



SEMI AUX029-0214
INTERLABORATORY STUDY TO DETERMINE PRECISION OF
METHOD 1 OF SEMI MF673, TEST METHOD FOR MEASURING
RESISTIVITY OF SEMICONDUCTOR SLICES OF SHEET RESISTANCE
OF SEMICONDUCTOR FILMS WITH A NONCONTACT EDDY-CURRENT
GAGE

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**ASTM Committee F-1 on Electronics
Subcommittee F-1.06 on Silicon Materials and Process Control**

RESEARCH REPORT F01-1018

**INTERLABORATORY STUDY TO DETERMINE PRECISION OF Method 1 of ASTM
Standard Test Methods F 673 for Measuring Resistivity of Semiconductor Slices
or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current
Gage**

1. Introduction

1.1 *Application of Study*—The commercial interchange of silicon wafers between suppliers and their customers involves the specification and determination of the bulk resistivity of those wafers. The precision and bias of such measurements affects the efficiency and utility of these wafers in their utilization for fabricating electron devices.

1.2 This is the *third interlaboratory* study of this method. The results of the first two studies, which were carried out in the mid-1980s, were reported in Annexes to the Test Methods through the 1996 edition. Because this was the edition in effect at the time of this new interlaboratory test, conducted in 1999, they appear with this standard in Appendix A.

1.3 *Nature of Study*—Eight 200 mm diameter, single-side polished single crystal silicon wafers, with thickness from about 690 μm to 740 μm , with bulk resistivity ranging from 0.002 to 0.020 $\Omega\cdot\text{cm}$ (low resistivity set) and seventeen similar 200 mm wafers with bulk resistivity ranging from 1.1 to 60 $\Omega\cdot\text{cm}$ (high resistivity set) were employed in a round-robin experiment. Four of the low resistivity samples were *p*-type and four were of unknown type. Ten of the high resistivity samples were *p*-type, three were *n*-type, and four were of unknown type. Radial resistivity gradient of each sample was reported by the sample supplier to be $\leq 3\%$.

1.4 *Purpose of Study*—The *study's* purpose was to determine the repeatability and reproducibility of Method I of F673, on 200 mm nominal diameter single-side polished single crystal silicon wafers with thickness between about 693 μm and about 736 μm and with bulk resistivity from 0.002 to 0.020 $\Omega\cdot\text{cm}$ in the low resistivity group and from 1.10 to 60.0 $\Omega\cdot\text{cm}$ in the high resistivity group.

2. Test Method – F673-90 (Reapproved 1996)^{e1}

2.1 See Attachment A for the text of the standard in place at the time the interlaboratory test was conducted.

2.2 Only Method I of this *standard* was tested in this study. Although an interlaboratory test of Method II was indicated to be planned, such a test has never been conducted nor is one planned at the present time.

3. Participating Laboratories

3.1 Eight laboratories were originally scheduled to participate in the experiment. Four laboratories withdrew from the experiment before it was completed. The remaining four participating laboratories are listed in alphabetical order in Table 1. Because it is no longer current and should not be used, no contact information is provided.

Table 1 Laboratory Participants

ADE Corporation 80 Wilson Way Westwood, MA 02090 Stephanie Vokey		MEMC El. Materials 501 Pearl Drive St Peters, MO 63376 William Hughes	
Leighton Electronics First & South Streets Leighton, PA 18235-0328 Austin Blew		SEH America 4111 NE 12th Avenue Vancouver, WA 98682-6776 John Rudat	
		Experiment Coordinator: Winthrop A. Baylies BayTech Group	

3.2 One of the four remaining laboratories reported data from two separate measurement systems, which were counted as two laboratories for statistical purposes. This created a total of five reporting laboratories.

3.3 One of the originally scheduled laboratories was intended to make four-point probe resistivity measurements on a standard set of test specimens in order to tie the results of this experiment to absolute resistivity values. However, this procedure was abandoned as unnecessary during the experiment.

4. Test Program Instructions

4.1 Each participating laboratory was given the following letter:

January 1999

Dear ASTM Round Robin Participants

Thank you for participating in this round robin study. The study's purpose is to determine the repeatability and reproducibility of Method I of F673, on single-side polished monocrystalline silicon wafers with thickness between $\sim 693\text{ }\mu\text{m}$ and $\sim 736\text{ }\mu\text{m}$ thickness and 200 mm nominal diameter, with bulk resistivity from 0.002 to 0.020 $\Omega\cdot\text{cm}$ and from 1.10 to 60.0 $\Omega\cdot\text{cm}$. All wafers are marked on the top surface by a label, with a unique serial number, to the right of the fiducial. In addition to the label, the wafers are marked with the same serial number in felt tip pen, immediately to the right of the label.

Measurements are scheduled to begin January 4, 1999 and finish April 30, 1999, with preliminary results reported January 25, 1999 week at the ASTM meeting in San Jose, CA.

If you have any questions on the protocol, the wafer run list or the schedule, please do not hesitate to contact me. Thank you again for participating in this critical experiment.

Kind Regards,

Stephanie Vokey
ADE Corporation

4.2 Each participating laboratory was provided with detailed instructions for carrying out the experiment as follows:

Experimental Procedure for Each Laboratory

1. Select measurement systems that
 - a. can collect data on disk and can provide data printout, and
 - b. are known to be under statistical control: either
 - i) one system for each sample range, or
 - ii) a single system for both ranges. If equipment is available for only one range, complete the appropriate portion of the experiment.
 - c. Record on the Calibration Data Report the last date on which "in control" operation of each system was ascertained, and describe the method by which this "in control" was ascertained.
2. Calibrate the system and ascertain systems linearity in accordance with F673 ¶9.1.
3. Run the wafers used for calibration three successive "passes" and record on the Calibration Data Report each measured value of the center point resistivity (23°C equivalent) and thickness.
4. Verify the order of the wafers in both sets is as shown in Table 1 (they are ordered by resistivity, with highest resistivity first to simplify identifying measurement data anomalies). Re-order if needed, and report this event on the Test Data Report. If problems with wafer condition are observed, call the coordinator before proceeding.
5. Verify that the polished side of each wafer is up (away from the bar end of the cassette).
6. Run the Hi-resistivity Sample Set through the appropriate system in three successive passes, cassette to cassette. Do not make any adjustments during this three pass sequence. Record on disk or attached Test Data Report:
 - a. The measured center point resistivity and thickness value for each wafer
 - b. Date, time and operator information.
 - c. Record, on the Calibration Data Report, the ambient temperature at the beginning and end of the Run.
7. Execute Steps 2-6 above for the Lo-resistivity Sample Set, using the appropriate system.

8. Repeat steps 2-6 above, on two additional successive business days (thus, totaling three successive business days).
9. Retain disks, printouts and one copy of the reported data for your files.
 Note - In the interest of data transfer accuracy, disks (either 3 1/2 or 5 1/4 inch) should contain data in DOS/ASCII format with data arranged in an obvious format such as:
 Run #
 Wafer #
 Thickness/Resistivity Values - delimited by a space
10. Forward completed report sheets, data hardcopy and disks, via traceable means to the test coordinator:
 Winthrop A. Baylies
 BayTech Group
 80 Windsor Way
 Weston, MA 02493
11. Forward all samples and the report data book via traceable, EXPEDITED shipping method to the next laboratory, to the attention of the contact shown. The final laboratory should forward samples to the test coordinator at the conclusion of the last measurement cycle.
12. Questions??
 Call or e-mail Win Baylies at the following:
 Winthrop A. Baylies
 BayTech Group
 80 Windsor Way
 Weston, MA 02493
 Tel: 781-893-9919
 Fax: 781-893-0313
 E-Mail: winb@mediaone.net

Analysis:

- a. Test results will be evaluated in accordance with Method E 691 by the RR Task Force.
- b. A repeatability/reproducibility statement will be prepared for F 01.06, and a Research Report will be submitted to ASTM by the RR Task Force.

Sample Order

High Range Samples		Low Range Samples	
Cassette Slot #	Wafer ID # ¹	Cassette Slot #	Wafer ID # ¹
1	H1	1	L1
2	H2	2	L2
3	H3	3	L3
4	H4	4	L4
5	H5	5	L5
6	H6	6	L6
7	H7	7	L7
8	H8	8	L8
9	H9		
10	H10		
11	H11		
12	H12		
13	H13		
14	H14		
15	H15		
16	H16		
17	H17		

¹ Wafer ID is marked with various content and style.

5. Test Specimen Characteristics

5.1 The description of the test specimens used in the experiment is given in the table on the following page.

ASTM F673 Non-Contact Resistivity Round Robin
Sample Material (200MM Polished Wafers; laser marked (variously))

High Resistivity Samples

<i>Slot #</i>	<i>Marker & Scribed S/N</i>	<i>P Type</i>	<i>N Type</i>	<i>Nominal Resistivity ohm-cm RRG $\leq$ 3%</i>	<i>Laser Marked</i>
1.	H1	P		1.1	Yes
2.	H2	P		1.1	Yes
3.	H3	?	?	3-4	Yes
4.	H4	?	?	3-4	Yes
5.	H5	P		~10.7	Yes
6.	H6	P		12-13	No
7.	H7	P		13	No
8.	H8	P		13-14	No
9.	H9	P		13-14	No
10.	H10		N	15-16	Yes
11.	H11		N	15-16	Yes
12.	H12		N	21	Yes
13.	H13	P		21-22	No
14.	H14	P		21-22	No
15.	H15	P		~25	Yes
16.	H16	?	?	55-56	Yes
17.	H17	?	?	55-56	Yes

Low Resistivity Samples

<i>Slot #</i>	<i>Marker & Scribed S/N</i>	<i>P Type</i>	<i>N Type</i>	<i>Nominal Resistivity ohm-cm RRG $\leq$ 3%</i>	<i>Laser Mark</i>
1.	L1	?	?	.002-.003	Yes
2.	L2	?	?	.002-.003	Yes
3.	L3	P		.006-.008	Yes
4.	L4	P		.006-.008	Yes
5.	L5	P		.006-.008	Yes
6.	L6	P		.006-.008	Yes
7.	L7	?	?	.016	Yes
8.	L8	?	?	.016	Yes

6. Data Report Forms

6.1 There were three report forms issued to the participants. These were calibration data reports for the High and Low resistivity samples and a test data report. The format of these forms is shown on the following pages. The calibration data report for the High resistivity samples was printed on both sides.

Calibration Data Report - ASTM Round Robin on F673 Method I

HIGH Resistivity Wafer Runs - Calibration Data	
Laboratory _____	
Operator _____	Measurement Date _____
ADE System 9300 9600 7200 (Please circle one) Software Revision _____	
Leighton System Model _____ Software Revision _____	
Other System Manufacturer _____ Model _____ Software Revision _____	
Most Recent "In Control Operation" Date _____ How determined _____	
Ambient Temperature Run Start: _____ °F _____ °C Run End: _____ °	
HIGH Resistivity Calibration Masters	Thickness Calibration Masters
HIGH Resistivity Master #1 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #1 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
HIGH Resistivity Master #2 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #2 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
HIGH Resistivity Master #3 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #3 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
HIGH Resistivity Master #4 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #4 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
HIGH Resistivity Master #5 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #5 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
HIGH Resistivity Master #6 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #6 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm

Calibration Data Report - ASTM Round Robin on F673 Method I

LOW Resistivity Wafer Runs - Calibration Data	
Laboratory _____	
Operator _____	Measurement Date _____
ADE System 9300 9600 7200 (Please circle one) Software Revision _____	
Leighton System Model _____ Software Revision _____	
Other System Manufacturer _____ Model _____ Software Revision _____	
Most Recent "In Control Operation" Date _____ How determined _____	
Temperature Run Start: _____ °F [] °C Run End: _____ °	
LOW Resistivity Calibration Masters	Thickness Calibration Masters
LOW Resistivity Master #1 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #1 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
LOW Resistivity Master #2 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #2 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
LOW Resistivity Master #3 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #3 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
LOW Resistivity Master #4 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #4 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
LOW Resistivity Master #5 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #5 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm
LOW Resistivity Master #6 Stated Value _____ Ω-cm, _____ μm Reading 1: _____ Ω-cm Reading 2: _____ Ω-cm Reading 3: _____ Ω-cm	Thickness Calibration Master #6 Stated Value _____ μm Reading 1: _____ μm Reading 2: _____ μm Reading 3: _____ μm

Test Data Report - ASTM Round Robin on F673 Method I

This is a suggested format for recorded data; other formats are acceptable provided the listed information is reported. A supply of these forms is included for laboratories wishing to use them.

