.245 Ω-cm. The thickness was initially 1.022 mm and the diameter, 26.85 mm. After measuring the resistivity with parallel faces on the wafer, one side was angle lapped to 11 min., then 22 min., then 33 min., and finally parallel again. The average thickness in each case was determined from five measurements; one at the center of the wafer, and four on perpendicular radii about half way between the center and the edge of the wafer. The resistivity was determined by averaging the results of ten measurements at the center of the wafer, five on each side. Between readings, the wafer was rotated about 15°. Although a small increase in average resistivity was detected as the taper angle was increased, the value in each case did not depart from the average of the two parallel cases by more than 0.33%. The spread on the averages of the two parallel cases was about 0.2%. Although the dependence on taper angle may be statistically significant, it would appear that it may be ignored as a practical matter at least under conditions similar to those of this test. The results are summarized in Table XXI.

Table XXI

Angle lapped wafer: thickness and resistivity

	thickness	thickness variation (edge-to-edge)		thickness variation (measured)		resistivity
Condition	(mm)	(mm)	(%)	(mm)	(%)	(Ω-cm)
parallel	1.0223	-	-	0.024	0.23	0.24496 ± 0.00047
11 min.	0.9670	0.0860	8.9	0.0356	3.7	0.24534 ± 0.00066
22 min.	0.8899	0.1718	19.3	0.1103	12.4	0.24585 ± 0.00047
33 min.	0.8449	0.2577	30.4	0.1350	16.0	0.24626 ± 0.00069
parallel	0.6083	-	-	0.032	0.38	0.24594 ± 0.00054

Nevertheless, uncertainty in thickness in very thin wafers causes equal uncertainty in resistivity. When the wafer has flat (though non-parallel) faces the uncertainty in thickness can be reduced considerably below the variation in thickness over the wafer; however, this can not be assumed always to be the case. As an example of an irregular shape, a ring 2.674 mm wide and 0.130 mm deep was cut ultrasonically from the outer edge of the wafer, leaving a "top-hat" structure. If the full diameter and maximum thickness are used in the resistivity computation, a value about 1% larger than the average of the two parallel cases was obtained. A weighted value of average thickness yielded a resistivity about 4% lower, a 3% overcorrection. No convenient means of obtaining the effective diameter or thickness in this case has been found.

3.4 EFFECT OF PROBE NEEDLE WANDER

Studies of the effect of probe needle wander require the use of probes with different amounts of needle wander. Several probe assemblies were tested during this reporting period but none which had a sample standard deviation on probe spacing larger than that allowed in the test method (cf. Appendix F, ¶8.1.3.1) was found.

Computations of expected effects of probe needle wander on thin wafers were carried out as part of the error analysis of Experiment 1. The results of these computations have been summarized in Section 2.

4. REVISION OF TEST METHOD

4.1 INTRODUCTION

The fifth draft of the test method has been published as a proposed method ("for information only") in the back of Part 8 of the 1967 ASTM Book of Standards. The published version has been accepted as a Tentative Method by the ASTM and now has the designation F84-67T.

As a result of the round-robin experiments which have been discussed in Section 2 of this report, the method could be extended to cover a wider range of resistivity than earlier drafts. Procedures for measurements on slices between 0.0005 and 2000 Ω-cm are included although the precision could be established only over the narrower range between 0.005 and 200 Ω-cm. The revision is attached as Appendix F. This version of the method will be submitted to letter ballot, first Subcommittee VI and then to the full Committee F-1. After additional revision which may be necessary as a result of the balloting, the method will be submitted to the ASTM as a Revised Tentative in time for inclusion in the 1968 Book of Standards. The ASTM designation would then become F84-68T.

4.2 STYLE OF THE DRAFT

In order to emphasize the parts of the method which have been changed, these parts have been typed with a different type face than the unchanged parts. Locations where material has been deleted without the addition of other material are marked with three dots: "...". Some changes which are strictly editorial in nature such as changes in footnote numbers have not been designated with the special type face.

4.3 SCOPE OF THE REVISION

Despite the increased resistivity range covered, the method remains essentially a referee method. Specific procedures and conditions appropriate to non-referee measurements such as routine production and quality control have not been included except for a statement of the precision expected when only a single pair of readings is made instead of the series of ten specified in the method. However, use of the various procedures in the method would be expected to result in improved precision of non-referee measurements. In particular, the sections on probe and measuring circuit evaluation and on thermal sinking of the wafer would be very useful in standardizing measurement equipment and their general use on a regular basis should be encouraged.

4.4 DISCUSSION OF THE REVISIONS

The basis for each significant change in the method is discussed briefly below.

¶1.1 - The resistivity range is extended and precision data from round-robin experiment 1 is included.

¶4.2.1 - The 0.5 per cent (R3S%) requirement of the original version could not be met in the round-robin experiments.

¶4.3.4.2 - Closer spaced sets of indentations should make the probe separation test more convenient. (See also ¶8.1.1.1.)

¶4.3.4.3 - The smaller increment represents 0.1 per cent of the recommended probe separation and is necessary if the conditions of ¶8.1 are to be met.

¶4.4.1 - One of the round-robin participants found that electrical grounding of the heat sink reduced the scatter of some measurements.

Note 3 - This suggestion was made by one of the round-robin participants.

¶4.6.1.1, Table 1 - Current values proposed and adopted at the November 1967 meeting of the Resistivity Task Force at St. Louis. It should be noted that these are different from the currents used in the round-robin experiment. However, preliminary tests, reported at the St. Louis meeting, showed that the values in Table 1 should not introduce additional error. It was felt that the advantage of current in factors of 10 was significant. Comments on this point are invited. Considerable overlap of the ranges is allowed.

¶4.6.1.3, ¶4.6.2, Table 2 - Recommended resistance values are selected to be within a factor of about 3 of the V/I ratio for slices 1.0 to 1.2 mm thick.

¶4.6.3 - Renumbered as ¶4.8.

¶8.1.1.1, Note 5 - See comment for ¶4.3.4.2.

¶8.1.2.4, ¶8.1.2.5 - The formula for F_{sp} has been simplified; for convenience in making computations these paragraphs have been interchanged.

¶8.1.3.1 - The small relaxation in deviation appears from the round-robin data to be permissible.

¶8.2.1.2, ¶8.2.1.3 - These changes reflect the fact that appropriate analog circuits and currents must be used. They also close a loophole in the earlier draft so that now the current to be used in the analog circuit measurement is specified explicitly.

¶8.2 - Nomenclature change: the analog resistor is now identified as r rather than R.

¶8.2.3.1, ¶8.2.3.2 - These tighter specs were achieved in the round-robin experiment when suitable analog test resistors were employed.

Note 6 - A specific procedure for calibrating the analog test resistor is included for convenience.

¶8.2.3.3 - The results of round-robin experiment 2 indicate that an explicit resolution statement is necessary.

Note 7 - Results based on preliminary tests of thermal equilibration time are included for information.

¶9.4 - Changed to permit appropriate current to be used.

Note 8 - See comment for ¶8.2.3.3.

¶10.3, Table 3 - Changed to omit reference to slice radius which was not defined in the method. A diameter measurement is specified in ¶7.1.

¶12.1, ¶12.2 - These are revised precision statements which are based on the results of round-robin experiment 1.

Fig. 3 - Changes to reflect the fact that different values of r are required to cover the resistivity range and to show location for measuring potential difference when calibrating r.

Fig. 4 - Change to indicate that order of measurement is not significant.

Fig. 5 - Caption note changed to correct an error. If satisfactory photographs can be obtained, good, adequate, and poor indentation patterns will be included in the figure.

Fig. 10 - New temperature coefficient data over an extended range are included. Addition of reference 8 will enable user to find supporting data and comments as he desires.

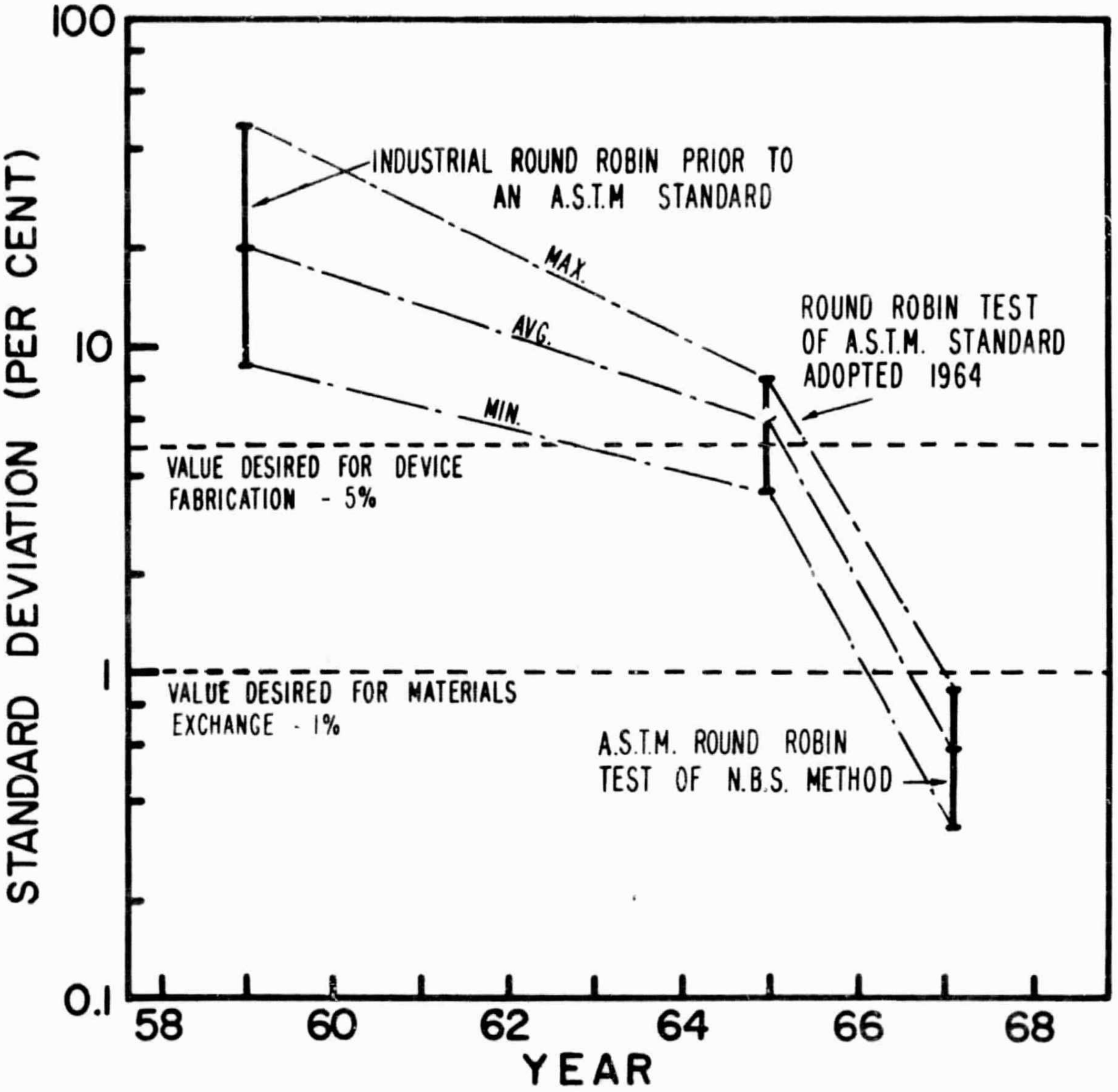


Figure 4. History of the Interlaboratory Precision of Measurement of Silicon Resistivity in the Range 0.01 to 100 Ω-cm, by the Four-Probe Method, Showing the Spread of Sample-Dependent Standard Deviations by the Vertical Bars.

5. SUMMARY

During the period covered by this project, the Method of Test for Resistivity of Silicon Slices Using Four Point Probes has been extended to cover the useful resistivity range. The precision which can now be obtained in the resistivity range between 0.005 and 120 ohm-cm is ±2 per cent (3 standard deviations). This is significantly better than was possible with earlier methods as shown by the plot of single standard deviation against time in Fig. 4.

Detailed analysis of the round-robin experiments leads to the following conclusions:

1) The desired precision can be obtained if correct procedures are followed and if the equipment used in the test meets the requirements of the test method. The importance of a well maintained, accurately calibrated, and properly used test system can not be overemphasized.
2) The major contribution to measurement error appears to be resistivity non-uniformity in the specimen under study.
3) If a single pair of readings is taken rather than the average of 10 pairs as required by the method, the precision is degraded somewhat; the relative standard deviation may double.
4) Relaxation of the geometrical requirements of the method would be expected to reduce the precision significantly. Some difficulty in maintaining the required precision in the determination of the thickness is expected if thin (0.25 to 0.5 mm) slices are measured.

The importance of knowing the temperature of the slice being measured and the effectiveness of the large copper heat sink in establishing this temperature were also demonstrated. Slices initially maintained at temperature well above or below the heat sink temperature reached a temperature within 0.2°C of the heat sink temperature less than 30 s after being placed on the mica insulator which electrically isolates the slice being measured from the heat sink.

Slices with flat, but non-parallel, sides could be measured precisely at the center if the average thickness was used. However, an appropriate correction could not be found for a slice with a "top-hat" configuration.

Difficulties with the low resistance analog circuits were attributed to instabilities in the circuits. New analog circuits of improved design are being assembled but they have not yet been tested to verify this conclusion. These tests are expected to be carried out soon in connection with an additional round-robin experiment designed to establish precision figures for very high and very low resistivity slices.

Determination of the precision to be expected from the method in non-referee applications such as routine production and quality control will require additional study of such factors as surface conditions, probe force, current levels, etc. Nevertheless, use of the various procedures of the method, in particular the sections on probe and measuring circuit evaluations and on thermal sinking of the wafer, would be expected to yield significantly improved precision in such applications. Use of these procedures on a regular and widespread basis should be encouraged.

APPENDIX A

Report to the ASTM F-1
Subcommittee VI Semiconductor
Resistivity Task Force on
a Special Round Robin

Introduction

This is a report on a special round robin on 4-point probe resistivity measurements held March 1 to June 1, 1966. The report was originally made to the Task Force in a preliminary form at the Chicago meeting in June 1966.

The idea for this round robin originated at a meeting of the Task Force in Dallas in February. Final plans were formulated at a later one-day meeting held at the National Bureau of Standards. The following laboratories participated (listed in alphabetical order):

Bell Telephone Labs., Allentown, Pennsylvania
Dow Corning Corp., Electronic Products Div., Hemlock, Michigan
International Business Machines Corp., East Fishkill Facility,
  Hopewell Junction, New York
Monsanto Chemical Co., Inorganic Chemicals Div., St. Louis, Mo.
National Bureau of Standards, Washington, D. C.

A special vote of thanks go to K. Benson and C. Paulnak at BTL for providing much of the material used and for making a special box to facilitate shipping. Thanks also go to all the participants for their expeditious handling of the measurements, allowing much useful data to be obtained and analyzed between two Sub-VI meetings!

Purpose

Before F43-64T[1] (Tentative Methods of Test for Resistivity of Semiconductor Materials) can be properly revised, the need exists to determine the contribution of each of the factors affecting the multilaboratory precision of resistivity measurements. The factors selected for investigation in this round robin were the precision of the probe spacing measurement and precision of electrical measuring equipment when doing four-point probe measurements at the 10 Ω-cm level. The measuring process was to be that typical of a good industrial standards laboratory. Future round robins including such factors as sample temperature measurement and sample preparation will be necessary.

Method

The procedure that was used for the round robin is given in Appendix B. As many of the variables as possible were controlled. The mechanical equipment required for the measurement was supplied along with silicon slices in the 10 Ω-cm range that had been prepared. Procedures for measuring the spacing of the probes in the furnished four-point probe and for determining the suitability of electrical equipment (supplied by each laboratory) were specified. Independent measurements of probe spacing, electrical equipment, slice diameter, and slice thickness in each lab provided a means of evaluating the reproducibility obtainable in such measurements. Since the thermal sink and thermometer were both supplied and since the sample surfaces were prepared at the beginning of the test, these conditions were not varied. A round robin "kit" encased in a sturdy wood box was shipped to each laboratory in turn. This kit contained:

(a) a four-point probe,
(b) a micrometer stage,
(c) a copper heat sink,
(d) a calibrated thermometer,
(e) mica for use as an insulator on the copper block,
(f) chemically polished silicon blanks for needle impressions,
(g) a four-point probe analog circuit,
(h) heat sink compound for making good thermal contact between the heat sink and specimen, and
(i) three lapped silicon slices on which to measure resistivity.

Results

First a word about notation. The abbreviation AVG will be used to denote the sample mean and the symbol s to denote the square root of the sample estimate of variance. (A capital S will be used to denote probe spacing.) Also, the idea of a confidence interval will be used.[2]

Let us first look at the resistivity results as taken directly from the data sheets which are shown in Table A-I. At first glance this is discouraging; even disregarding lab. 5 the most probable multi-laboratory precision is no better than ±2 per cent (R2S%).[3] Lab. 5 is obviously in error but this was probably due to difficulty in interpreting the instructions for use with direct reading equipment.

After finding out exactly how lab. 5 proceeded and recalculating their results, and also correcting obvious errors on the rest of the data sheets (e.g. misreading correction factors or errors like 49.9 mils = 0.1277 cm), the resistivities shown in Table A-II were obtained. If we disregard lab. 5 we can assert that: 1) under the conditions of this round robin, the most probable multi-lab precision for resistivity measurement is ±0.7 per cent (R2S%), and 2) the multi-laboratory precision for resistivity measurement using the methods of this round robin is better than ±2 per cent (R2S%) at a confidence level of 95 per cent. The latter statement means that if we were to repeat the round robin a very large number of times, there is only a 5 per cent

Table A-I - Average Resistivity (Ω-cm) at 23°C (as Reported)

Lab. No.	Sample BTL-2	Sample BTL-4	Sample KN-4
1	10.24	10.36	14.80
2	10.083	10.209	14.763
3	10.065	10.137	14.545
4	10.036	10.125	14.648
5	1.853	1.778	3.261
AVG	8.455	8.522	12.403
s	3.691 (44%)	3.77 (44%)	5.11 (41%)
AVG(a)	10.106	10.208	14.689
s(a)	0.091 (0.90%)	0.108 (1.1%)	0.116 (0.79%)
One-sided,(a) 95% confidence upper bound on σ	1.22 (2.2%)	0.26 (2.5%)	0.27 (1.9%)

Table A-II - Average Resistivity (Ω-cm) at 23°C (Recomputed)

Lab. No.	Sample BTL-2	Sample BTL-4	Sample KN-4
1	10.042	10.148	14.513
2	10.081	10.209	14.538
3	10.055	10.148	14.557
4	10.036	10.125	14.523
5	9.899	10.036	14.338
AVG	10.023	10.133	14.494
s	0.071 (0.71%)	0.063 (0.62%)	0.089 (0.61%)
AVG(a)	10.054	10.158	14.533
s(a)	0.020 (0.2%)	0.036 (0.36%)	0.019 (0.13%)
One-sided, 95% confidence upper bound on σ(a)	0.058 (0.6%)	0.105 (1.0%)	0.045 (0.3%)

(a) disregarding lab. 5

chance that the final precision obtained would be worse than ±2 per cent (R2S%).

Discussion

We wish to analyze and compare the relative magnitudes of the sources of error. The sources to be analyzed are:

(1) probe separation,
(2) V/I measurement,
(3) temperature measurement, and
(4) slice thickness measurement.

(1) Probe separation measurement. Probe separation was measured in each lab according to the specified procedure. The results, shown in Table A-III, show that this is an excellent procedure for measuring probe spacing, there being only a 5 per cent chance that the multi-lab precision is worse than ±0.5 per cent (R2S%). What part of this precision is due to needle-wobble, what part to needle-tip condition, and what part to the measuring apparatus is not certain.

Good probes can be selected by placing a maximum allowable single-lab s for the series of 10 determinations performed by each lab. The values for s obtained by the individual labs in this round robin, using a "good" probe, are given in Table A-IV. If we accept 0.16 per cent as the "true" standard deviation for the probe used, and if we want only a 5 per cent chance of rejecting a "good" probe (i.e. one at least as good as the one used in this round robin) we should require a single-lab s measurement of less than 0.26 per cent (see page 4-3 of Handbook 91).[2]

In a measurement of the resistivity of a slice a probe spacing error will show up in three places. The ratio[4] S_2/r, where r is the sample radius, is used to determine the correction factor for finite diameter, F_2. The ratio[4] w/S_2, where w is the slice thickness, is used to determine the correction factor, F(w/S). The individual values S_1, S_2, and S_3 are used to determine a correction for unequal probe spacing, $F_{sp} = 1 + 0.721(1 - S_2/2S_1 - S_2/2S_3)$.[5]

For the slice diameter and thickness used, an error of 0.1 per cent in measuring S would cause,

(1) an error of 0.01 per cent in F_2,
(2) an error of 0.02 per cent in F(w/S),
(3) an error of 0.08 per cent in F_{sp}.

Adding the effect of these errors directly (since they are not independent) gives a total error of 0.11 per cent.

Note the following about F_{sp}, a correction factor which has not been previously used. In this round robin the average value was 1.004. This is a 0.4 per cent correction and thus should not be neglected.

Table A-III - Probe Separation Measurement in Millimeters

Lab	S_1	S_2	S_3
1	1.60160	1.58572	1.58572
2	1.60396	1.58590	1.58882
3	1.60134	1.58354	1.58496
4	1.6027	1.5857	1.5862
5	1.6050	1.5880	1.5875
AVG	1.60294	1.58577	1.58664
s	0.00157 (0.10%)	0.00157 (0.10%)	0.00152 (0.10%)
One-sided, 95% confidence upper bound on σ	0.00373 (0.23%)	0.00373 (0.23%)	0.00361 (0.23%)

Table A-IV - Single Laboratory Relative Standard Deviation

Lab	S_1	S_2	S_3
1	0.10%	0.06%	0.12%
2	0.17	0.10	0.21
3	0.08	0.07	0.06
4	0.04	0.04	0.04
5	0.27	0.24	0.19
Overall AVG of table above	0.12		
Overall s of table above	0.08		
One sided, 95% confidence upper bound on AVG	0.16		

(2) V/I measurement. The justification for disregarding the lab. 5 results for V/I comes from measurement of the four-point probe analog circuit. The resistor in the black box was a 10Ω ± 0.5% resistor. The four other resistors were 3000Ω ± 5% resistors. The measured values are given in Table A-V. Note that lab. 5 measured a resistance about 3 per cent low and was also low on the slice resistivity measurement, (1.5 per cent, 1.2 per cent, and 1.2 per cent for the 3 slices, respectively). This could have been caused by using a measuring system with an input impedance a little too low. Note that a quantitative correction can not be determined from the analog circuit measurement, but that a wrong measurement indicates that an incorrect resistivity will probably be obtained.