Laboratory _____ Operator _____
ADE System 9300 9600 7200 (Please circle one) Software Revision _____
Leighton System Model _____ Software Revision _____
Other System Manufacturer _____ Model _____ Software Revision _____
Measurement Date _____ [] Data is recorded on disk with file name _____

[illegible]

Questions

7. Results as Reported by Participants

7.1 Table 2 lists the results of the measurements on the high resistivity test samples as reported by the participants.

Table 2 Resistivity Measurements in Ω -cm on High Resistivity Test Samples as Reported by Participants

Lab #	Pass #	Sample #								
		1	2	3	4	5	6	7	8	9
1	1	1.089	1.122	3.943	3.980	10.84	12.55	13.03	13.62	13.64
	2	1.088	1.122	3.944	3.869	10.84	12.56	13.04	13.63	13.64
	3	1.089	1.122	3.945	3.988	10.84	12.56	13.05	13.62	13.64
	4	1.088	1.121	3.937	3.976	10.82	12.53	13.02	13.60	13.63
	5	1.088	1.121	3.943	3.981	10.84	12.56	13.04	13.63	13.65
	6	1.088	1.122	3.943	3.981	10.84	12.56	13.04	13.63	13.66
	7	1.091	1.122	3.944	3.984	10.84	12.56	13.05	13.63	13.64
	8	1.089	1.123	3.946	3.983	10.85	12.57	13.05	13.64	13.65
	9	1.090	1.122	3.946	3.983	10.85	12.57	13.06	13.64	13.65
2	1	1.089	1.121	3.939	3.988	10.83	12.54	13.03	13.62	13.64
	2	1.089	1.122	3.941	3.989	10.82	12.55	13.03	13.62	13.65
	3	1.089	1.122	3.939	3.990	10.83	12.55	13.05	13.63	13.66
	4	1.089	1.122	3.943	3.984	10.83	12.54	13.04	13.63	13.65
	5	1.090	1.122	3.942	3.982	10.83	12.54	13.03	13.62	13.64
	6	1.089	1.122	3.940	3.983	10.83	12.55	13.03	13.61	13.64
	7	1.090	1.122	3.943	3.987	10.84	12.55	13.03	13.63	13.65
	8	1.090	1.123	3.945	3.983	10.84	12.55	13.03	13.63	13.65
	9	1.090	1.122	3.944	3.986	10.84	12.55	13.04	13.63	13.65
3	1	1.095	1.126	3.953	3.996	10.89	12.62	13.10	13.70	13.72
	2	1.094	1.127	3.953	3.996	10.89	12.62	13.11	13.70	13.73
	3	1.095	1.127	3.953	3.998	10.89	12.63	13.11	13.71	13.73
	4	1.094	1.126	3.956	3.998	10.89	12.61	13.11	13.70	13.73
	5	1.095	1.127	3.956	3.998	10.90	12.62	13.11	13.71	13.73
	6	1.094	1.126	3.957	4.002	10.90	12.62	13.10	13.71	13.73
	7	1.095	1.126	3.957	4.002	10.90	12.62	13.11	13.71	13.74
	8	1.094	1.127	3.955	4.002	10.90	12.62	13.11	13.70	13.73
	9	1.094	1.127	3.955	4.002	10.90	12.62	13.11	13.71	13.73
4	1	1.098	1.130	3.970	4.010	10.92	12.64	13.13	13.73	13.75
	2	1.100	1.132	3.978	4.017	10.95	12.67	13.16	13.74	13.77
	3	1.100	1.132	3.978	4.017	10.95	12.67	13.16	13.75	13.78
	4	1.096	1.129	3.967	4.006	10.91	12.63	13.13	13.72	13.75
	5	1.099	1.131	3.975	4.013	10.93	12.66	13.15	13.75	13.77
	6	1.100	1.131	3.976	4.014	10.93	12.66	13.15	13.75	13.77
	7	1.101	1.133	3.980	4.020	10.95	12.67	13.17	13.76	13.80
	8	1.103	1.136	3.989	4.028	10.97	12.70	13.20	13.78	13.81
	9	1.104	1.137	3.993	4.031	10.98	12.71	13.21	13.80	13.81
5	1	1.101259	1.134193	3.958755	4.001934	10.87418	12.65798	13.09727	13.68827	13.70343
	2	1.103550	1.136737	3.973577	4.007016	10.89960	12.65003	13.12645	13.73883	13.77688
	3	1.105541	1.137442	3.975222	4.014207	10.91640	12.65212	13.12599	13.72826	13.74362
	4	1.101891	1.132633	3.963221	3.998242	10.87394	12.62908	13.09006	13.69548	13.71064
	5	1.105241	1.135280	3.968981	4.003555	10.90371	12.63642	13.13374	13.72343	13.72331
	6	1.105393	1.137978	3.968501	4.014635	10.90856	12.66361	13.12626	13.72020	13.75418
	7	1.100537	1.132859	3.955460	3.988268	10.86317	12.59752	13.08285	13.67804	13.69321
	8	1.102499	1.135071	3.962687	3.998642	10.87678	12.62365	13.09762	13.69161	13.71410
	9	1.103629	1.136009	3.966557	4.003266	10.89875	12.63090	13.11936	13.71951	13.74498

Lab #	Pass #	Sample #							
		10	11	12	13	14	15	16	17
1	1	15.42	16.58	21.30	21.48	21.55	25.42	52.12	55.97
	2	15.43	16.60	21.32	21.51	21.56	25.44	52.14	56.00
	3	15.44	16.60	21.32	21.50	21.58	25.45	52.15	56.01
	4	15.47	16.63	21.30	21.49	21.57	25.42	52.11	55.96
	5	15.49	16.66	21.33	21.51	21.59	25.43	52.14	56.02
	6	15.49	16.66	21.34	21.52	21.58	25.44	52.16	56.04
	7	15.50	16.67	21.34	21.48	21.56	25.42	52.09	55.96
	8	15.51	16.68	21.34	21.51	21.58	25.45	52.14	56.02
2	9	15.51	16.68	21.34	21.51	21.59	25.45	52.19	56.02
	1	15.42	16.60	21.31	21.51	21.57	25.41	52.21	56.18
	2	15.43	16.59	21.31	21.51	21.58	25.41	52.17	56.15
	3	15.43	16.60	21.32	21.51	21.58	25.43	52.21	56.20
	4	15.48	16.65	21.31	21.50	21.57	25.43	52.28	56.17
	5	15.47	16.63	21.32	21.51	21.58	25.44	52.20	56.19
	6	15.47	16.63	21.30	21.50	21.58	25.43	52.26	56.17
	7	15.48	16.65	21.33	21.52	21.59	25.45	52.28	56.23
3	8	15.49	16.65	21.33	21.51	21.59	25.48	52.30	56.16
	9	15.49	16.66	21.33	21.51	21.59	25.45	52.27	56.23
	1	15.58	16.77	21.48	21.66	21.73	25.60	51.56	55.27
	2	15.58	16.78	21.48	21.67	21.74	25.63	52.37	56.07
	3	15.60	16.78	21.47	21.66	21.73	25.61	52.20	56.00
	4	15.59	16.77	21.48	21.66	21.73	25.59	52.60	56.57
	5	15.60	16.77	21.49	21.67	21.72	25.61	52.59	56.60
	6	15.58	16.78	21.47	21.65	21.74	25.60	52.58	56.60
4	7	15.59	16.78	21.48	21.67	21.75	25.60	52.60	56.59
	8	15.60	16.77	21.50	21.67	21.75	25.63	52.65	56.60
	9	15.60	16.78	21.50	21.67	21.75	25.64	52.60	56.67
	1	15.64	16.82	21.52	21.71	21.78	25.68	52.63	56.54
	2	15.67	16.85	21.55	21.72	21.81	25.72	52.63	56.48
	3	15.67	16.86	21.55	21.74	21.81	25.72	52.65	56.47
	4	15.64	16.80	21.50	21.69	21.77	25.65	52.60	56.38
	5	15.66	16.84	21.51	21.72	21.76	25.69	52.55	56.41
5	6	15.67	16.85	21.54	21.70	21.80	25.68	52.57	56.41
	7	15.69	16.87	21.57	21.76	21.82	25.73	52.61	56.46
	8	15.72	16.89	21.60	21.77	21.85	25.76	52.68	56.50
	9	15.72	16.90	21.61	21.80	21.86	25.77	52.68	56.52
	1	15.61640	16.81122	21.43513	21.69007	21.72812	25.57965	52.35887	56.35833
	2	15.67645	16.86221	21.52701	21.70659	21.81705	25.55523	52.47237	56.16117
	3	15.67190	16.85592	21.53580	21.69910	21.77336	25.60104	52.56920	56.18439
	4	15.64191	16.83253	21.44227	21.61844	21.68834	25.52263	52.37543	56.17150
5	5	15.65043	16.81344	21.46256	21.67763	21.70823	25.58272	52.28904	56.12545
	6	15.67379	16.84886	21.47897	21.66318	21.74455	25.60480	52.25496	56.29947
	7	15.60134	16.77691	21.38075	21.58593	21.67401	25.50078	52.23192	56.12897
	8	15.65125	16.82035	21.43057	21.66628	21.68606	25.52703	52.37812	56.20842
	9	15.66187	16.82531	21.45677	21.69724	21.72021	25.55929	52.39162	56.17580

7.2 Table 3 lists the results of the measurements on the low resistivity test specimens as reported by the participants.

Table 3 Resistivity Measurements in mΩ-cm on Low Resistivity Test Samples as Reported by Participants

Lab #	Pass #	Sample #							
		1	2	3	4	5	6	7	8
1	1	2.845	2.849	6.872	6.993	7.945	7.988	16.38	16.59
	2	2.841	2.849	6.887	6.992	7.947	7.988	16.39	16.57
	3	2.843	2.851	6.885	6.991	7.947	7.988	16.39	16.58
	4	2.842	2.846	6.878	6.983	7.942	7.986	16.38	16.56
	5	2.839	2.846	6.878	6.983	7.942	7.986	16.38	16.56
	6	2.839	2.846	6.878	6.984	7.942	7.986	16.38	16.56
	7	2.844	2.850	6.880	6.989	7.950	7.989	16.39	16.57
	8	2.843	2.849	6.880	6.987	7.950	7.989	16.39	16.57
	9	2.843	2.849	6.878	6.987	7.950	7.987	16.39	16.57
2	1	2.828	2.834	6.849	6.958	7.911	7.953	16.33	16.52
	2	2.828	2.834	6.849	6.958	7.910	7.953	16.33	16.52
	3	2.828	2.834	6.849	6.958	7.910	7.953	16.33	16.52
	4	2.829	2.835	6.853	6.964	7.914	7.957	16.34	16.53
	5	2.828	2.835	6.852	6.960	7.914	7.956	16.34	16.53
	6	2.828	2.835	6.852	6.963	7.915	7.956	16.34	16.53
	7	2.827	2.834	6.853	6.961	7.914	7.957	16.34	16.53
	8	2.826	2.834	6.852	6.961	7.916	7.956	16.34	16.53
	9	2.826	2.834	6.852	6.961	7.916	7.956	16.34	16.53
3	1	2.790	2.796	6.783	6.889	7.837	7.879	16.21	16.39
	2	2.789	2.796	6.783	6.889	7.837	7.879	16.21	16.39
	3	2.789	2.796	6.780	6.889	7.837	7.879	16.21	16.39
	4	2.790	2.796	6.783	6.890	7.837	7.881	16.21	16.40
	5	2.790	2.796	6.783	6.890	7.837	7.879	16.21	16.40
	6	2.790	2.796	6.784	6.889	7.839	7.881	16.21	16.40
	7	2.789	2.796	6.783	6.890	7.837	7.879	16.21	16.39
	8	2.789	2.796	6.784	6.890	7.837	7.879	16.21	16.39
	9	2.790	2.796	6.783	6.890	7.837	7.879	16.21	16.39
4	1	2.829	2.836	6.856	6.966	7.923	7.966	16.37	16.55
	2	2.829	2.836	6.858	6.966	7.923	7.966	16.37	16.55
	3	2.829	2.836	6.858	6.966	7.922	7.966	16.37	16.55
	4	2.827	2.834	6.850	6.958	7.914	7.954	16.35	16.53
	5	2.827	2.834	6.850	6.958	7.914	7.957	16.35	16.53
	6	2.827	2.834	6.850	6.958	7.913	7.957	16.35	16.53
	7	2.827	2.836	6.853	6.963	7.921	7.962	16.36	16.54
	8	2.827	2.836	6.855	6.963	7.919	7.962	16.36	16.54
	9	2.827	2.836	6.855	6.963	7.919	7.962	16.36	16.54
5	1	2.823916	2.831072	6.914389	7.023828	7.996580	8.044038	16.56454	16.75706
	2	2.826955	2.833392	6.916788	7.025778	7.998293	8.038082	16.56611	16.75906
	3	2.827317	2.834462	6.916894	7.024783	7.998864	8.039223	16.56679	16.76006
	4	2.825877	2.831072	6.917926	7.028494	8.003482	8.043657	16.56252	16.76252
	5	2.825410	2.833272	6.918034	7.029254	8.004625	8.044989	16.57289	16.76627
	6	2.827788	2.834519	6.918556	7.027235	8.005578	8.045750	16.57357	16.76702
	7	2.823793	2.831245	6.916260	7.026145	8.003291	8.043467	16.56387	16.75657
	8	2.826294	2.833031	6.915952	7.027633	7.997151	8.044608	16.56499	16.77197
	9	2.825677	2.832421	6.918021	7.028593	8.002339	8.042706	16.57765	16.75607

8. Statistical Data Summary

8.1 The 45 measurements on each test specimen were analyzed in accordance with the procedures in ASTM Practice E 691¹ using the following relationships from the practice (the equation numbers listed are those found in the 2005 edition of the practice):

8.1.1 Cell average center point resistivity (avg):

$$avg = \sum_1^n x / n \quad (1)$$

where x = the individual test results in one cell and n = the number of test results in one cell.

8.1.2 The cell standard deviation of the measured center point resistivity (s):

$$s = \sqrt{\sum_1^n (x - avg)^2 / (n - 1)} \quad (2)$$

where the symbols have the same meaning as for Equation 1.

8.1.3 The average of the cell averages for one material (AVG):

$$AVG = \sum_1^p avg / p \quad (3)$$

where p = the number of laboratories in the study (in this case taken as 5).

8.1.4 The cell deviation (d) for each of the five laboratories:

$$d = avg - AVG \quad (4)$$

where the symbols have the same meaning as for Equations 1 and 3.

8.1.5 The standard deviation of the cell averages (s_x):

$$s_x = \sqrt{\sum_1^p d^2 / (p - 1)} \quad (6)$$

where the symbols have the same meaning as for Equations 2 and 3.

8.1.6 The repeatability standard deviation (s_r):

$$s_r = \sqrt{\sum_1^p s^2 / p} \quad (7)$$

where the symbols have the same meaning as for Equations 3 and 4.

8.1.7 The provisional value of the reproducibility standard deviation (s_R)*:

$$(s_R)^* = \sqrt{(s_x)^2 + (s_r)^2 (n - 1) / n} \quad (8)$$

where the symbols have the same meaning as for Equations 1, 6 and 7.

8.1.7.1 Note that the reproducibility standard deviation (s_R) is taken as the larger of (s_r) and (s_R)*

8.1.8 The between-laboratory consistency statistic (h):

$$h = d / s_x \quad (9)$$

where the symbols have the same meaning as for Equations 4 and 6.

8.1.9 The within-laboratory consistency statistic (k):

$$k = s / s_r \quad (10)$$

where the symbols have the same meaning as for Equations 3 and 4.

8.2 *Development of h- and k-Statistics:* First, the h and k statistics are examined for outliers and other problems as indicated in Practice E 691. These statistics are plotted both in order of laboratory and of material in Figures 1 through 8 for both the high resistivity specimens and the low resistivity specimens.

8.2.1 For the case of plots in laboratory order, the first laboratory is on the left hand side of the plot and the fifth laboratory is on the right hand side of the plot. For each laboratory, the first material (1) is on the left hand side of the group and the last material (17 or 8) is on the right hand side of the group.

¹ ASTM Practice E 691 – 05 —Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

8.2.2 For the case of plots in material order, the first material is on the left hand side of the plot and the last (17 or 8) material is on the right hand side of the plot. For each material, the first laboratory is on the left hand side of the group and the fifth laboratory is on the right hand side of the group.