The V/I values, in ohms corrected to 23°C, obtained by the first four labs are shown in Table A-VI. Again this is good agreement, with only a 5 per cent chance that the multi-lab precision is worse than ± 0.9 per cent (R2S%), the most probable value being ± 0.3 per cent (R2S%). One of the major aources of error here is probably specimen nonuniformity although an attempt was made to reduce this as much as possible by selecting uniform slices and by recentering after each of ten measurements in each lab, thus tending to average out the nonuniformity.

(3) Temperature measurement. This factor was largely eliminated as a source of error by sending around the same thermometer and same heat sink to every lab. Thus thermometer calibration and thermometer specimen heat path were uniform. Correction for temperature was quite important, however, corrections ranging as high as 3 per cent. A future round robin should help determine the effect of using different thermometers and heat sinks.

(4) Slice thickness measurement. The reported thicknesses are listed in Table A-VII. The thickness plays a double role in introducing error. For the sample thickness used an error of 0.11 per cent in w should cause,

(a) an error of 0.02 per cent due to the change of F(w/S), and
(b) an error of 0.11 per cent because w is a direct multiplier in the formula for calculating resistivity.

These two errors are in opposite directions; this gives a resultant error of about 0.09 per cent.

(5) Slice diameter measurement. The slices were not perfectly round so everyone did not measure the same diameter. The reported values, are listed in Table A-VIII. The last line shows the standard deviation in the correction factor, F_2, corresponding to the s in the diameter measurement. It is smaller because, at the diameter used, the correction factor is a slowly varying function of the diameter.

Table A-V - Average Resistance (Ω) (Recomputed)

Lab.	R
1	10.002
2	10.004
3	10.003
4	10.006
5	9.71

Table A-VI - V/I (Ω) Corrected to 23°C

Lab. No.	Sample BTL-2	Sample BTL-4	Sample KN-4
1	21.743	22.045	26.616
2	21.752	22.083	26.544
3	21.796	22.035	26.644
4	21.718	21.970	26.602
AVG	21.752	22.033	26.602
s	0.032	0.047	0.042
	(0.15%)	(0.21%)	(0.16%)
One sided, 95% confidence upper bound on σ	0.093 (0.44%)	0.137 (0.61%)	0.123 (0.47%)

Table A-VII - Slice Thickness (cm)

Lab. No.	Sample BTL-2	Sample BTL-4	Sample KN-4
1	0.1054	0.1049	0.1267
2	0.1054	0.1052	0.1270
3	0.10511	0.10481	0.12672
4	0.1052	0.1049	0.1267
5	0.1052	0.1049	0.1267
AVG	0.10522	0.10494	0.12676
s	0.00011 (0.10%)	0.00015 (0.14%)	0.00013 (0.10%)
One sided, 95% confidence upper bound on σ	0.00032 (0.29%)	0.00044 (0.41%)	0.00038 (0.29%)

Table A-VIII - Slice Diameter (cm)

Lab. No.	Sample BTL-2	Sample BTL-4	Sample KN-4
1	3.216	3.236	3.292
2	3.228	3.236	3.294
3	3.223	3.2322	3.297
4	3.236	3.238	3.302
5	3.221	3.241	3.299
AVG	3.2248	3.2367	3.2969
s	0.0076 (0.23%)	0.0033 (0.10%)	0.0041 (0.12%)
Resultant s in correction factor	(0.02%)	(0.01%)	(0.01%)
One sided, 95% confidence upper bound on σ in correction factor	(0.05%)	(0.02%)	(0.02%)

(6) Total. If each source of error is assumed to be random and independent, an estimate of the overall standard deviation may be found as the square root of the sum of the measured sample variances $[(\sum s^2)^{1/2}]$:

Probe spacing: $s = 0.11\%$
V/I measurement: $s = 0.15\%$
Thickness measurement: $s = 0.09\%$
Diameter measurement: $s = 0.02\%$

Total $s = 0.21\%$

Conclusions

The following conclusions can be drawn from the round robin results:

(1) We have a good method for the measurement of probe spacing.
(2) Using the black box analog circuit procedure is a good test of the electrical equipment being used to measure resistivity, at least for samples in the 10 Ω-cm range finished with a 5 micron lapping compound.
(3) For an "industrial standards lab" procedure at the 10 Ω-cm level, the contribution of the electrical measurement associated with a four-point probe method is on the order of ± 1/2 per cent (R2S%).
(4) The necessity for detailed, explicit instructions covering every important detail of the measurement can not be overemphasized. Neither can the need for detailed data sheets that show all the data taken, all the correction factors used, and all the computations made in arriving at the final values of the resistivity. When two laboratories are comparing resistivity measurements, these data sheets should be exchanged along with the samples.

Notes and References

1. ASTM Book of Standards, Part 8.
2. For a full explanation of the statistical terms and the method of computation used, see NBS Handbook 91, "Experimental Statistics", by M. G. Natrella, Chapters 1 through 4.
3. (R2S%) is the two-sigma precision index expressed in relative per cent, as defined in "Use of the Terms Precision and Accuracy as Applied to Measurement for a Property of a Material", ASTM Designation: E177 (see ASTM Book of Standards, Part 30).

4. There is an ambiguity in the method of Appendix B because it is not specified whether to use S_1, S_2, S_3 or the average in determining F_2 and $F(w/S)$. This is important since the difference in the spacings is larger than the precision in their measurement. This will affect the s in the final resistivity values, but not the s for each factor.

5. Note that F_{sp} applies to slice measurement <u>only</u>. A more convenient form of F_{sp} is: $F_{sp} = 1 + 1.082(1 - S_2/\bar{S})$, where $\bar{S}$ is the average of S_1, S_2, and S_3.

APPENDIX B

Special Round Robin Procedure

1. Measurement of Probe Spacing:

1.1 Measure the spacing of the probe provided (serial no. SI 62-1440C) using the method below. If any deviations in this technique are used, describe the deviations.

1.2 The technique used for measuring probe spacings is that of observing and measuring the probes indentations in a polished silicon surface.

1.3 Apparatus: The following apparatus is needed:

(1) A flat polished silicon surface. Some are provided but use your own if desired.

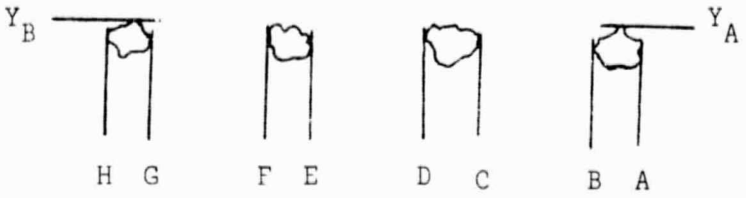
(2) A micrometer movement to move the probe or silicon in a direction perpendicular to a line through the probe points.

(3) A toolmaker's microscope for measuring distances between the indentations.

The silicon surface can be that of a slice or block which can be conveniently placed under the probe. The surface should be polished and reasonably flat. The micrometer movement for moving the probe or silicon surface should be capable of moving increments of 10 to 15 mils (0.25 to 0.375 mm) in a direction perpendicular to a line through the probe points. The toolmaker's microscope should be capable of measuring increments of 0.1% of the probe spacing S (0.06 mils (1.5μm) for a 62.5 mil (1.59 mm) probe spacing).

1.4 Procedure: With the four-point probe make a series of indentations on a polished silicon surface. These indentations are made by applying the probe to the surface using normal point pressures and measurement routine. The probes are then lifted and the silicon surface or probe is moved 10 to 15 mils (0.25 to 0.375 mm) in a direction perpendicular to a line through the probe points. Again the probe is applied to the silicon surface and the procedure repeated until a series of 30 indentation sets is obtained. The indentations obtained are often irregular in shape and may show several areas of contact for each probe. Place the silicon sample in the toolmaker's microscope. For 10 of the 30 indentation sets record the readings A through

H on the X axis of the toolmaker's microscope and the readings Y_A and Y_B on the Y axis for the locations shown in the figure below:



The angle of placement of the silicon sample on the microscope should be such that the Y axis readings do not differ more than 6 mils (0.125 mm). Record all readings on the data sheet provided. Calculate S_1, S_2, and S_3 using the data sheet provided and the formulas:

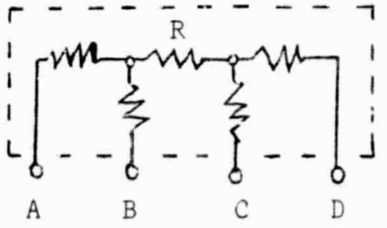
$$S_1 = \frac{C + D}{2} - \frac{A + B}{2}$$

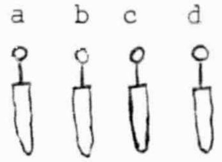
$$S_2 = \frac{E + F}{2} - \frac{C + D}{2}$$

$$S_3 = \frac{G + H}{2} - \frac{E + F}{2}$$

2. Measurements of the analog circuit.

2.1 In the apparatus to be used to measure sample resistivity connect the four leads of the analog circuit. Leads A, B, C, and D correspond to the four leads of a four-point probe a, b, c, and d as shown below:





with A and D being the current leads and B and C being the leads between which the voltage is measured.

2.2 At a current level of approximately one milliampere measure the current and voltage first in one direction (the "forward" direction) and then with current reversed (the "reverse" direction). Record these values on the data sheet provided. If the instrument being used measures V/I directly, record this instead of currents and voltages. Repeat for ten determinations. For each determination calculate V/I for forward and reverse direction and the averages and record on the data sheet provided.

PROBE SPACING DATA

RUN NUMBER	A	B	C	D	E	F	G	H	Y_A	Y_B
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

PROBE SPACING CALCULATION

RUN NUMBER	$\frac{A+B}{2}$	$\frac{C+D}{2}$	$\frac{E+F}{2}$	$\frac{G+H}{2}$	S_1	S_2	S_3
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
				Average:			

LABORATORY: ____________________

DATE: ____________________

PROBE SERIAL No.: ____________________

$$S_1 = \frac{C+D}{2} - \frac{A+B}{2}$$

$$S_2 = \frac{E+F}{2} - \frac{C+D}{2}$$

$$S_3 = \frac{G+H}{2} - \frac{E+F}{2}$$

ANALOG CIRCUIT MEASUREMENT DATA

RUN NUMBER	forward current I_f ma	forward voltage V_f mv	reverse current I_r ma	reverse voltage V_r mv		for direct reading equip. V/I_f ohms	V/I_r ohms
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

CALCULATIONS

RUN NUMBER	$\frac{V_f}{I_f}$ ohms	$\frac{V}{I_r}$ ohms	average of each run
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
		Average:	

3. Sample Measurement

3.1 Measure each of the three samples by the method that follows.

3.2 Use the heat sink and micrometer stage provided. Make sure each sample is electrically isolated from the heat sink by measuring the resistance between sample and heat sink with an ohmmeter. Electrical isolation is accomplished with a mica layer (provided with heat sink). Measure the sample temperature by placing one of the thermometers provided in the hole in the heat sink. Two thermometers are provided. Please use thermometer NBS 63 (tagged with an H) unless it has been broken during the round robin, in which case use thermometer NBS 64 (tagged with an L). Note on the data sheet which thermometer was used.

3.3 Before measuring each sample, clean ultrasonically in warm water and detergent, then rinse with flowing deionized water. Then ultrasonically degrease in acetone, rinse with alcohol and air dry.

3.4 Center the four point probe within 0.010" (0.25 mm) of the center of the sample being measured.

3.5 Using the probe provided make ten determinations of current, voltage, and temperature. Remove, replace, and recenter the sample between each determination. Record the following data in the data sheet provided:

(a) T, the temperature of the sample as measured by the thermometer placed in the heat sink.

(b) I_f, the current through the two outer probes.

(c) V_f, the voltage across the two inner probes with current in the direction of I_f.

(d) I_r, the current through the two outer probes when the current direction is reversed.

(e) V_r, the voltage across the two inner probes with the current in the direction of I_r.

(f) For direct reading equipment only record V/I in both forward and reverse directions instead of I_f, V_f, I_r, and V_r.

3.6 Carry out the calculations on the data sheet. Obtain C_T from Figure B-1, F_2 by linear interpolation of Table B-I and F(w/S) (where w is the slice thickness) by linear interpolation of Table B-II. Use the data on slice thickness and diameter provided.

LABORATORY:

DATE:

SAMPLE No.:

Thickness = __________

Diameter = __________

Thermometer used: ______

DATA

RUN NUMBER	I_f ma	V_f mv	I_r ma	V_r mv	T °C		for direct reading equipment T °C	V/I_f ohm	V/I_r ohm
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

CALCULATIONS

RUN NUMBER	$\frac{V_f}{I_f}$	$\frac{V_r}{I_r}$	Avg	F_T	ρ ohm-cm
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
				Average:	

C_T = __________

$F_T = 1 - C_T(T - 23)$

$F_{sp} =$

$1 + 0.721(1 - S_2/2S_1 - S_2/2S_3)$

= __________

F_2 = __________

$F(w/s)$ = __________

$F = F_2 \times w \times F(w/s) \times F_{sp}$

= __________

$\rho = \text{Avg} \times F_T \times F$

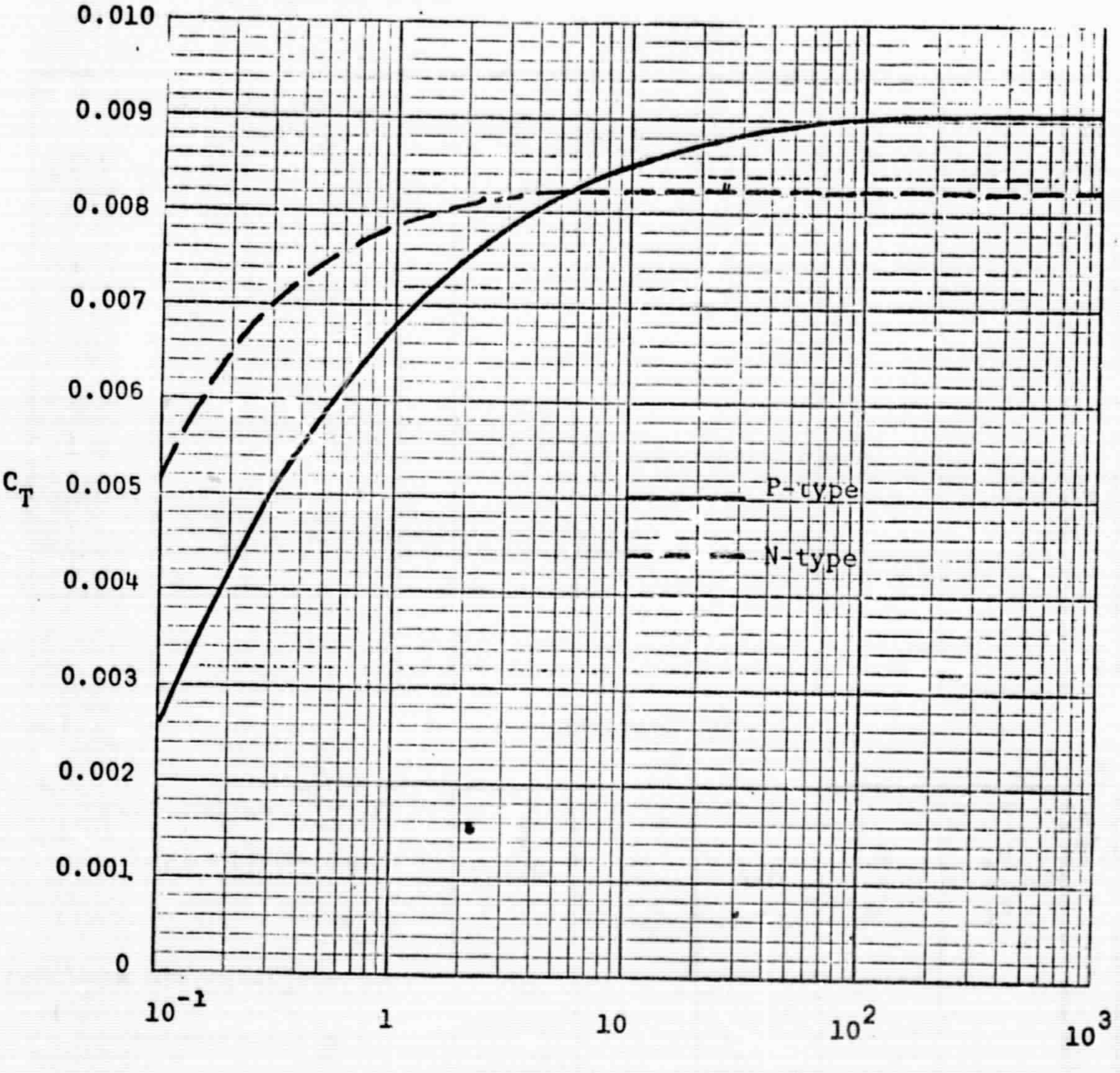

0.010
0.009
0.008
0.007
0.006
C_T
0.005
0.004
0.003
0.002
0.001
0
10^{-1}
1
10
10^2
10^3
P-type
N-type
ρ, Ω-cm, calculated using F_T = 1

Figure B-1.

Table B-I

s/r	F_2	s/r	F_2	s/r	F_2
0	4.532	0.07	4.485	0.14	4.348
0.01	4.531	0.08	4.470	0.15	4.322
0.02	4.528	0.09	4.454	0.16	4.294
0.03	4.524	0.10	4.436	0.17	4.265
0.04	4.517	0.11	4.417	0.18	4.235
0.05	4.508	0.12	4.395	0.19	4.204
0.06	4.497	0.13	4.372	0.20	4.171

Table B-II

w/s	F(w/s)
0.5	0.997
0.6	0.992
0.7	0.982
0.8	0.966
0.9	0.944
1.0	0.921

APPENDIX C

Probe Separation Correction Factor for Thin Slices

The resistivity of large, thin sheets of homogeneous material when measured with an in-line four-point probe is given by:

$$\rho = \frac{V}{I}\frac{\pi}{\ln 2} w F_{sp}, \qquad \text{(C-1)}$$

where V is the potential difference between the inner probes, I is the current through the outer probes, w is the slice thickness, and

$$F_{sp} = \frac{2 \ln 2}{\ln\left[\frac{(S_1 + S_2)(S_2 + S_3)}{S_1 S_3}\right]} \qquad \text{(C-2)}$$

is the factor which corrects for unequal separations S_1, S_2, and S_3 between adjacent probes. If the separations differ from their mean value, S, by no more than a few per cent the expression can be simplified to:[1]

$$F_{sp} = 1 + \frac{1}{\ln 4}[1 - \frac{S_2}{2S_3} - \frac{S_2}{2S_1}] = 1 + 0.721[1 - \frac{S_2}{2S_3} - \frac{S_2}{2S_1}], \qquad \text{(C-3)}$$

where S_2 is the separation between the inner probes. This expression was the one used in the Proposed Method. With the same assumptions it is also possible to transform (C-2) into an equivalent form which involves only S_2 and $\bar{S}$:

$$F_{sp} = 1 + \frac{3}{2 \ln 4}(1 - \frac{S_2}{\bar{S}}) = 1 + 1.082(1 - \frac{S_2}{\bar{S}}). \qquad \text{(C-4)}$$

If the slice thickness w exceeds $\bar{S}/2$ or if the slice diameter is less than $50\bar{S}$ additional correction factors must be used with (C-1). The effect of unequal probe separation on these factors has not been investigated. If the probe separations do not differ from their means by more than a few per cent (as required in the derivations above) it is thought that significant error will not be introduced into the result by multiplying the appropriate factors together for the $w/\bar{S}$ and $\bar{S}/D$ ratios allowed by the method. Correction factors appropriate to semi-infinite volumes have been discussed by Valdes[2] and Hargreaves and Millard.[3]

The magnitude of F_{sp} will be affected by uncertainty in the measured values of the probe separations. This effect can be determined from the differential:

$$dF_{sp} = -\frac{3}{2\bar{S}^2 \ln 4} (\bar{S}dS_2 - S_2 d\bar{S}), \qquad \text{(C-5)}$$

If the measured positions of the four probes differ from their correct values by small amounts dx_1, dx_2, dx_3, dx_4 (as a result either of probe wander or error in measurement of an impression) then $dS_2 = dx_3 - dx_2$, $d\bar{S} = (dx_4 - dx_1)/3$ and (C-5) becomes:

$$dF_{sp} = -\frac{1}{2\bar{S}^2 \ln 4} (S_2 dx_1 - 3\bar{S}dx_2 + 3\bar{S}dx_3 - S_2 dx_4). \qquad \text{(C-6)}$$

If it is assumed that the probe displacements are random and independent each with standard deviation δx and that $S_2 \approx \bar{S}$, then $F_{sp} \approx 1$ and the relative standard deviation in F_{sp} becomes:

$$\frac{\delta F_{sp}}{F_{sp}} = \frac{\sqrt{5}}{\ln 4} \frac{\delta x}{\bar{S}}. \qquad \text{(C-7)}$$

One measures the standard deviation, s, of the probe separation rather than the standard deviation of the probe displacement. These are related by $s = \sqrt{2}\delta x$ and (C-7) becomes:

$$\frac{\delta F_{sp}}{F_{sp}} = \frac{\sqrt{10}}{2 \ln 4} \frac{s}{\bar{S}} = 1.14 \frac{s}{\bar{S}}. \qquad \text{(C-8)}$$

It should be noted that the standard deviation of the mean probe separation is $\delta\bar{S} = s/3$ because only the first and last probe displacements enter into the calculation. If the variation of $\bar{S}$ and S_2 are considered to be independent, the following form of (C-5) is convenient in the error analysis of ¶2.2.2:

$$\frac{dF_{sp}}{F_{sp}} = -c \frac{dS_2}{\bar{S}} + c \frac{d\bar{S}}{\bar{S}}, \qquad \text{(C-9)}$$

where $c = 3/2 \ln 4 = 1.082$. In this form, the second term may be combined directly with terms in $d\bar{S}/\bar{S}$ from Appendix D. (C-8) follows directly since $\sqrt{10}/2 \ln 4 = 3\sqrt{1 + (1/9)}/2 \ln 4$.

Probe wander will also influence the measured voltage. If all other factors are held constant:

$$\frac{dV}{V} = (a - b - c) \frac{d\bar{S}}{\bar{S}} + c \frac{dS_2}{\bar{S}} \qquad \text{(C-10)}$$

where a and b are coefficients associated with the factors F_2 and $F(w/\bar{S})$ discussed in Appendix D. For typical slices ($D = 16\bar{S}$, $w = 0.75\bar{S}$) $a = 0.07$ and $b = 0.12$. Therefore (C-10) becomes:

$$\frac{dV}{V} = 1.13 \frac{d\bar{S}}{\bar{S}} + 1.08 \frac{dS_2}{\bar{S}} \qquad \text{(C-11)}$$

and the relative standard deviation in the voltage reading is:

$$\frac{\delta V}{V} = \sqrt{\frac{(1.13)^2}{9} + (1.08)^2}\ \frac{\delta S_2}{\bar{S}} \approx 1.14 \frac{\delta S_2}{\bar{S}}. \qquad \text{(C-12)}$$

It can be seen that the principal contribution to the deviation in V comes from F_{sp}. Although (C-8) and (C-12) are similar in form, the former refers to the uncertainty in measured probe separation which is characterized by s while the latter refers to probe wander which is characterized by δS_2. In general one would expect that $\delta S_2 < s$. If data from a series of measurements are averaged to obtain the value of voltage, the contribution to the uncertainty which arises from probe wander is reduced significantly. For a single reading, it could be as much as 0.34 per cent for the conditions of the method, but in general would be expected to be significantly less than this.