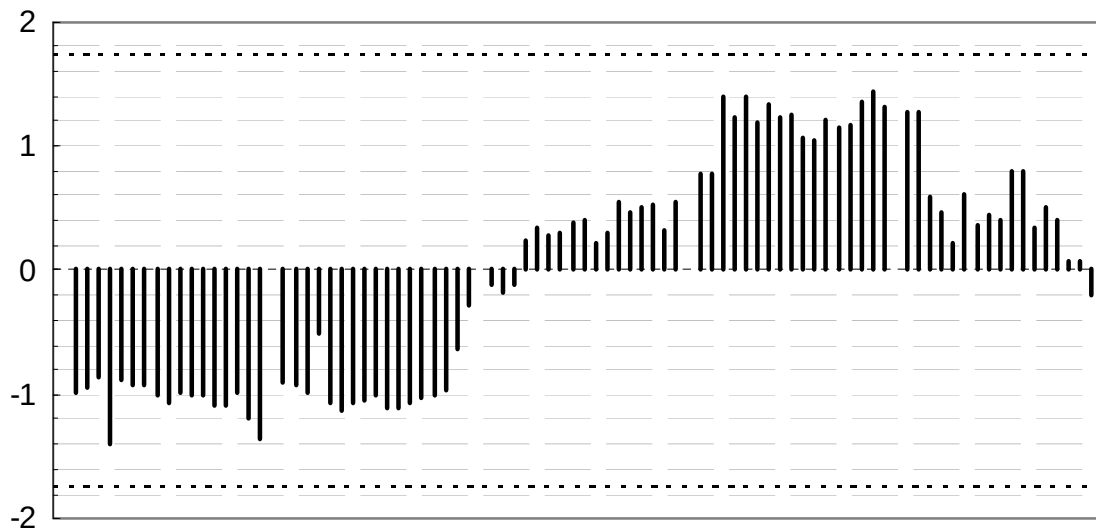


Figure 1
***h*-Statistic for High Resistivity Specimens, by Laboratory**

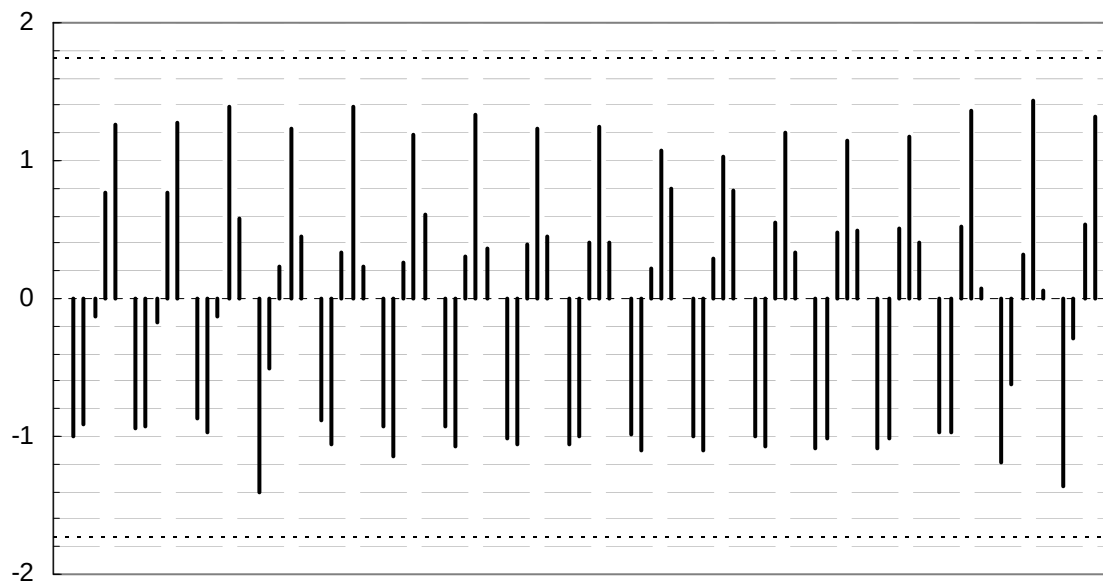


Figure 2
***h*-Statistic for High Resistivity Specimens, by Material**

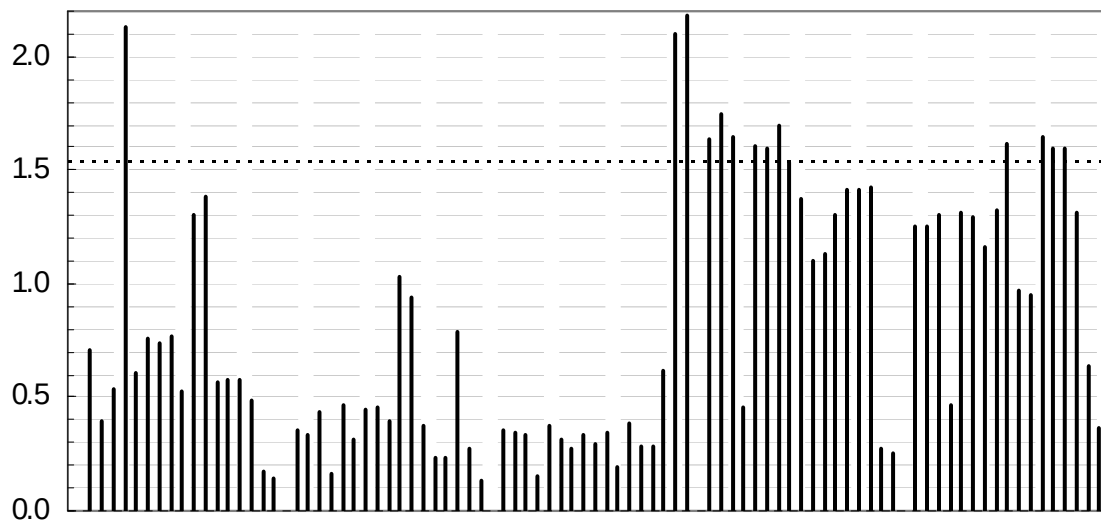


Figure 3
k-Statistic for High Resistivity Specimens, by Laboratory

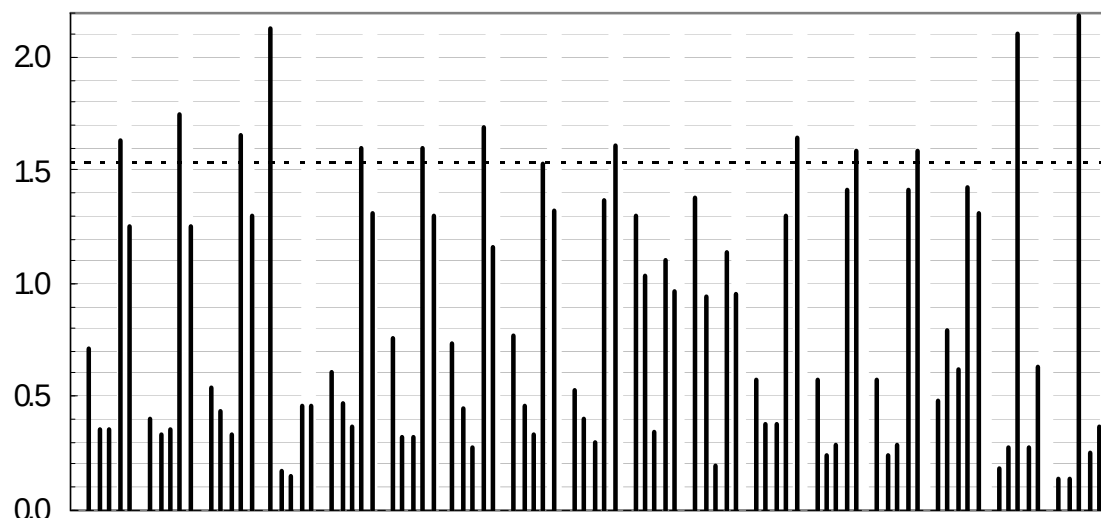


Figure 4
k-Statistic for High Resistivity Specimens, by Material

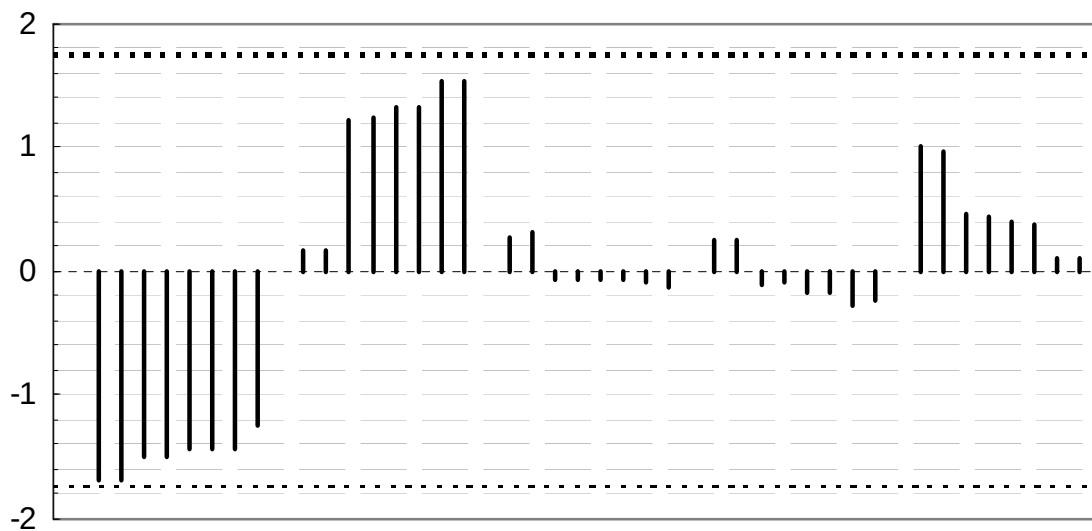


Figure 5
***h*-Statistic for Low Range Specimens, by Laboratory**

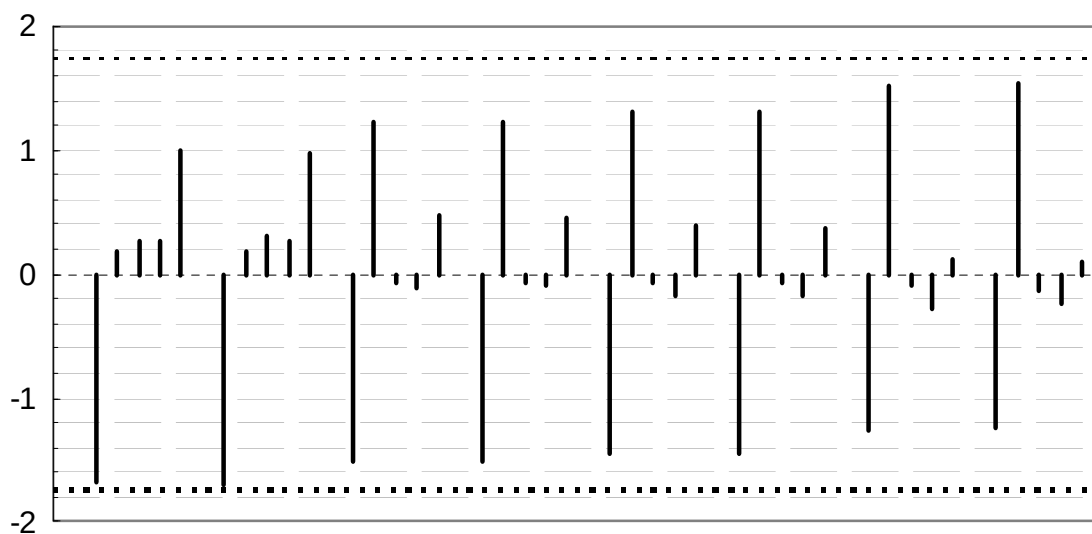


Figure 6
***h*-Statistic for Low Range Specimens, by Material**

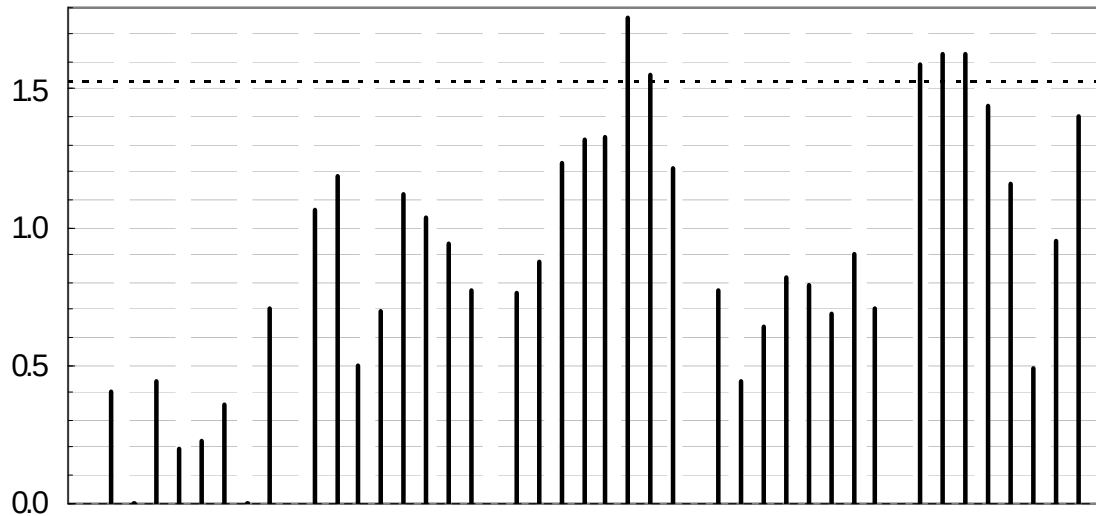


Figure 7
k-Statistic for Low Range Specimens, by Laboratory

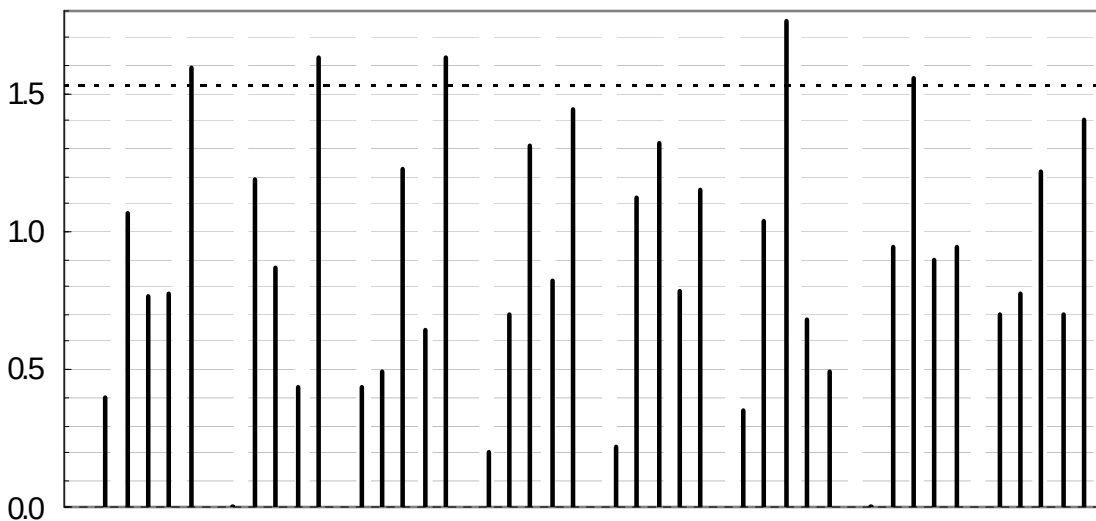


Figure 8
k-Statistic for Low Range Specimens, by Material

8.3 Analysis of *h*- and *k*-Statistics: For this experiment, if the data for the laboratory with the very high *k*-statistic values for the 16th and 17th samples are removed, one of the repeatability values actually increased while the other decreased. Consequently, the 95% repeatability and reproducibility were calculated using the full data set as follows:

8.3.1 The 95% repeatability (*r*):²

$$r = 2.7718s_r \quad (11)$$

where s_r is calculated from Equation 7.

8.3.2 The 95% reproducibility (*R*):

$$R = 2.7718s_R \quad (12)$$

where s_R is the larger of s_R^* calculated from Equation 8 or s_r calculated from Equation 7.

² Note that this factor, recommended by the Committee F-! Statistician, Paul Langer, is slightly smaller than the factor of 2.8 used in the 2005 edition of ASTM Practice F 691.

8.4 *Results for High Resistivity Specimens:* The results of all these calculations for the high resistivity specimens are given in Table 4 and in Table 5 for the low resistivity specimens. These tables are the equivalent of Table 11 in the 2005 edition of ASTM Practice F 691, with the addition of the columns showing the percent deviation between (1) the pooled within-laboratory relative repeatability and the calculated 95% repeatability (r) found for each sample from Equation (11) and (2) the percent deviation between the pooled between-laboratory reproducibility and the calculated 95% reproducibility (R) found for each sample from Equation (12).

Table 4 Data Analysis Calculations for the High Resistivity Samples

Sample #	AVG ($\Omega\text{-cm}$)	s_x ($\Omega\text{-cm}$)	s_r ($\Omega\text{-cm}$)	s_R^* ($\Omega\text{-cm}$)	r ($\Omega\text{-cm}$)	% Diff	R ($\Omega\text{-cm}$)	% Diff
1	1.0952	0.0063829	0.0014831	0.0065342	0.0041109	153.5926%	0.018112	-15.9447%
2	1.1276	0.006075	0.0015146	0.0062405	0.0041983	396.6347%	0.017298	-9.3857%
3	3.9569	0.015478	0.005010	0.016182	0.013887	125.2086%	0.044854	22.6214%
4	3.9950	0.018198	0.017758	0.024728	0.049222	-15.2822%	0.068542	-18.9823%
5	10.880	0.045406	0.014296	0.047364	0.039627	31.5390%	0.13128	15.1974%
6	12.606	0.052147	0.015880	0.054254	0.044016	42.1072%	0.15038	16.5196%
7	13.092	0.053228	0.016409	0.055431	0.045484	60.4417%	0.15364	18.4375%
8	13.684	0.056447	0.015986	0.058425	0.04431	88.2209%	0.16194	17.4530%
9	13.706	0.058339	0.016873	0.060469	0.04677	100.6195%	0.16761	13.6664%
10	15.570	0.098571	0.026979	0.101800	0.07478	39.4095%	0.28217	-23.2988%
11	16.745	0.104844	0.027876	0.108088	0.07727	48.4144%	0.29960	-22.3106%
12	21.428	0.102083	0.029351	0.105767	0.08135	53.7716%	0.29317	1.5956%
13	21.615	0.10448	0.025458	0.107200	0.07056	92.0574%	0.29714	1.1158%
14	21.685	0.10318	0.027013	0.106278	0.07487	94.9269%	0.29458	2.3221%
15	25.551	0.11816	0.027689	0.121014	0.07675	103.7469%	0.33543	5.8829%
16	52.358	0.18390	0.16763	0.24248	0.46463	-64.1005%	0.67211	8.2820%
17	56.236	0.17309	0.21479	0.26640	0.59535	-70.2319%	0.7384	5.8622%

8.5 The repeatability and reproducibility for the high resistivity specimens are plotted as a function of average resistivity in Figure 9. In this figure the calculated 95% repeatability and 95% reproducibility are for each specimen are plotted as data points and the pooled within-laboratory relative repeatability and the pooled between-laboratory reproducibility are plotted as straight lines. The former is estimated to be $0.49\% \times \text{Average Resistivity}$ and the latter is estimated to be $1.39\% \times \text{Average Resistivity}$.