REFERENCES

1. L. J. Swartzendruber, unpublished work.
2. L. B. Valdes, "Resistivity Measurements on Germanium for Transistors," Proc. IRE 42, 420-427 (1954).
3. J. K. Hargreaves and D. Millard, "The Accuracy of Four-Probe Resistivity Measurements on Silicon," Brit. J. Appl. Phys. 13, 231-234 (1962).

APPENDIX D

Errors Introduced Through Diameter and Thickness Correction Factors

The correction factors F_2 and $F(w/\bar{S})$ are tabulated in Tables 1 and 2 of the Special Round Robin Procedure (see Appendix B). These factors approach constant values as $\bar{S}/D$ and $w/\bar{S}$, respectively, become small. Hence the error introduced into the calculation for resistivity will be a function of these ratios.

Consider first the factor F_2. The diameter, D, is permitted to go from $10\bar{S}$ to infinity. The effect of error in the determination of D or $\bar{S}$ on F_2 is given by

$$\frac{dF_2}{F_2} = a\,\frac{dD}{D} = -a\,\frac{d\bar{S}}{\bar{S}}. \qquad (D-1)$$

The coefficient, a, varies from 0.154 at $D = 10\bar{S}$ to 0.0 at $D = \infty$. The following table lists several quantities of interest:

D	F_2	a	$\delta F_2/F_2(\%)\big\|_{\delta D}$	$\Delta F_2/F_2(\%)\big\|_{\Delta D}$	$\delta F_2/F_2(\%)\big\|_{\delta \bar{S}}$	E(%)
$10\bar{S}$	4.171	0.154	0.031	0.31	-0.015	+8.7
$16\bar{S}$	4.383	0.066	0.013	0.21	-0.006	+3.4
$25\bar{S}$	4.470	0.028	0.006	0.14	-0.003	+1.4
$100\bar{S}$	4.528	0.0015	0.0003	0.03	-0.0001	+0.1

The first three columns list the diameter, the correction factor, and the coefficient respectively. The fourth column lists the relative deviation due to deviations in the measurement of average diameter obtained in the round-robin experiment (0.2 per cent). The fifth column lists the maximum uncertainty in the correction factor due to permitted eccentricity of the wafer; the diameter is required to be constant to within $D/5\bar{S}$ per cent. The sixth column lists the relative sample standard deviation due to deviation in the measurement of average probe separation (0.1 per cent). The seventh column lists the error in computed resistivity if the diameter correction factor is not used and clearly demonstrates the importance of using it if $D \lesssim 25\bar{S}$. It should be noted that this correction factor is appropriate only for measurements taken at the center of the wafer. Additional factors are required if measurements are made elsewhere on the wafer. These have been incorporated into an extended table of F_2.[1]

The factor $F(w/\bar{S})$ is discussed in detail by Smits.^{2} The thickness, w, is permitted in the present method to vary from $\bar{S}$ to as small a value as can be accurately controlled and measured. The effect of error in the determination of w or $\bar{S}$ on $F(w/\bar{S})$ is given by:

$$\frac{dF}{F} = -b\,\frac{dw}{w} = b\,\frac{d\bar{S}}{\bar{S}} \qquad \text{(D-2)}$$

The coefficient, b, varies from 0.27 for $w = \bar{S}$ to 0.018 for $w = \bar{S}/2$. The following table lists several quantities of interest:

w	$F(w/\bar{S})$	b	$\delta F/F(\%)\big\|_{\delta w}$	$\delta F/F(\%)\big\|_{\delta \bar{S}}$
$\bar{S}$	0.921	0.27	-0.081	0.027
$0.75\bar{S}$	0.974	0.12	-0.036	0.012
$0.65\bar{S}$	0.987	0.066	-0.0020	0.0007
$0.5\bar{S}$	0.997	0.018	-0.00054	0.0002

The first three columns list the thickness, the correction factor and the coefficient. The fourth column lists the relative deviation due to deviations in the measurement of thickness obtained in the round-robin experiment (0.3 per cent). The fifth column lists the column deviation due to deviations in the measurement of average probe separation (0.1 per cent).

REFERENCES

1. L. J. Swartzendruber, "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin Circular Semiconductor Samples," NBS Technical Note 199, April 15, 1964.
2. F. M. Smits, "Measurement of Sheet Resistivities with the Four-Point Probe," Bell System Tech. J. 37, 711-718 (1958).

APPENDIX E

Temperature Coefficient of Resistivity of Silicon and Germanium Near Room Temperature

W. M. Bullis, F. H. Brewer, C. D. Kolstad and L. J. Swartzendruber

National Bureau of Standards
Washington, D. C. 20234

ABSTRACT

Temperature coefficients for the resistivity of n- and p-type germanium and silicon in the neighborhood of room temperature have been determined over a wide range of resistivity. Linear temperature coefficients have been found for the extrinsic exhaustion region ($<5\ \Omega\cdot\text{cm}$ for germanium and $<5000\ \Omega\cdot\text{cm}$ for silicon). The results are presented as plots of temperature coefficient against resistivity at 23°C. The plots may be used in connection with measurements of resistivity on extrinsic germanium and silicon doped with the usual shallow impurities such as boron, aluminum, gallium, phosphorus, arsenic, and antimony. Accurate linear coefficients cannot be found for specimens doped with deep-lying impurities in sufficient amounts to affect the carrier density nor for specimens with resistivity in the transition region between extrinsic and intrinsic conduction.

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APPENDIX F

Tentative Method of Test for Resistivity of Silicon Slices Using Four Point Probes

1. Scope

1.1 The resistivity of a silicon crystal is an important materials acceptance requirement. This method describes a procedure which will enable interlaboratory comparisons of the resistivity of silicon slices with room temperature (23 C) resistivity between 0.005 and 120 ohm·cm to be made with a precision of ±2 per cent (R3S%) as defined in ASTM Recommended Practice E177, for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material.[2]

1.2 The method is intended for use on single crystals of silicon in the form of circular slices with a diameter greater than 16 mm (0.625 in.) and a thickness less than 1.6 mm (0.0625 in.). Both n- and p-type slices can be evaluated. Geometrical correction factors required for these measurements are available in tabulated form.[3]

1.3 This method is to be used as a referee method for determining the resistivity of single crystal silicon slices in preference to ASTM Methods F 43, Test for Resistivity of Semiconductor Materials.[4]

Note 1 - The method is also applicable to silicon of higher or lower resistivity if appropriate changes in measurement conditions are made. Round-robin measurements to establish correct measurement conditions and expected precision are now being carried out.

Note 2 - The method is also applicable to other semiconductor materials but neither the appropriate conditions of measurement nor the expected precision have been experimentally determined. Other geometries for which correction factors are not available can also be measured by this method but only comparative measurements using similar geometrical conditions should be made in such situations.

2. Summary of Method

2.1 A collinear four-probe array is used in determining the resistivity in this method. A direct current is passed through the specimen between the outer probes and the resulting potential difference is measured between the inner probes. The resistivity is calculated for the measured current and potential values using factors appropriate to the geometry.

2.2 This method includes procedures for checking both the probe assembly and the electrical measuring apparatus.

2.2.1 The spacing between the four probe tips is determined from measurements of indentations made by the tips in a polished silicon surface. This test also is used to determine the condition of the tips.

2.2.2 The accuracy of the electrical measuring equipment is tested by means of an analog circuit containing a known resistance standard together with other resistors which simulate the resistance at the contacts between the probe tips and the semiconductor surface.

2.3 Procedures for preparing the specimen, for measuring its size, and for determining the temperature of the specimen during the measurement are also given. Abbreviated tables of correction factors appropriate to circular slice geometry and ... plots of temperature coefficient versus resistivity are included with the method so that appropriate calculations can be made conveniently.

3. Definition

3.1 Resistivity - The resistivity of a semiconductor for the purposes of this method is the volume resistivity, which is defined as the ratio of the potential gradient parallel to the current in the material to the current density.

4. Apparatus

4.1 Slice Preparation:

4.1.1 Lapping facilities which permit the lapping of a slice so that the thickness varies by no more than ±1 per cent from its value at the center.

4.1.2 An ultrasonic cleaner of suitable frequency (18 to 45 kHz) and adequate power.

4.1.3 Chemical laboratory apparatus such as plastic beakers, graduates, and plastic-coated tweezers suitable for use both with acids (including hydrofluoric) and with solvents. Adequate facilities for handling and disposing of acids and their vapors are essential.

4.2 Measurement of Specimen Geometry:

4.2.1 Thickness - Calibrated mechanical or electronic thickness gage capable of measuring the slice thickness to ±1.0 per cent (R3S%) at various positions on the slice.

4.2.2 Diameter - A micrometer or vernier caliper.

4.3 Probe Assembly:

4.3.1 Probes - The probes shall have conical tungsten carbide tips with included angle of 45 to 150 deg. The nominal radius of a probe tip should be initially 25 to 50 μm.

4.3.2 Probe Force - The force on each probe shall be 1.75 ± 0.25 N when the probes are against the specimen in measurement position.

4.3.3 Insulation - The electrical isolation between a probe (with its associated spring and external lead) and any other probe or part of the probe assembly shall be at least 10^8 ohms.

4.3.4 Probe Alignment and Separation - The four probe tips shall be in an equally spaced linear array. The probe spacing (separation between adjacent probe tips) shall have a nominal value of 1.6 mm. Probe spacing shall be determined according to the procedure of 8.1 in order to establish the suitability of the probe assembly as defined in 8.1.3. The following apparatus is required for this determination:

4.3.4.1 A silicon surface such as that of a slice or block which can be conveniently placed under the probe assembly. The surface must be polished and have a flatness characteristic of semiconductor wafers used in transistor fabrication.

4.3.4.2 A micrometer movement capable of moving the probe assembly or silicon surface in increments of 0.05 to 0.10 mm in a direction perpendicular to a line through the probe tips and parallel to the plane of the surface.

4.3.4.3 A toolmaker's microscope capable of measuring increments of 1.5 μm.

4.4 Specimen and Probe Supports:

4.4.1 Specimen Support - A copper block at least 100 mm (4 in.) in diameter and at least 38 mm (1.5 in.) thick, or a rectangular block of equivalent mass and thickness shall be used to support the specimen and provide a heat sink. It shall contain a hole which will accommodate a thermometer (see 4.5) in such a manner that the center of the bulk of the thermometer is not more than 10 mm below the central area of the heat sink where the specimen will be placed. A layer of mica 12 to 25 μm thick is placed on top of the heat sink to provide electrical isolation between the specimen and heat sink (see Fig. 1). Mineral oil or silicone heat sink compound is used between the mica layer and copper block to reduce the thermal resistance. The heat sink shall be arranged so that the center of the probe tip array can be placed within 0.25 mm of the center of the specimen. (See Note 3.) The heat sink shall be connected to the ground point of the electrical measuring apparatus. (See 4.6.)

Note 3 - Shallow rings, concentric with the center of the copper block, may be machined into the heat sink in order to assist in rapid centering of slices.

4.4.2 Probe Support - The probe support shall allow the probes to be lowered onto the surface of the specimen with negligible lateral movement of the probe tips. (See 8.1.3.4.)

4.5 <u>Thermometer</u> - ASTM Precision Thermometer having a range from -8 to 32 C and conforming to the requirements for thermometer 63C as prescribed in ASTM Specifications E 1, for ASTM Thermometers.[5] The thermometer hole should be filled with mineral oil or silicone heat sink compound to provide good thermal contact between heat sink and thermometer.