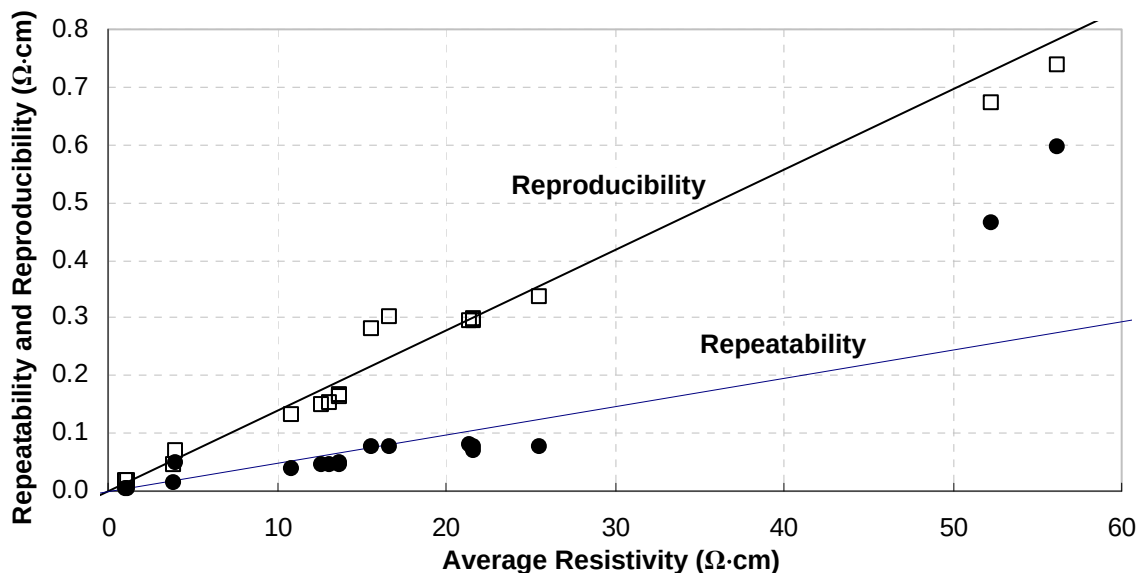


Figure 9
Repeatability and Reproducibility for High Resistivity Specimens as a Function of Average Resistivity

8.6 *Results for Low Resistivity Specimens:* The results of all these calculations for the low resistivity specimens are given in Table 5. This table is the equivalent of Table 11 in the 2005 edition of ASTM Practice F 691, with the addition of the columns showing the percent deviation between (1) the pooled within-laboratory relative repeatability and the calculated 95% repeatability (r) found for each sample from Equation (11) and (2) the percent deviation between the pooled between-laboratory reproducibility and the calculated 95% reproducibility (R) found for each sample from Equation (12).

Table 5 Data Analysis Calculations for the Low Resistivity Samples

Sample #	AVG (mΩ-cm)	s_x (mΩ-cm)	s_r (mΩ-cm)	s_R^* (mΩ-cm)	r (mΩ-cm)	% Diff	R (mΩ-cm)	% Diff
1	2.8226	0.019581	0.0013119	0.019620	0.0036364	14.6176%	0.054383	14.6176%
2	2.8293	0.019651	0.0011491	0.019680	0.0031851	2.2862%	0.054550	2.2862%
3	6.8569	0.049096	0.0026808	0.049161	0.0074305	-1.5079%	0.13627	-1.5079%
4	6.9654	0.050149	0.0026635	0.050211	0.0073827	-3.7816%	0.13918	-3.7816%
5	7.9233	0.059390	0.0030488	0.059459	0.0084508	-3.1337%	0.16481	-3.1337%
6	7.9653	0.059177	0.0025216	0.059225	0.0069895	-25.3567%	0.16416	-25.3567%
7	16.372	0.12872	0.0055750	0.12883	0.0154529	-16.5429%	0.35709	-16.5429%
8	16.558	0.1324	0.0071491	0.13257	0.0198159	8.0829%	0.36746	8.0829%

8.7 The repeatability and reproducibility for the low resistivity specimens are plotted as a function of average resistivity in figure 10. In this figure the calculated repeatability and reproducibility are for each specimen are plotted as data points and the pooled within-laboratory relative repeatability and the pooled between-laboratory reproducibility are plotted as straight lines. The former is estimated to be $0.11\% \times \text{Average Resistivity}$ and the latter is estimated to be $2.05\% \times \text{Average Resistivity}$.

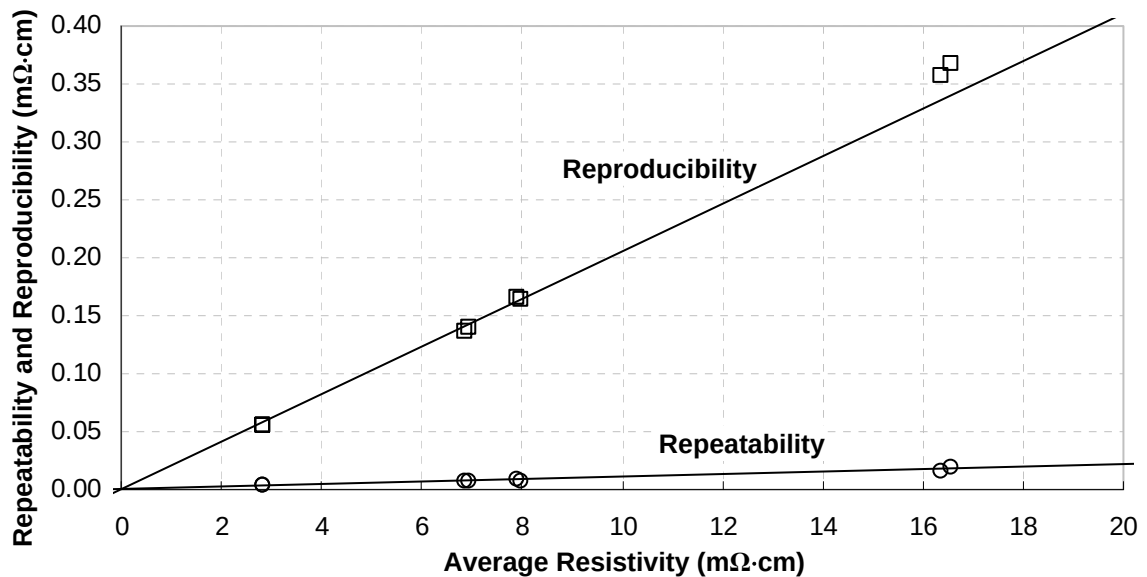


Figure 10
Repeatability and Reproducibility for Low Resistivity Specimens as a Function of Average Resistivity

9. Research Report Summary

9.1 *Significance:* Although the number of laboratories in this study is less than the six-laboratory minimum for determining precision prescribed in accordance with Practice E 691, the number of samples and determinations meets the requirements of Practice E 691.

9.2 Conclusions

9.2.1 Precision

9.2.1.1 Based on the results of the statistical analysis of the data from the two sets of specimens, the pooled, within-laboratory relative repeatability is estimated to be 0.11% for the low range sample set and 0.49% for the high range sample set.

9.2.1.2 The pooled, between-laboratory reproducibility is estimated to be 2.05% for the low range sample set and 1.39% for the high range sample set.

9.2.1.3 There is no apparent dependence on the conductivity type of the specimens.

9.2.2 Bias

9.2.2.1 Each laboratory used its own in-house calibration wafers. The bias between those wafers and NIST SRMs[®] is unknown. Therefore no bias statement can be made based on the results from this experiment.

9.2.2.2 However, ASTM Guide F 1527 (now SEMI MF1527³) can be used to establish bias within any laboratory.

³ SEMI MF1527 — Guide for Application of Certified Reference Materials and Reference Wafers for Calibration and Control of Instruments for Measuring Resistivity of Silicon

Attachment A

F 673 – 90 (Reapproved 1996)^{ε1} Standard Test Methods for Measuring Resistivity of Semiconductor Slices or Sheet Resistance of Semiconductor Films with a Noncontact Eddy-Current Gage⁴

This standard is issued under the fixed designation F 673; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords were added editorially in January 1996.

INTRODUCTION

This test method is intended to outline the principles of eddy-current measurements as they relate to semiconductor substrates and certain thin films fabricated on such substrates as well as requirements for setting up and calibrating such instruments for use particularly at a buyer-seller interface. Because such eddy-current measurements for semiconductor materials are made almost exclusively with commercial instrumentation from one of several suppliers, some details included here such as specific range limits and manner of entering slice/wafer thickness values to obtain resistivity values may not apply strictly to all instruments. In all such cases, the owner's manual for the particular instrument shall be considered to contain the correct information for that instrument. It is to be noted that an eddy-current instrument directly measures conductance of a specimen. Values of sheet resistance and resistivity are calculated from the measured conductance, with the resistivity values also requiring a measurement of specimen thickness.

1. Scope

1.1 These test methods cover the nondestructive measurement of bulk resistivity of silicon and certain gallium-arsenide slices and of the sheet resistance of thin films of silicon or gallium-arsenide fabricated on a limited range of substrates at the slice center point using a noncontact eddy-current gage.

1.1.1 The measurements are made at room temperature between 18 and 28°C.

1.2 These test methods are presently limited to single-crystal and polycrystalline silicon and extrinsically conducting gallium-arsenide bulk specimens or to thin films of silicon or gallium-arsenide fabricated on relatively high resistivity substrates but in principle can be extended to cover other semiconductor materials.

1.2.1 The bulk silicon or gallium-arsenide specimens may be single crystal or poly crystal and of either conductivity type (p or n) in the form of slices (round or other shape) that are free of diffusions or other conducting layers that are fabricated thereon, that are free of cracks, voids or other structural discontinuities, and that have (1) an edge-to-edge dimension, measured through the slice center point, not less than 25 mm (1.00 in.); (2) thickness in the range 0.1 to 1.0 mm (0.004 to 0.030 in.), inclusive, and (3) resistivity in the range 0.001 to 200 $\Omega\cdot\text{cm}$, inclusive. Not all combinations of thickness and resistivity may be measurable. The instrument will fundamentally be limited to a fixed sheet resistance range such as given in 1.2.2; see also 9.3.

1.2.2 The thin films of silicon or gallium-arsenide may be fabricated by diffusion, epitaxial or ion implant processes. The sheet resistance of the layer should be in the nominal range from 2 to 3000 Ω per square. The substrate on which the thin film is fabricated should have a minimum edge to edge dimension of 25 mm, measured through the centerpoint and an effective sheet resistance at least 1000 $\times$ that of the thin film. The effective sheet resistance of a bulk substrate is its bulk resistivity (in $\Omega\cdot\text{cm}$) divided by its thickness in cm.

1.2.3 Measurements are not affected by specimen surface finish.

1.3 These test methods require the use of resistivity standards to calibrate the apparatus (see 7.1), and a set of reference specimens for qualifying the apparatus (see 7.1.2).

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

⁴ This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.06 on Electrical and Optical Measurement.

Current edition approved April 27, 1990. Published June 1990. Originally published as F673 – 80. Last previous edition F673 – 89.

F 673

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- E 1 Specification for ASTM Thermometers⁵
- F 81 Test Method for Measuring Radial Resistivity Variation on Silicon Slices⁶
- F 84 Test Method for Measuring Resistivity of Silicon Slices with an In-Line Four-Point Probe³
- F 374 Test Method for Sheet Resistance of Silicon Epitaxial, Diffused, and Ion-Implanted Layers Using an In-Line Four-Point Probe³
- F 533 Test Method for Thickness and Thickness Variation of Silicon Slices³
- F 1527 Guide for Application of Certified Reference Materials and Reference Wafers for Calibration and Control of Instruments for Measuring Resistivity of Silicon³

3. Summary of Test Method

3.1 There are two methods that may be used. They differ in calibration technique, sample measurement value range, data correction techniques, and suitability of instrumentation and are as follows:

Factors	Methods	
	I	II
Calibration	Ascertains linearity 5 samples Sheet or bulk	Ascertains slope 2 samples Sheet or bulk
Application range	Broad: 2 decades	Narrow: $\pm 25\%$
Sample data	Direct reading, then correct for temperature.	Direct reading, then correct for slope.
Suitable instruments	Manual/Automatic	Automatic: computer-controlled

3.2 Method I ascertains the conformance of the apparatus to linearity and slope limits (± 1 digit) over a broad range (2 decades) of calibration standard values. It qualifies apparatus for use over a wide range of sample values.

3.2.1 The apparatus is first calibrated using standards of known resistivity or sheet resistance. Then the apparatus is subjected to a test for linearity that involves measuring a set of five reference specimens. As a part of the linearity test, a plot is made of the indicated values as a function of the known values; two limiting curves are also plotted on the same graph. (If all the plotted points fall within the limit curves, the apparatus is regarded as satisfactory.)

3.2.2 For subsequent measurements of bulk samples, the thickness of each sample (wafer) is measured and entered directly, or by the operator, according to the design of the instrument. The conductance of the specimen is then measured by the apparatus and converted to a resistivity value that is displayed. These measurements are subsequently corrected to 23°C-equivalents.

3.2.3 For subsequent measurement of thin film specimens, the sheet conductance is measured, converted to sheet resistance, and displayed. See 9.1.3 for relations between these quantities.

3.3 Method II assumes instrument linearity between calibration standards whose values are narrowly separated (typically $\pm 25\%$ of the anticipated sample range median point).

3.3.1 The apparatus is first calibrated using standards of known resistivity or sheet resistance. The apparatus is then subjected to a test that quantifies apparatus slope at two points, and provides a means of correcting subsequent sample measurements, for values between these two points, to a calibration line established between the two standards employed. These latter standards may be of either known bulk resistivity or sheet resistance; their material must be the same electrical type as the samples to be measured (see Note 8).

3.3.2 For subsequent measurements of bulk samples, the thickness of each sample (wafer) is measured and entered directly, or by the operator, according to the design of the instrument. These measurements are subsequently referred to the standard reference plot. The corrected values are known to a greater precision than those obtained following Method I, and in most instances are also 23°C-equivalents.

⁵ Annual Book of ASTM Standards, Vol 14.03, Annual Book of ASTM Standards, Vol 06.01, ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Telephone: 610-832-9500, Fax: 610-832-9555, Website: www.astm.org

⁶ Annual Book of ASTM Standards, Vol 10.05.

3.3.3 For subsequent measurement of thin film specimens, the sheet conductance is measured, converted to sheet resistance and displayed. See 9.1.3 for relations between these quantities. These measurements are subsequently referred to the standard reference plot.

4. Significance and Use

4.1 Resistivity is a primary quantity for characterization and specification of material used for semiconductor electronic devices. Sheet resistance is a primary quantity for characterization, specification, and monitoring of thin film fabrication processes.

4.2 This test method requires no specimen preparation.

4.3 Method II is particularly well suited to computer-based systems where all measurements can be quickly and automatically corrected for value offset and for temperature coefficient of resistivity.

4.4 Method I has been evaluated by interlab comparison (see Section 11). Until Method II has been evaluated by interlaboratory comparison, it is not recommended that the test method be used in connection with decisions between buyers and sellers.

5. Interferences

5.1 Radial resistivity variations or other resistivity nonuniformity under the transducer are averaged by this test method in a manner which may be different from that of other types of resistivity or sheet resistance techniques which are responsive to a finite lateral area. The results may therefore differ from those of four-probe measurements depending on dopant density fluctuation and the four-probe spacing used.

NOTE 1—Test Method F 81 provides a means for measuring radial resistivity variation of silicon slices.

5.2 Uncertainty of thickness values for the reference (bulk) wafers can introduce, in Method I, an uncertainty in the linearity plot; in Method II it can introduce a corresponding uncertainty in the reference line connecting the two reference wafer values, if calibration is performed in resistivity values.

5.3 Uncertainty of thickness value of sample (bulk) wafers can introduce an error in both measured and reported values for both Methods I and II. These uncertainties can be eliminated by executing the procedure using sheet resistance values for reference and sample wafers.

5.4 Spurious currents can be introduced in the test equipment when it is located near high-frequency generators. If equipment is located near such sources, adequate shielding must be provided. Power line filtering may also be required.

5.5 Semiconductors have a significant temperature coefficient of resistivity. Temperature-correction factors for extrinsic silicon specimens are given in Test Method F 84. Temperature differences between any of the reference or sample wafers, during calibration or measurement, or both, will introduce a measurement error in Method II.

5.6 High levels of humidity may affect the indicated value.

6. Apparatus

6.1 *Electrical Measuring Apparatus*, with instructions for use and consisting of the following assemblies:

6.1.1 *Eddy-Current Sensor Assembly*, having a configuration of a fixed gap, between two opposed transducers, into which a specimen slice is inserted. The assembly shall include support(s) on which the slice rests, a device for centering the slice, and a high-frequency oscillator to excite the sensing elements. The frequency of the oscillator shall be chosen to provide a skin depth at least five times the thickness of the slice or thin film to be measured. The skin depth is a function of the resistivity of the specimen. The assembly shall provide an output signal proportional to sheet conductance. This assembly and associated apparatus are shown schematically in Fig. 1.

NOTE 2—A typical conductance apparatus is described in detail in a paper by Miller, Robinson, and Wiley.⁷ This paper also discusses skin-depth as a function of thickness and resistivity.

6.1.2 *Signal Processor*—Means for electronically converting, by analog or digital circuitry, the sheet conductance signal to a sheet resistance value, and in the case of bulk substrate measurements, means for conversion to resistivity values using the measured thickness of the substrate. The processor shall incorporate a means for displaying sheet resistance or resistivity, a means of zeroing the conductance signal in the absence of a specimen and a means for calibrating the instrument with known calibration specimens.

⁷ Miller, G. L., Robinson, D. A. H., and Wiley, J. D., "Contactless Measurement of Semiconductor Conductivity by Radio Frequency-Free-Carrier Power Absorption," *Review of Scientific Instruments*, Vol 47, No. 7, July 1976.

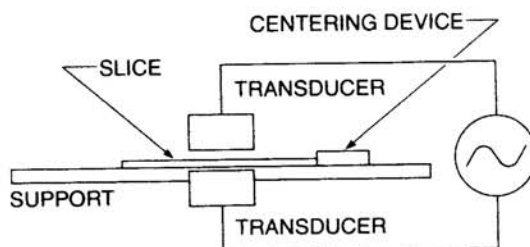


FIG. 1 Schematic of Eddy-Current Sensor Assembly

NOTE 3—For Method I, the linearity of the apparatus is checked in an operational qualification test (see 9.1.3).

NOTE 4—A typical apparatus operates as follows. When a specimen is inserted into the fixed gap between the two colinear sensing elements, or transducers, in a special oscillator circuit, eddy currents are induced in the specimen by the alternating field between the transducers. The current needed to maintain a constant voltage in the oscillator is determined internally; this current is a function of the specimen conductance. The specimen conductance is obtained by monitoring this current. Sheet resistance or resistivity values are obtained from the specimen conductance by analog or digital electronic means; calculation of resistivity values also requires knowledge of specimen thickness.

6.2 *Thermometer* — ASTM Precision Thermometer having a range from -8 to $+32^{\circ}\text{C}$ and conforming to the requirements for Thermometer 63C as specified in Specification E 1.

6.3 *Thickness Gage*, — as specified in 7.1 of Test Method F 533 (included for completeness).

6.4 Calibration and linearity checking must be done in consistent units, whether resistivity, sheet resistance or sheet conductance, according to the requirements of the given instrument. If resistivity values are used, knowledge of the specimen thickness is also required. For bulk calibration or linearity-check specimens, the thickness is the as-measured thickness in centimetres. For thin film specimens, the total thickness of the thin film plus substrate should be measured and used; if this cannot be done, an effective thickness of 0.0508 cm (0.020 in.) should be used. (See 9.1.3.)

7. Reagents and Materials

7.1 Resistivity standards or other reference specimens to check the accuracy and linearity of the instrument. Preferably, these are bulk silicon slices but may also be fabricated by ion implantation into silicon.

7.1.1 Bulk silicon standards or other reference specimens are to be measured for resistivity in accordance with Test Method F 84. The thickness of these specimens shall be within $\pm 25\%$ of the specimens to be measured unless otherwise agreed to by the parties to the test.

7.1.2 Ion implant specimens are to be measured for sheet resistance by four point probe in accordance with Test Method F 374.

7.1.3 The standards and other reference specimens for Method I shall be at least five in number and should have a range of values that span the full range of the instrument. For Method II, where the specimens to be measured have a narrow range of resistivity or sheet resistance values, the standards and other reference specimens shall be two in number. Their values shall span a range at least as large as the specimens to be measured. Table 1 gives a list of values recommended for checking the full range of a typical instrument for Method I application; these values should be met within $\pm 25\%$.

NOTE 5—Resistivity standards are available from the National Bureau of Standards in the form of bulk silicon slices with nominal thicknesses of 0.025 in. at the following resistivity levels: 0.01, 0.1, 1, 10, 25, 75 and $180\ \Omega\cdot\text{cm}$.

NOTE 6—Ion implanted sheet resistance reference specimens may be used subject to the requirement that the sheet resistance of the substrate is at least $1000 \times$ the sheet resistance of the implanted layer.

NOTE 7—It is possible to load the oscillator of the instrument by using a bulk specimen which has too much conductance. This may be caused by a combination of low resistivity and large thickness. Such loading of the oscillator will make it generally impossible to simultaneously meet the linearity requirements of Section 9 for this specimen and the less conductive reference specimens being used.

8. Sampling

8.1 If the test method is not used as a 100% inspection test, sampling procedures shall be agreed upon by the parties to the test.

8.2 If sampling by lot is required, the determination of what constitutes a lot and the procedures for sampling by lot shall be agreed upon by the parties to the test.

TABLE 1 Resistivity Values for Apparatus Qualification Test

Section I		Section II
For Test-Equipment Measurement Range, $\Omega\text{-cm}$	Use These Reference Specimens, $\Omega\text{-cm}$	Implanted Specimen Sheet Resistance Equivalent to a 50.8- μm (0.020-in.) Thick Bulk Resistivity Specimen, $\Omega/\square$
0.001–0.999	0.01	0.2
	0.03	0.6
	0.10	2
	0.30	6
	0.90	16
0.1–99.9	0.9	18
	3	60
	10	200
	30	600
	90	1800

9. Method I (Full Range)

9.1 Calibration:

9.1.1 With the thermometer, measure the room ambient temperature, T , to the nearest 0.1°C and record this value.

9.1.2 If bulk silicon resistivity standards or reference specimens are used, or both, calculate in accordance with the following equation, the resistivity, ρ_T , at the ambient temperature. (Proceed to 9.3 if implanted reference specimens are used.)

$$\rho_T = \rho_{23}(1 + C_T(T - 23))$$

where:

- ρ_{23} = is the resistivity at 23°C , and
 C_T = silicon temperature coefficient of resistivity (Test Method F 84, Table 5).

9.1.3 Convert the values of the standards or reference specimens, or both, to units of sheet resistance, resistivity, conductivity; or conductance as necessary according to the calibration section of the instrument's instruction manual, using the following relation:

$$R = \rho / t = 1 / \sigma = 1 / G$$

where:

- R = sheet resistance,
 ρ = resistivity
 σ = conductivity
 G = conductance, and
 t = thickness of specimen in centimetres (taken to be 0.0508 cm for the thin films).

9.1.4 Test the apparatus linearity as follows:

9.1.4.1 Position the specimen between the transducers so that the center of the specimen is within 1 mm (0.04 in.) of the axis of the transducer assembly.

9.1.4.2 With the apparatus, measure the resistivity of each of the five reference specimens in accordance with the instructions provided by the apparatus manufacturer. Record these values.

9.1.4.3 Plot each specimen's measured values, in whatever units are chosen, against the values, in the same units, obtained from four probe measurements where the four probe values have been corrected to ambient temperature in the case of bulk specimens. (See Fig. 2 for examples of plots in units of resistivity and of conductance.)

9.1.4.4 Construct two curves corresponding to the following relations:

$$\begin{aligned} \text{indicated value} &= \text{known value} + 5\% \text{ of the known value} + 1 \text{ digit} \\ \text{indicated value} &= \text{known value} - 5\% \text{ of the known value} - 1 \text{ digit} \end{aligned}$$

9.1.4.5 Examine the plot. If all five points fall between the two curves, proceed to 9.2. If all five points do not fall between the two curves, refer to the manufacturer's instructions and ensure that all adjustments to the electrical measuring apparatus have been properly made, and repeat 9.1.4.1 through 9.1.4.5. If only three or four adjacent points fall within the

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two curves, the instrument may be used over the inclusive range bounded by the upper and lower values of these adjacent points. If fewer than three adjacent points fall within the two curves, discontinue the test, as the apparatus does not satisfy the linearity requirement.

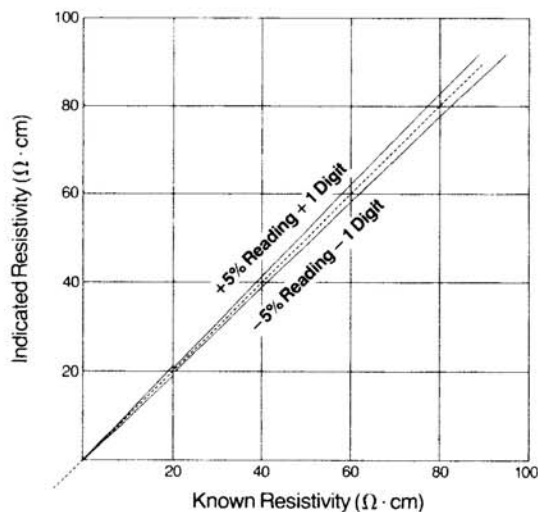


FIG. 2 Linearity Check Plot

9.2 Procedure:

9.2.1 With the thermometer, measure the room ambient temperature to the nearest 0.1°C and record this value.

9.2.2 If bulk specimens are being measured, first measure and record specimen centerpoint thickness in accordance with Test Method F 533. Omit this step if thin film specimens are being measured.