4.6 <u>Electrical Measuring Apparatus</u>:

4.6.1 Any circuit that meets the requirements of 8.2 may be used to make the electrical measurements. The recommended circuit, connected as shown in Fig. 2, consists of the following:

4.6.1.1 <u>Constant Current Source</u> - The value of current to be used depends on the specimen resistivity. ... Currents between 10^{-1} and 10^{-5} amp are required if the resistivity range between 0.0005 and 5000 ohm·cm is to be covered. (See Table 1.)

4.6.1.2 <u>Current Reversing Switch</u>.

4.6.1.3 <u>Standard Resistor</u> - The resistance of the standard resistor shall be selected so that it is within a factor of 100 of that of the specimen to be measured. Recommended values of resistance for various resistivity ranges are listed in Table 2.

4.6.1.4 <u>Double-Throw, Double-Pole Potential Selector Switch</u>.

4.6.1.5 <u>Potentiometer-Galvanometer or Electronic Voltmeter</u> - The instrument may be used to read the potential drop in volts or it may be calibrated in conjunction with the current source to read the volt-current ratio directly. The instrument must be capable of measuring potential differences between 10^{-4} and 1 v.

4.6.2 <u>Analog Test Circuit</u> - Five resistors connected as shown in Fig. 3 shall be used in testing the electrical measuring apparatus according to the procedure given in 8.2. The resistance of the central resistor, <u>r</u>, shall be selected according to the resistivity of the specimen to be measured as listed in Table 2.

...

4.7 <u>Conductivity-Type Determination</u> - Apparatus in accordance with Method A of ASTM Methods F 42, Test for Conductivity Type of Semiconductors.[4]

4.8 <u>Ohmmeter</u> capable of indicating a leakage path of 10^8 ohms.

5. Reagents and Materials

5.1 Purity of Reagents - Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Purity of Water - Reference to water shall be understood to mean either distilled water or deionized water having a resistivity greater than 2 megohm·cm at 25 C as determined by the Non-Referee Method of ASTM Methods D 1125, Test for Electrical Conductivity of Industrial Water and Industrial Waste Water.[7]

5.3 The recommended chemicals shall have the following nominal assays:

Hydrofluoric acid, per cent	49.0 ± 0.25
Nitric acid, per cent	70.5 ± 0.5

5.4 Etching Solution 15:1 - Mix 90 ml of nitric acid (HNO_3) and 6 ml of hydrofluoric acid (HF).

5.5 Acetone $(CH_3)_2CO$.

5.6 Methanol (CH_3OH).

5.7 Lapping Abrasive - Aluminum oxide commercially specified as 5 μm grade.

5.8 Detergent Solution - An aqueous, nonionic surfactant solution.

5.9 Mineral Oil or Silicone Heat Sink Compound.

6. Safety Precautions

6.1 The acids used in this method are extremely hazardous. All precautions normally used with these chemicals should be strictly observed. See Appendix A1 for safety precautions for handling hydrofluoric acid.

7. Preparation of Test Specimen

7.1 The specimen shall be circular. The average specimen diameter (D) shall be greater than ten times the average probe spacing ($\bar{S}$). The diameter shall be constant to $\pm(D/5\bar{S})$ per cent of D as determined by measurements of the diameter made at 15 deg. intervals. **Record the value of D.**

7.2 After sawing, at least 0.1 mm shall be taken from each side to remove saw damage. This may be done conveniently by etching before lapping using the etching solution listed in 5.4.

Note 4 - Rotating the specimen during etching helps provide a more uniform etch.

7.3 Finish lapping shall be carried out using 5 μm aluminum oxide abrasive. The finished surface shall have a matte rather than a polished nature. The finished thickness (w) shall be less than the average probe spacing ($\bar{S}$). Thickness shall be determined at nine locations on the specimen (see Fig. 4). It shall not vary more than ±1 per cent from the value at the center. Record the value of w at the center of the specimen.

7.4 After lapping, the specimen shall be cleaned ultrasonically in warm water and detergent, rinsed with flowing deionized water, ultrasonically degreased in acetone, rinsed with methanol, and air dried. The specimen should be cushioned with paper or placed in a pliable plastic beaker during ultrasonic agitation in order to reduce the risk of breakage.

8. Suitability of Test Equipment

8.1 Probe Assembly - The probe spacing and tip condition shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

8.1.1 Procedure:

8.1.1.1 Make a series of indentations on a polished silicon surface with the four-point probe. Make these indentations by applying the probe to the surface using normal point pressures. Lift the probes and move either the silicon surface or the probes 0.05 to 0.10 mm in a direction perpendicular to a line through the probe tips. Again apply the probes to the silicon surface. Repeat the procedure until a series of ten indentation sets is obtained.

Note 5 - It is recommended that the surface or the probes be moved twice the usual distance after every other or every third indentation set in order to assist the operator in identifying the indentations belonging to each set.

8.1.1.2 Ultrasonically degrease the specimen in acetone, rinse with methanol, and let dry. (See 7.4.)

8.1.1.3 Place the polished silicon specimen on the stage of the toolmaker's microscope so that the Y axis readings (Y_A and Y_B in Fig. 5a) do not differ by more than 0.150 mm (0.006 in.). For each of the ten indentation sets record the readings A through H (defined in Fig. 5a) on the X axis of the toolmaker's microscope and the readings Y_A and Y_B on the Y axis. Use a data sheet similar to that shown in Fig. 6.

8.1.2 Calculations:

8.1.2.1 For each of the ten sets of measurements calculate the probe separations S_{1j}, S_{2j}, and S_{3j} from the equation:

$$S_{1j} = \frac{C_j + D_j}{2} - \frac{A_j + B_j}{2},$$

$$S_{2j} = \frac{E_j + F_j}{2} - \frac{C_j + D_j}{2}, \text{ and} \qquad (1)$$

$$S_{3j} = \frac{G_j + H_j}{2} - \frac{E_j + F_j}{2}.$$

In Eq 1, the index j is the run number and has a value between 1 and 10.

8.1.2.2 Calculate the average value for each of the three separations using the S_{ij} calculated above and the equation:

$$\bar{S}_i = \frac{1}{10} \sum_{j=1}^{10} S_{ij}. \qquad (2)$$

8.1.2.3 Calculate the sample standard deviation s_i for each of the three separations using the $\bar{S}_i$ calculated from Eq 2, the S_{ij} calculated from Eq 1, and the equation:

$$s_i = \frac{1}{3} \left[\sum_{j=1}^{10} (S_{ij} - \bar{S}_i)^2 \right]^{1/2}. \qquad (3)$$

8.1.2.4 Calculate the average probe spacing $\bar{S}$:

$$\bar{S} = \frac{1}{3} (\bar{S}_1 + \bar{S}_2 + \bar{S}_3). \qquad (4)$$

8.1.2.5 calculate the probe spacing correction factor F_{sp}:

$$F_{sp} = 1 + 1.082[1 - (\bar{S}_2/\bar{S})]. \qquad (5)$$

8.1.3 Requirements - For the probe to be acceptable, it must meet the following requirements:

8.1.3.1 Each of the three sets of ten measurements for S_i shall have a sample standard deviation s_i of less than 0.30 per cent of S_i.

8.1.3.2 The average values of the separations ($\bar{S}_1$, $\bar{S}_2$, and $\bar{S}_3$) shall not differ by more than 2 per cent.

8.1.3.3 The indentations obtained should show only a single area of contact for each probe (see Fig. 5b). If the indentations obtained show disconnected areas of contact for one or more of the probes, the probe or probes should be replaced and the test rerun.

8.2 Electrical Equipment - The suitability and accuracy of the electrical equipment shall be established in the following manner. It is recommended that this be done immediately prior to a referee measurement.

8.2.1 Procedure:

8.2.1.1 Disconnect the electrical leads from the four-point probe.

8.2.1.2 Attach the current leads (1 and 2 of Fig. 2) to the current terminals (I) of the analog circuit appropriate to the resistivity of the specimen to be measured (see Fig. 3 and Table 2). Attach the potential leads (3 and 4 of Fig. 2) to the potential terminals (V) of the analog circuit.

8.2.1.3 With the current ... in one direction (forward) adjust its magnitude to the appropriate value as given in Table 1. Measure the potential drop across the standard resistor (V_{sf}). Record this value on a data sheet such as the one shown in Fig. 7. Measure and record the potential drop across the resistor in the analog circuit (V_{af}). Reverse the direction of the current. Measure and record the potential drop across the standard resistor (V_{sr}). Measure and record the potential drop across the resistor in the analog circuit (V_{ar}).

8.2.1.4 Repeat the procedure of 8.1.2.3 four more times.

8.2.1.5 If another means of measuring the current is being used, read the current directly rather than measuring the potential drop across the standard resistor. If equipment which reads resistance (voltage-current ratio) directly is being used, measure the resistance ten times. Reverse the direction of the current after each reading, and record the data using only the last two columns of the data sheet (see Fig. 7).

8.2.2 Calculations:

8.2.2.1 If the current is measured according to 8.2.1.3 calculate the resistance from each pair of voltage readings (V_s and V_a) using the following relation:

$$r = V_a R_s / V_s , \qquad (6)$$

where:

r = resistance in ohms,
V_s = potential difference across the standard resistor in millivolts,
V_a = potential difference across the analog resistor in millivolts, and
R_s = resistance of standard resistor in ohms.

If the current is measured directly, instead of Eq 6 use the relation:

$$r = V_a / I , \qquad (7)$$

where:

I = the current in milliamps.

If the resistance is measured directly begin the calculations with 8.2.2.2.

8.2.2.2 Calculate the average resistance $\bar{r}$ from the equation:

$$\bar{r} = \frac{1}{10} \sum_{j=1}^{10} r_i , \qquad (8)$$

where:

r_i = one of the ten values of resistance determined above.

8.2.2.3 Calculate the sample standard deviation s from the equation:

$$s = \frac{1}{3} \left[\sum_{i=1}^{10} (r_i - \bar{r})^2 \right]^{1/2} . \qquad (9)$$

8.2.3 Requirements - For the electrical measuring equipment to be suitable, it must meet the following requirements.

8.2.3.1 The value of $\bar{r}$ must be within 0.15 per cent of the known value of r.

Note 6 - The value of the test resistor, r, may be determined with the use of ordinary standards laboratory procedures if the potential difference V' is measured. (See Fig. 3.)

8.2.3.3 The resolution of the equipment must be such that differences in resistance of 0.05 per cent can be detected.

9. Procedure

9.1 Immediately before measuring the specimen, clean ultrasonically in warm water and detergent solution, rinse in flowing deionized water, ultrasonically degrease in acetone, rinse with methanol, and air dry (see 7.4).

9.2 Using clean tweezers place the specimen on the mica insulator on top of the heat sink. Measure the resistance between specimen and heat sink with an ohmmeter in order to verify that the specimen is electrically isolated ($>10^8$ ohms) from the heat sink. With the thermometer in place, allow sufficient time after placing the specimen on the heat sink for thermal equilibrium to be established.

Note 7 - For specimens which have been in the same room as the heat sink for 30 min. or more, the time required for equilibration will not exceed 30 sec. The heat sink itself should have been allowed to come to equilibrium with the room (the temperature of which should not vary by more than a few degrees) for 48 hrs. before referee measurements are made.

9.3 Lower the probes onto the surface of the specimen so that the center of the probe tip array is within 0.25 mm of the center of the specimen.