9.2.3 Enter the thickness value into the apparatus in accordance with the instructions provided by the apparatus manufacturer if measuring bulk specimens; if measuring thin film sheet resistance, follow the instructions provided by the instrument manufacturer for entering a normalizing thickness or using a mode switch to select an output in units of sheet resistance.

9.2.4 With the apparatus, measure the resistivity or sheet resistance of each specimen slice in accordance with the instructions provided by the apparatus manufacturer. Record these values.

9.2.4.1 Position each slice between the transducers so that the center of the slice is within 1 mm (0.04 in.) of the axis of the transducer assembly.

9.2.4.2 If bulk specimens are being measured, correct the measured resistivity values of each specimen to 23°C equivalent in accordance with Test Method F 84. Record these values. If the specimen is a thin film or is bulk gallium arsenide, omit this step.

9.3 Report:

9.3.1 Report the following information:

9.3.1.1 Date of test,

9.3.1.2 Identification of operator,

9.3.1.3 Identification of resistivity standards and reference specimens (7.1),

9.3.1.4 Identification of specimen,

9.3.1.5 Ambient temperature during calibration, °C, and, for each slice,

(a) Ambient temperature during measurement, °C,

(b) Thickness, cm (or in.),

(c) Measured resistivity, Ω·cm (room temperature), and

(d) Corrected resistivity, Ω·cm (23°C equivalent).

10. Method II (Partial Range)

10.1 Calibration:

10.1.1 With the thermometer, measure the room ambient temperature, T , to the nearest 0.1°C and record this value.

10.1.2 Select two reference specimens whose values span the range of samples to be measured.

10.1.3 Position the specimen between the transducers so that the center of the specimen is within 1 mm (0.04 in.) of the axis of the transducer assembly.

10.1.4 With the apparatus, measure the resistivity of the two specimens in accordance with the instructions provided by the manufacturer. Record these values.

10.1.5 Plot the specimen's ambient-measured values on a graph as a function of their 23°C-certified values (see Fig. 3, Fig. 4).

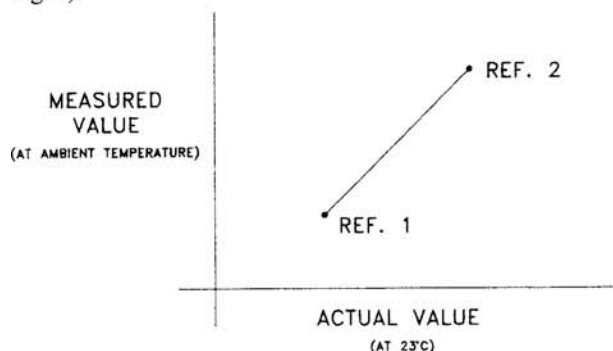


FIG. 3 Reference Line Construct

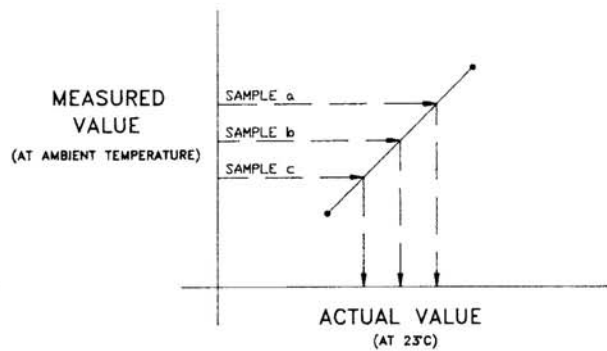


FIG. 4 Sample Value Construct

10.1.6 Construct a line connecting the two data points.

10.2 Procedure:

10.2.1 If bulk specimens are being measured, locate the samples' measured values on the graph's ordinate, and extend a horizontal line from the measured value to its intersection with the calibration line constructed in 10.1.6. Extend a vertical line from above the calibration intersect point to the actual abscissa (see Fig. 4). Record the abscissa intersect point as the sample's actual value.

10.2.1.1 For calibration specimens and samples with resistivity above 0.1 $\Omega\cdot\text{cm}$, the temperature coefficient of resistivity of silicon is monotonic and changes gradually with increasing resistivity (see Test Method F 84, Table 5). Procedure II produces for these samples actual values automatically corrected to 23°C.

10.2.1.2 For calibration specimens spanning a resistivity range that contains a non-monotonic temperature coefficient of resistivity, the measured values should be corrected to their 23°C-equivalents using Test Method F 84, Table 5 factors, prior to locating the measured values on the graph ordinate. The actual correction values in this range are extremely small.

NOTE 8—The maximum error derived from calibrating on one conductivity type and measuring the other [is] $<0.12\%/^{\circ}\text{C}$ in the resistivity range 0.01 $\Omega\cdot\text{cm}$ to 1000 $\Omega\cdot\text{cm}$.

10.3 Report:

10.3.1 Report the following information:

10.3.1.1 Date of test,

10.3.1.2 Identification of operator,

10.3.1.3 Identification of resistivity standards and reference specimens (7.1),

10.3.1.4 Identification of specimen,

10.3.1.5 Ambient temperature during calibration, $^{\circ}\text{C}$, and, for each slice,

(a) Ambient temperature during measurement, $^{\circ}\text{C}$,

(b) Thickness, cm (or in.),

(c) Measured resistivity, $\Omega\cdot\text{cm}$ (room temperature), and

(d) Corrected resistivity, $\Omega\cdot\text{cm}$ (23°C equivalent).

11. Precision and Bias

11.1 Method I precision has been determined by round-robin test (see Annex A1).

11.1.1 The first interlaboratory test used to estimate multilaboratory precision, or reproducibility, of this test method is detailed in Annex A1. It used three large-grain polycrystalline silicon specimens of very similar resistivity as the test specimens and ten single-crystal silicon-reference specimens to check instrument linearity. The two lowest-resistivity value reference specimens are strictly applicable only to low-range eddy current instruments and are not necessary to qualify instruments for use on the polycrystal test specimens. While none of the five participating laboratories successfully passed the linearity test, within $\pm 5\%$ limits, for all reference specimens, an analysis was made based on assumptions detailed in A1.4

and A1.5. Based on these assumptions, an estimate of multilaboratory reproducibility of the average of four measurements of the polycrystalline specimens is $\pm 12\%$ (3S%).

11.1.2 A second multilaboratory test was conducted to estimate multilaboratory reproducibility of this test method for single crystal silicon specimens over a wide range of resistivity values. This test is detailed in Annex A2. Owing to the large number of test specimens being evaluated, only three reference specimens were used for linearity check of low range instruments, and four reference specimens were used to check high range instruments. Collectively, these reference specimens covered the range 0.003 to 30 $\Omega\cdot\text{cm}$. For laboratories demonstrating instrument linearity of $\leq 5\%$ or better, the estimate of multilaboratory reproducibility over this resistivity range is $\pm 9\%$ (3S%). For the extrapolated test specimen range to 90 $\Omega\cdot\text{cm}$, the estimate of reproducibility is $\pm 15\%$ (3S%) and from 90 to 12 $\Omega\cdot\text{cm}$, it is $\pm 18\%$ (3S%).

11.1.3 *Bias*—Estimates of bias for this test method can only be established with respect to a resistivity scale determined by another technique with comparable spatial averaging over material nonuniformities, such as the four-probe technique, Test Method F 84. However, eddy current instruments are calibrated and checked for linearity of response using specimen resistivity values determined by four-probe measurements. Therefore, within the ability to establish response to $\pm 5\%$ of reference value over a large range of resistivity values, as was demonstrated by a number of the participating laboratories, no additional bias is expected.

11.2 It is planned to determine the precision of Method II by round-robin test.

12. Keywords

12.1 contactless measurements; eddy current; nondestructive evaluation; resistivity; semiconductor; sheet resistance; silicon; thin films; wafer

ANNEXES

(Mandatory Information)

A1. RESULTS OF FIRST MULTILABORATORY TEST FOR METHOD I

A1.1 A preliminary estimate of precision was made by five laboratories taking measurements on nine single crystal and three large-grain polycrystal specimens. The single crystal specimens were measured for thickness and for resistivity at the center using a four point probe (Test Method F 84) by the laboratory which donated the specimens and also by the laboratory which coordinated the round robin. The polycrystal specimens were measured for thickness only. A summary of this data is given in Table A1.1. Thickness and resistivity values as measured by the originating laboratory were provided to all participants as were temperature coefficient of resistivity values for the single crystal specimens.

TABLE A1.1 Thickness and Four-Probe Resistivity Values as Taken by Supervisory Labs

Measurement	Single Crystal Linearity-Test Specimens											
	-4	-17	-8	-12	-58	-6	-9	-56	-54	-2	0	P
Center Thickness (μm) Supervisory Lab 1	525.3	523.0	501.9	503.7	505.5	512.1	505.7	522.5	491.7	512.5	406.9	407.7
Center Thickness (μm) Supervisory Lab 2	524.8	522.2	501.0	503.0	504.0	511.2	504.6	522.1	491.2	512.3	406.0	406.5
Center Thickness (μm) Supervisory Lab 2 (2 months later)	524.4	522.2	501.5	broken	504.4	511.5	504.8	522.2	491.6	512.4	406.8	409.5
Worst Case Thickness Difference Lab 1 versus Lab 2	0.1%	0.15%	0.18%	0.14%	0.30%	0.18%	0.22%	0.08%	0.10%	0.04%	...	...
FPP Resistivity ($\Omega\cdot\text{cm}$) Supervisory Lab 1	0.0028	0.00834	0.1096	0.4802	0.8371	7.999	11.52	20.12	30.23	133.7	...	...
FPP Resistivity ($\Omega\cdot\text{cm}$) Supervisory Lab 2	0.00270	0.00835	0.1106	broken	0.8302	7.992	11.09	19.55	30.23	114.7	...	...
+ 5 % Limits Based on Supervisory Lab 1	0.00266	0.00792	0.1041	0.4562	0.7952	7.599	10.94	19.11	28.72	127.0	...	...
Resistivity	0.00294	0.00876	0.1151	0.5042	0.8790	8.8399	12.10	21.13	31.74	140.4	...	...
+ 5 % Limits Based on Supervisory Lab 2	0.00256	0.00793	0.1051	...	0.7887	7.592	10.54	18.57	28.72	109.0	...	...
Resistivity	0.00284	0.00877	0.1166	...	0.8717	8.392	11.64	20.53	31.74	120.4	...	...

A1.2 The participating laboratories were requested to calibrate and test the linearity of the contactless resistivity instruments using the single crystal specimens and to measure the polycrystal specimens as unknown. All specimens were measured on each of 3 days. Each day, the single crystal specimens were measured four times at the center with the polished surface face up, then four times with the polished surface face down; the specimens were rotated 90° about a vertical axis between each of the four measurements. The polycrystal specimens were measured each day with one face up only; four measurements were taken on each specimen with 90° specimen rotations between the measurements. A summary of the data is given in Table A1.2.

A1.3 Differences between the measurement averages for the two face-up conditions of the single crystal specimens are generally less than 1% and for almost all combinations of specimens and laboratories, they are smaller than the standard deviation of a set of four measurements for that specimen-laboratory combination.

A1.4 Tests of instrument linearity appear to show two kinds of results, one related to specimens and one related to instruments. The largest number of measurements which are outside of 5% linearity limits based on four probe

measurements occur for the two lowest and the very highest resistivity specimens, with a very large number of such measurements also occurring for the 20 $\Omega\cdot\text{cm}$ specimen. Since the specimen just below and just above 20 $\Omega\cdot\text{cm}$ have very few measurements reported outside of such $\pm 5\%$ limits, the large number of poor measurements on the 20 $\Omega\cdot\text{cm}$ specimen are likely due to some property of the specimen, such as resistivity non-uniformity near the center, rather than malfunction of the instruments used. Laboratory 5 (see Table A1.2) shows a very high number of measurements outside the 5% linearity limits, suggesting particular instrument problems.

TABLE A1.2 Summary of Measurements on Linearity Test Specimens and on Unknown Test Specimens

Note: 1. Data entries are averages of four "face-up" measurements and four "face-down" measurements on each of three days.