9.4 With the current in the forward direction adjust its magnitude to the appropriate value as given in Table 1, measure the following, and record the data on a data sheet such as the one in Fig. 8a:

9.4.1 T, the temperature in deg C of the sample as measured by the thermometer placed in the heat sink.

9.4.2 V_{sf}, The potential drop in millivolts across the standard resistor. (Substitute I_f, the current, if using another means of measuring the current; omit this measurement if using direct reading equipment.)

9.4.3 V_f, the potential drop in millivolts between the two inner probes. (Substitute R_f, the resistance between the two inner probes, if using direct reading equipment, and use the data sheet of Fig. 8b.)

Note 8 - To obtain the precision stated in 12.1 the temperature must be measured to the nearest 0.1 C and the potential drops with a resolution of ±0.1 per cent.

9.5 Reverse the direction of the current. Measure the following and record the data:

9.5.1 V_{sr}, the potential drop in millivolts across the standard resistor. (Substitute I_r, the current, if using another means of measuring the current; omit this measurement if using direct reading equipment.)

9.5.2 V_r, the potential drop in millivolts between the two inner probes. (Substitute R_r, the resistance between the two inner probes, if using direct reading equipment.)

9.6 Raise the probe and rotate the specimen about 15 deg.

9.7 Repeat the procedure of 9.3, 9.4, 9.5, and 9.6 until ten sets of data have been taken.

9.8 Record on the data sheet the specimen thickness in centimeters as measured at its center (see 7.3) and the average specimen diameter in centimeters (see 7.1).

9.9 Determine the conductivity type of the specimen according to Method A of Methods F 42. Follow the procedure as given with the exception that the surface treatment of this method (see 7.3) shall be used.

9.10 Precautions - In making resistivity measurements, spurious results can arise from a number of sources.

9.10.1 Photoconductive and photovoltaic effects can seriously influence the observed resistivity, particularly with nearly intrinsic material. Therefore, all determinations should be made in a dark chamber unless experience shows that the material is insensitive to ambient illumination.

9.10.2 Spurious currents can be introduced in the testing circuit when the equipment is located near high frequency generators. If equipment is located near such sources, adequate shielding must be provided.

9.10.3 Minority carrier injection during the measurement can occur due to the electric field in the specimen. With material possessing high lifetime of the minority carriers and high resistivity, such injection can result in a lowering of the resistivity for a distance of several centimeters. Carrier injection can be detected by repeating the measurements at lower current. In the absence of injection no increase in resistivity should be observed. The current level recommended should reduce the probability of difficulty from this source to a minimum but in cases of doubt the measurements of 9.4 and 9.5 should be repeated at a lower current. If the proper current is being used, doubling or halving its magnitude should cause a change in observed resistance which is less than 0.5 per cent.

9.10.4 Semiconductors have a significant temperature coefficient of resistivity. Consequently, the current used should be small to avoid resistive heating. If resistive heating is suspected it can be detected by a change in readings as a function of time starting immediately after the current is applied.

9.10.5 Vibration of the probe sometimes causes troublesome changes in contact resistance. If difficulty is encountered, the apparatus should be shock mounted.

10. Calculations

10.1 Calculate the resistance for the current in both forward and reverse directions:

$$R_f = V_f R_s / V_{sf} = V_f / I_f, \text{ and}$$

$$R_r = V_r R_s / V_{sr} = V_r / I_r \qquad (10)$$

where:

R_f = resistance in ohms with current in the forward direction,
I_f = forward current in milliamperes,
R_r = resistance in ohms with current in the reverse direction,
I_r = reverse current in milliamperes,
R_s = resistance of standard resistor in ohms, and
V_f, V_r, V_{sf}, and V_{sr} are defined in 9.4 and 9.5.

The second form of Eq 10 is most convenient for use when the current is measured directly. This calculation is not required if direct reading equipment is employed. In all cases, R_f and R_r must agree to within 10 per cent of the larger for the measurement to be accepted for referee purposes. These and subsequent calculations may be summarized conveniently in the data sheet of Fig. 9.

10.2 Calculate the average value of resistance (R_{avg}) for each run:

$$R_{avg} = \frac{1}{2}(R_f + R_r) \qquad (11)$$

10.3 Calculate the ratio of the average probe separation ($\bar{S}$) (see 8.1.2.5) to the slice diameter (D). Find the correction factor F_2 from Table 3 using linear interpolation.

10.4 Calculate the ratio of the slice thickness (w) to the average probe separation ($\bar{S}$). Find the correction factor $F(w/\bar{S})$ from Table 4 using linear interpolation.

10.5 Calculate the geometrical correction factor F:

$$F = F_2 \times w \times F(w/\bar{S}) \times F_{sp} \qquad (12)$$

where:

F_{sp} = probe spacing correction factor (see 8.1.2.5) and
w = specimen thickness in cm.

10.6 Calculate the resistivity of the sample at the temperature of measurement:

$$\rho_T = R_{avg} \times F \qquad (13)$$

where:

ρ_T = resistivity in ohm·cm of specimen at temperature T,
R_{avg} = average resistance in ohms (see 10.2), and
F = geometrical correction factor in cm (see 10.5).

10.7 Find the appropriate temperature coefficient[8] from Fig. 10. Calculate the temperature correction factor F_T:

$$F_T = 1 - C_T(T - 23) \qquad (14)$$

where:

T = temperature in deg. C, and
C_T = coefficient read from Fig. 10.

10.8 Calculate the resistivity corrected to 23 C:

$$\rho_{23} = \rho_T \times F_T \qquad (15)$$

where:

ρ_{23} = resistivity in ohm·cm corrected to 23 C.

10.9 Calculate the value of the grand average of the corrected resistivity:

$$\rho_{23}(\text{Average}) = \frac{1}{10} \sum_{i=1}^{10} \rho_{23}(i) \qquad (16)$$

where $\rho_{23}(i)$ are corrected resistivities found from Eq 15.

11. Report

11.1 For referee tests the report shall include all information called for on data sheets (Figs. 6, 7, 8, and 9).

12. Precision

12.1 For silicon slices with room temperature (23 C) resistivity between 0.005 and 120 ohm·cm the interlaboratory precision is ±2 per cent (R3S%) when the measurement is performed by experienced operators. This precision was achieved during two round-robin experiments involving five laboratories and 11 slices. The precision for silicon slices outside this resistivity range is now being determined by additional round-robin experiments.

12.2 In addition, the effect which relaxation of certain of the requirements of the method has on the precision is also under investigation. Preliminary results suggest that if only a single pair of measurements is made instead of the series of ten specified in 9.7, the interlaboratory precision is ±4 per cent (R3S%) when the measurement is performed by experienced operators.

APPENDIX A1

A1.1 Several reagents required for these methods contain hydrofluoric acid (HF). This acid can cause painful and dangerous burns which sometimes leave bad scars.

A1.2 Wear eye protection and acidproof gloves at all times when handling HF. Instruct all immediate personnel in first acid measures for HF burns.

A1.3 If HF comes in contact with the body the affected areas should be immediately washed thoroughly in water for at least 15 min. If this procedure is applied within a few seconds of the time the HF comes in contact with the skin further treatment is rarely required. If, however, pain is noted after 1 hr. the patient should see a doctor for injection of calcium gluconate at the doctor's discretion.

A1.4 In cases where the affected areas are eyes, lips, under fingernails, or other soft tissues the patient should be taken to a doctor immediately after the affected area has been washed.

Footnotes

[1] Under the standardization procedure of the Society, this specification is under the jurisdiction of the ASTM Committee F-1 on Materials for Electron Devices and Microelectronics. A list of members may be found in the ASTM Year Book.

Accepted Aug. 25, 1967.

[2] 1967 Book of ASTM Standards, Part 30.

[3] Smits, F. M., "Measurement of Sheet Resistivities with the Four-Point Probe," Bell System Technical Journal, Vol. 37, 1948, p. 711; Swartzendruber, L. J., "Correction Factor Tables for Four-Point Probe Resistivity Measurements on Thin, Circular Semiconductor Samples," Technical Note 199, National Bureau of Standards, April 15, 1964.

[4] 1968 Book of ASTM Standards, Part 8.

[5] 1968 Book of ASTM Standards, Part 18.

[6] "Reagent Chemicals, American Chemical Society Specification," Am. Chemical Soc., Washington, D. C.

[7] 1968 Book of ASTM Standards, Part 23.

[8] Bullis, W. M., Brewer, F. H., Kolstad, C. D., and Swartzendruber, L. J., "Temperature Coefficient of Resistivity of Silicon and Germanium Near Room Temperature," to be published.

Table 1 - Current Values Required for Measurements of Resistivity

Resistivity (ohm·cm)	Current (mA)[a]
<0.012	100
0.008 to 0.6	10
0.4 to 60	1
40 to 1200	0.1
>800	0.01

[a] Value must be within ±20 per cent of the nominal value listed and must be stable to within ±0.05 per cent during the time of the measurement.

Table 2 - Resistivity Range Appropriate to Analog Test Circuit Resistance and Recommended Standard Resistance Values

Resistance (ohm)[a]	Resistivity (ohm·cm)
0.0010	<0.002
0.010	0.0015 to 0.02
0.10	0.015 to 0.2
1.0	0.15 to 2.0
10.	1.5 to 20.
100.	15. to 200.
1000.	>150.

[a] Value must be within ±20 per cent of the nominal value listed and must be known to ±0.05 per cent.

Table 3 –

Correction factor F_2 as a function of the ratio of probe separation ($\overline{S}$) to slice diameter (D).

$\overline{S}/D$	F_2	$\overline{S}/D$	F_2	$\overline{S}/D$	F_2
0	4.532	0.035	4.485	0.070	4.348
0.005	4.531	0.040	4.470	0.075	4.322
0.010	4.528	0.045	4.454	0.080	4.294
0.015	4.524	0.050	4.436	0.085	4.265
0.020	4.517	0.055	4.417	0.090	4.235
0.025	4.508	0.060	4.395	0.095	4.204
0.030	4.497	0.065	4.372	0.100	4.171

Table 4 –

Thickness correction factor $F(w/\overline{S})$ as a function of the ratio of slice thickness (w) to probe separation ($\overline{S}$).

$w/\overline{S}$	$F(w/\overline{S})$
0.5	0.997
0.6	0.992
0.7	0.982
0.8	0.966
0.9	0.944
1.0	0.921

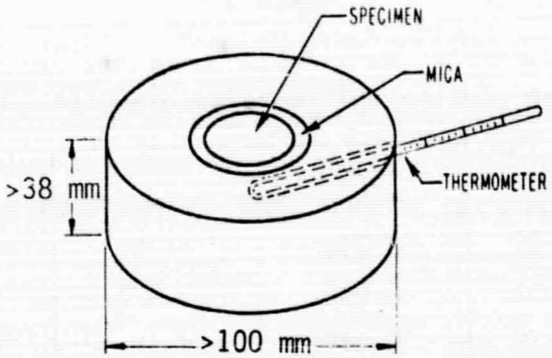


FIG. 1—Heat Sink with Specimen, Mica Insulator, and Thermometer.

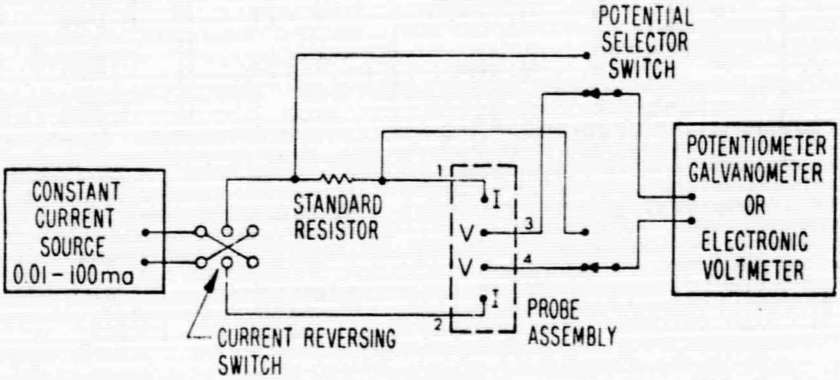


FIG. 2—Recommended Electrical Circuit.

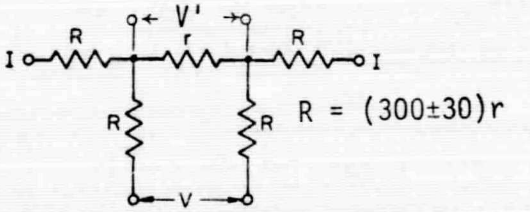


FIG. 3—Analog Test Circuit to Simulate Four-Probe Measurement. (See Table 2 for appropriate values of r.)

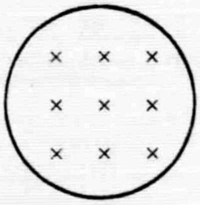
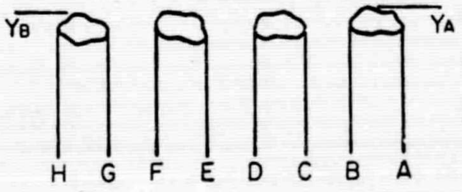


FIG. 4-Crosses Indicate Approximate Locations at Which Specimen Thickness is to be Measured.



(a) Measurement Locations.

(b) Photograph Showing Three Indentations of a Satisfactory Tip.

NOTE—The indentations are 0.05 mm apart

FIG. 5—Typical Probe Tip Indentation Pattern.

PROBE SERIAL NO.____________________
DATE____________________
OPERATOR____________________

$\bar{S}$____________________
F_{sp}____________________

DATA

Run No.	A	B	C	D	E	F	G	H	Y_A	Y_B
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

COMPUTATIONS

Run No.	$\frac{A+B}{2}$	$\frac{C+D}{2}$	$\frac{E+F}{2}$	$\frac{G+H}{2}$	S_1	S_2	S_3
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

$\bar{S}$ (AVERAGE)			
s (SAMPLE STD. DEV.)			

FIG. 6—Typical Data Sheet for Computing Probe Spacing.

ANALOG CIRCUIT DATA

DATE______________________________

R_s______________________________Ω

Run No.	V_{sf}(mv)	V_{nf}(mv)	V_{sr}(mv)	V_{nr}(mv)	ρ_f(Ω)	ρ_r(Ω)
1						
2						
3						
4						
5						

$\bar{\rho}$ (GRAND AVERAGE)	
s (SAMPLE STD. DEV.)	

NOTE—Use only the last two columns for direct-reading equipment.

FIG. 7—Typical Data Sheet for Analog Circuit Measurements.

SPECIMEN____________________
PROBE____________________
DATE____________________
R_s____________________Ω

THICK____________________cm
DIA.____________________cm
TYPE n p

Run No.	V_{sf}(mv)	V_f(mv)	V_{sr}(mv)	V_r(mv)	T (deg C)
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

(*a*)

Run No.	ρ_f(Ω)	ρ_r(Ω)	T (deg C)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

(*b*)

(*a*) For Standard Circuit. (*b*) For Direct-Reading Equipment.
NOTE—Record all information above columns in both cases.

FIG. 8—Typical Data Sheet for Four-Probe Resistivity Measurement.

Run No.	$R_f(\Omega)$	$R_r(\Omega)$	$R_{avg}(\Omega)$	$\rho_T(\Omega - cm)$	F_T	$\rho_{23}(\Omega - cm)$
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						

F_{sp}	
$\bar{S}/D$	
F_2	
$W/\bar{S}$	
$F(W/\bar{S})$	
F	
C_T	

ρ_{23} (AVERAGE) ☐

FIG. 9—Typical Computation Sheet for Four-Probe Resistivity Measurement.

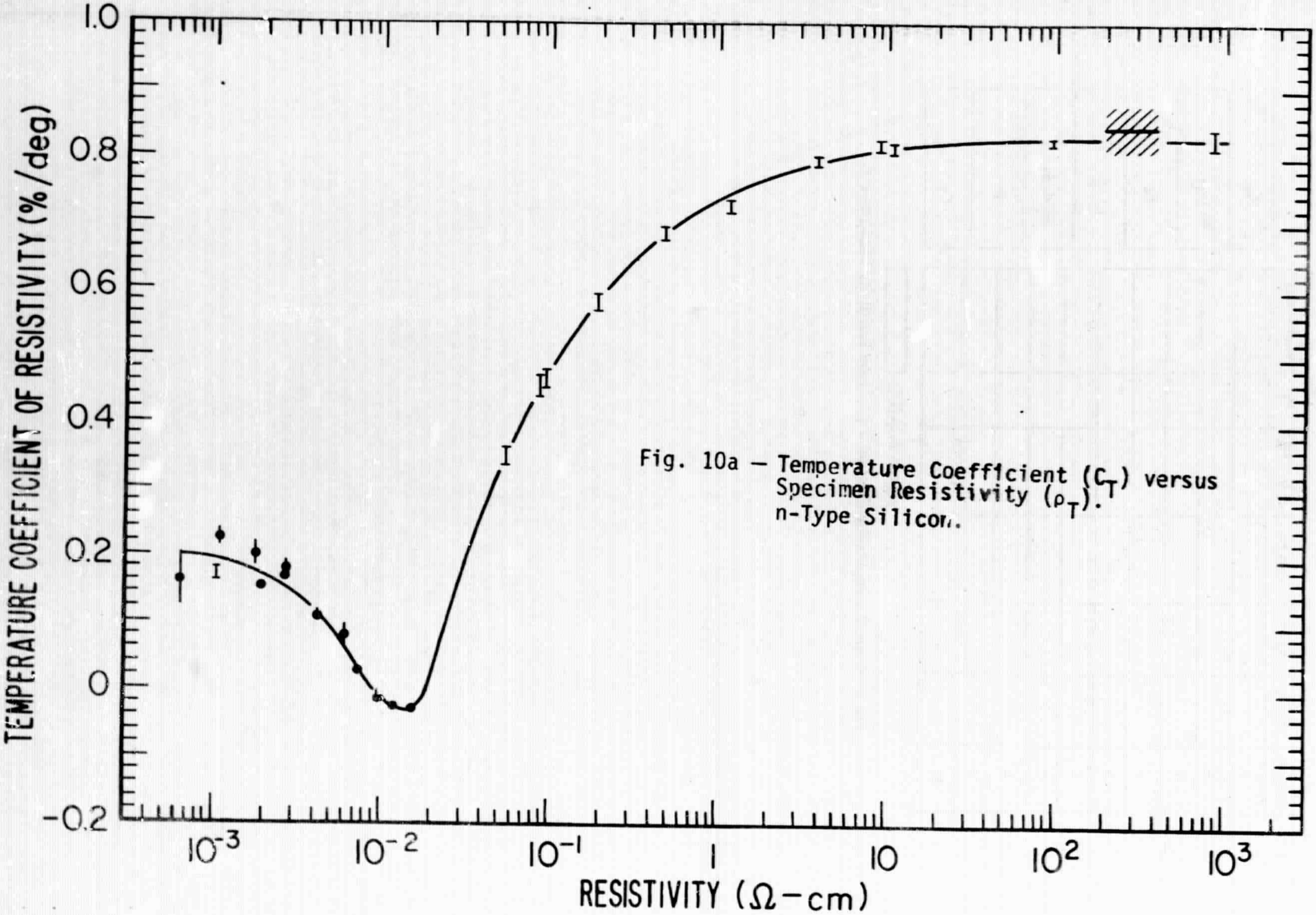


Fig. 10a — Temperature Coefficient (C_T) versus Specimen Resistivity (ρ_T). n-Type Silicon.

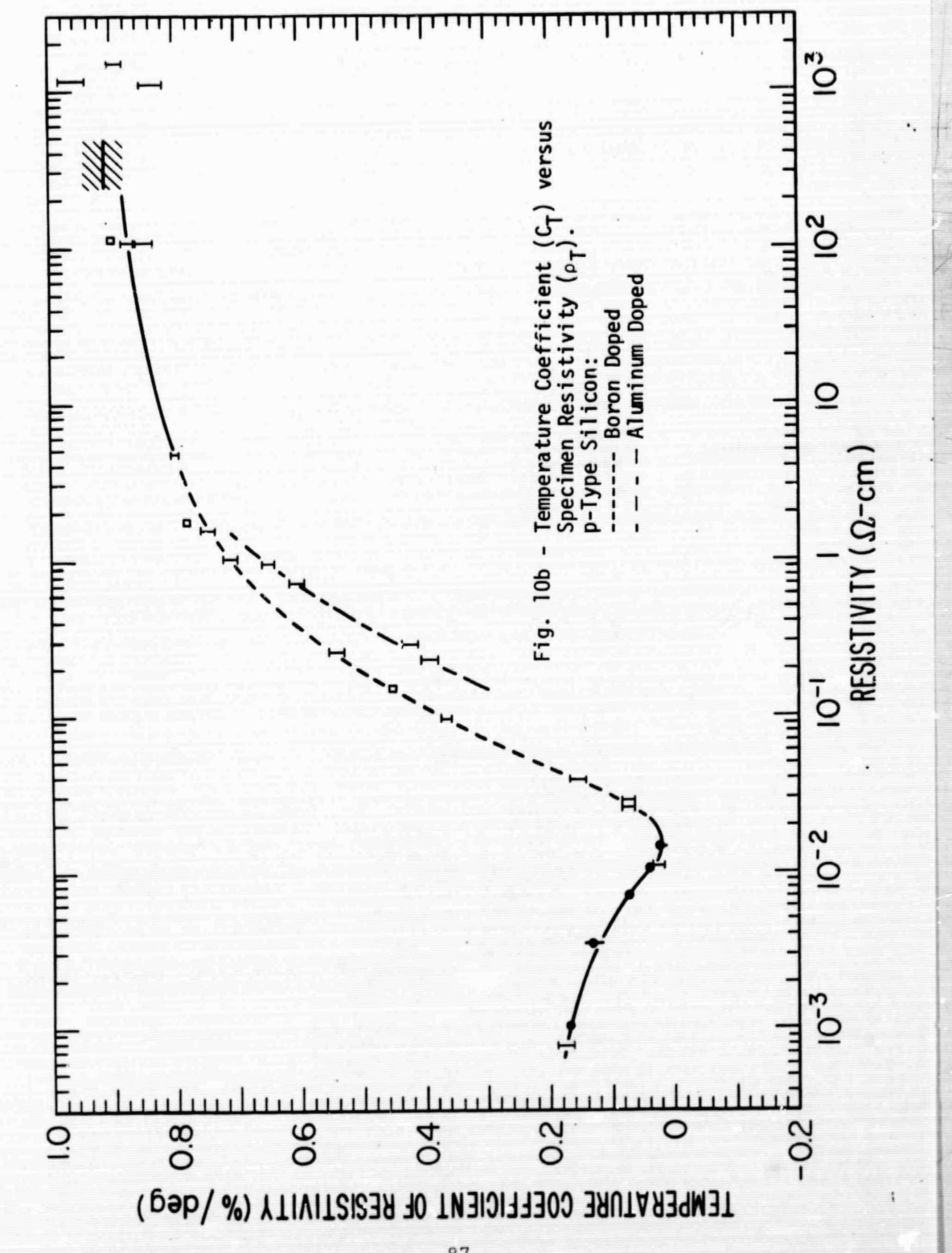


Fig. 10b - Temperature Coefficient (C_T) versus Specimen Resistivity (ρ_T).
p-Type Silicon:
------ Boron Doped
— — — Aluminum Doped