Lab		Day	Single Crystal Linearity-Test Specimens								Polycrystal Unknowns				
			-4	-17	-8	-12	-58	-6	-9	-56	-54	-2	0	P	Q
1	1	0.002655 ^A	0.00824	0.1116	0.4838	0.8300	8.361	11.16	20.38	29.97	...	5.165	4.392	5.60	
		0.002650 ^A	0.00826	0.11195	0.4858	0.8356	8.415 ^{A, H}	11.21	20.45	29.98	...	...	...	...	
		0.00259 ^A	0.00807	0.1096	0.4764	0.8190	8.026	11.69 ^A	20.96 ^B	30.75	...	5.116	4.305	5.62	
	3	0.00259 ^A	0.00808	0.1096	0.4766	0.8208	8.056	11.72 ^A	20.99 ^B	30.91	...	...	...	...	
		0.00261 ^A	0.00822	0.1111	0.4832	0.8272	7.838	11.00	21.25 ^{A, B}	31.04	...	5.08	4.30	5.53	
		0.00261 ^A	0.00822	0.1110	0.4826	0.8350	7.878	11.01	21.15 ^{A, H}	31.08	...	...	...	...	
2	1	0.00286 ^B	0.00872	0.1141	0.4759	0.8257	7.916	10.93 ^A	20.38	29.39	...	5.097	4.155	5.431	
		0.00284	0.00860	0.1141	0.4767	0.8267	7.945	10.93 ^A	20.48	29.42	...	...	...	...	
	2	0.00286 ^B	0.00846	0.1129	0.4785	0.8301	7.921	10.97	20.63 ^B	29.59	...	5.18	4.15	5.50	
		0.00286 ^B	0.00843	0.1126	0.4772	0.8289	7.933	10.97	20.51	29.64	...	...	...	...	
	3	0.00282	0.00828	0.1118	0.4776	0.8303	7.921	11.04	20.52	29.52	...	5.26	4.17	5.55	
		0.00282	0.00825	0.1113	0.4778	0.8271	7.958	11.01	20.47	29.52	...	...	...	...	
3	1	0.002705	0.008304	0.1094	0.4785	0.8245	8.129	11.35	20.93 ^B	30.6	114.1 ^A	6.23	4.56	5.94	
		0.002705	0.008304	0.1094	0.4790	0.8250	8.127	11.35	20.95 ^B	30.6	111.9 ^A	...	...	...	
	2	0.002705	0.008304	0.1094	0.4763	0.8230	8.169	11.44	21.25 ^{A, B}	30.8	115.6 ^A	6.21	4.77	6.30	
		0.002705	0.008304	0.1095	0.4770	0.8220	8.141	11.40	21.00 ^B	30.75	114.9 ^A	...	...	...	
	3	0.002607 ^A	0.008206	0.1094	0.4888	0.8750	8.192	11.47	21.25 ^{A, B}	30.6	114.0 ^A	6.39	4.78	6.25	
		0.002607 ^A	0.008206	0.1093	0.4885	0.8740	8.187	11.45	21.25 ^{A, B}	30.53	114.4 ^A	...	...	...	
4	1	0.003005 ^{A, B}	0.008895 ^{A, B}	0.1143	0.4901	0.8495	8.202	11.48	21.19 ^{A, B}	30.77	117.4 ^A	5.155	4.299	5.645	
		0.00301 ^{A, H}	0.008903 ^{A, B}	0.1142	0.4901	0.8525	8.210	11.47	21.02	30.85	119.4 ^A	...	...	...	
	2	0.002934 ^B	0.008833 ^{A, B}	0.1146	0.4960	0.8564	8.210	11.448	21.11	30.33	112.2 ^A	5.111	4.175	5.589	
		0.002934 ^B	0.008833 ^{A, B}	0.1146	0.4992	0.8558	8.301	11.43	21.15 ^{A, B}	30.44	113.4 ^A	...	...	...	
	3	0.002934 ^B	0.008833 ^{A, B}	0.1145	0.4910	0.8512	8.218	11.448	21.19 ^{A, B}	30.77	117.4 ^A	5.113	4.268	5.645	
		0.002934 ^B	0.008833 ^{A, B}	0.1145	0.4967	0.8524	8.248	11.486	21.02 ^B	30.85	119.4 ^A	...	...	...	
5	1	0.00283	0.00842	0.1058	0.5131 ^A	0.8819 ^{A, B}	8.291	11.38	20.38	31.00	128.1 ^B	5.451	4.668	6.003	
		0.00283	0.00838	0.1055	0.5103 ^A	0.8819 ^{A, B}	8.236	11.38	20.58 ^B	30.90	125.9 ^B	...	...	...	
	2	0.00298 ^{A, H}	0.00900 ^{A, B}	0.1088	0.5143 ^A	0.8820 ^{A, B}	8.375	11.58	21.00 ^B	30.85	125.1 ^B	5.617	4.380	6.180	
		0.00298 ^{A, H}	0.00900 ^{A, B}	0.1098	0.5128 ^A	0.8895 ^{A, B}	8.350	11.65 ^H	20.40	31.18	119.6	...	...	...	
	3	0.00299 ^{A, H}	0.00895 ^{A, B}	0.1093	0.5083 ^A	0.8820 ^{A, B}	8.325	11.53	20.78	31.30	128.7 ^B	5.541	4.578	5.787	
		0.00300 ^{A, H}	0.00895 ^{A, B}	0.1100	0.5095 ^A	0.8895 ^{A, B}	8.450 ^{A, B}	11.50	21.05 ^B	31.25	126.7 ^B	...	...	...	

^A Value is outside $\pm 5\%$ limits based on Supervisory Lab 1 data.

^H Value is outside $\pm 5\%$ limits based on Supervisory Lab 2 data.

A1.5 The polycrystal specimens have measured resistivity values in a region of little instrument nonlinearity as tested with the single crystal specimens. Because of the wide range of temperatures at which data was taken by the different laboratories, a temperature coefficient of resistivity with a value of 0.8% per $^{\circ}\text{C}$ was assumed for these specimens and was applied to calculate the values shown in Table A1.2.

A1.6 The estimate of the multilaboratory precision which was made for the polycrystalline specimens was based on the assumption that each day's measurements by each laboratory was an independent set of data. However, since Laboratory number 3 differed from the other four laboratories regarding the relative values of the specimens, separate data tabulations were done with and without the data from Laboratory 3. Table A1.3 shows the calculated overall averages and relative standard deviations of the three specimens for all five laboratories (15 laboratory-day combinations for each specimen) and for the laboratories other than Laboratory 3 (12 laboratory-day combinations). If data from Laboratory 3 is omitted, a reasonably constant of relative standard deviation is calculated for all three specimens. The estimate of multilaboratory precision for the average of four individual measurements is based on the four laboratory analyses and is taken to be 4%.

TABLE A1.3 Averages ($\Omega\cdot\text{cm}$) and Relative Standard Deviations of Daily Averages

Specimen	O, $\Omega\cdot\text{cm}$ (%)	P, $\Omega\cdot\text{cm}$ (%)	Q, $\Omega\cdot\text{cm}$ (%)
Laboratories 1-5	5.448 (8.47)	4.397 (5.03)	5.771 (5.01)
Omit Laboratory 3	5.240 (3.58)	4.320 (3.83)	5.673 (3.83)

A2. RESULTS OF SECOND MULTILABORATORY TEST FOR METHOD 1

A2.1 This test, conducted in 1986 and 1987, used most of single-side polished bulk silicon slices that were used as reference specimens in the first multilaboratory tests; these can be identified by comparing slice identification numbers for the two experiments. To these were added five double-side lapped slices from the NIST Standard Reference Material series, identifiable by the use of letters *U* through *Y* for slice identification in Table A2.1. Three of the single-side polished slices were replaced due to breakage following Laboratory 7; the replacements have four-digit identification numbers.

A2.1.1 The available slices were divided into two categories: three reference (linearity-check) specimens plus five test specimens to test the resistivity range 0.002 to 1 Ω -cm, and four reference specimens plus nine test specimens to test the resistivity range 0.1 to 125 Ω -cm.

A2.2 A summary of the data taken by the supervisory laboratory for the slices used in the low resistivity range test is given in Table A2.1. This table shows the slice identification, conductivity-type, thickness, in micrometers, measured by an electromechanical contacting gage, temperature coefficient of resistivity, and resistivity at 23°C determined by four probe, as well as the $\pm 5\%$ and -5% limits on these resistivity values allowed by the linearity requirements of the test method. Table A2.2 gives corresponding values for the slices used in the high resistivity range test. Thickness values for all slices were provided to the participating laboratories. They were, however, allowed to use in situ measurement of thickness if their instruments took such data. Temperature coefficients of resistivity values were provided only for the reference specimens. No prequalification of the reference or test specimens was performed beyond the thickness and center point resistivity measurements given in Table A2.1 and Table A2.2.

TABLE A2.1 Low Resistivity Range Specimens: Thickness and Four-Probe Resistivity Information Taken by the Coordinating Laboratory

Slice Identification Conductivity Type	Reference Slices					Test Slices				
	4 <i>N</i>	1928 <i>N</i>	8 <i>P</i>	58 <i>P</i>	17 <i>P</i>	<i>T</i> <i>P</i>	<i>U</i> <i>P</i>	43 <i>N</i>	2051 <i>N</i>	<i>V</i> <i>P</i>
Thickness (μm)	524.6	587.0	501.2	504.2	522.2	633.4	636.0	372.6	601.0	506.9
ρ_{23}	0.0027	0.00302	0.1106	0.830	0.00835	0.0144	0.09816	0.4286	0.407	0.8186
$\rho_{23} + 5\%$	0.00284	0.00317	0.1161	0.872	0.00877	0.0151	0.1030	0.4500	0.427	0.8595
$\rho_{23} - 5\%$	0.00257	0.00286	0.1051	0.789	0.00793	0.0136	0.0933	0.4072	0.386	0.7777

TABLE A2.2 High Resistivity Range Specimens: Thickness and Four Probe Resistivity Information Taken by the Coordinating Laboratory

Reference Slices														
Slice Identification	8													
Conductivity Type	P													
Thickness (μm)	501.2													
ρ_{23}	0.1106													
$\rho_{23} + 5\%$	0.1161													
$\rho_{23} - 5\%$	0.1051													
Test Slices														
Slice Identification	U	43	2051	V	6	W	56	1055	X	Y	Z			
Conductivity Type	P	N	N	P	N	N	N	P	N	N	N			
Thickness (μm)	636.0	372.6	601.0	506.9	511.2	565.7	522.1	605.0	633.4	603.5	512.3			
ρ_{23}	0.0982	0.4286	0.407	0.8186	7.992	10.51	19.55	21.1	26.38	74.53	114.7			
$\rho_{23} + 5\%$	0.1031	0.4500	0.427	0.8595	8.391	11.08	20.53	22.2	27.91	78.25	108.9			
$\rho_{23} - 5\%$	0.0982	0.4072	0.386	0.7777	7.592	10.02	18.57	20.0	25.25	70.80	120.4			

A2.3 Each laboratory was required to establish the linearity of its instrument, using the reference specimens provided, on each of 3 days, and to take four measurements at the center of each test sample on each day. The averages of those four measurements are reported in the following tables: Table A2.3—low resistivity range for Laboratories 1 through 7, Table A2.4—low resistivity range for Laboratories 8 through 11, Table A2.5—high resistivity range for Laboratories 1 through 7, and Table A2.6—high resistivity range for Laboratories 8 through 11. All these values have been converted to 23°C values for comparison with four-probe values.

A2.4 Observations on reported data not contained in previous tables.

A2.4.1 Specimen thickness values provided by the coordinating laboratory were used by Laboratories 1, 10, and 11; all other laboratories reported thickness values on the data sheets that indicated they had taken their own measurements. The worst case percent offset in these thickness values compared to the reference values, was noted for each of these laboratories; these percent differences ranged from 0.3% for Laboratories 7 and 8 to 1.5% for Laboratories 3 and 5; Laboratory 9 has a worst case offset of 0.7% for low resistivity range specimens but had a 4% offset for several high range specimens. There was no meaningful difference between single-side polished and double side lapped specimens in terms of sign or magnitude of the thickness offsets.

TABLE A2.3 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of Low Range Specimens, Laboratories 1 through 7

Laboratory Number	Slice Identification				Test			
	4	8	58	17	T	U	43	V
1	0.00264	0.1098	0.921	0.00820	0.01401	0.0969	0.4562	0.9084
	0.00260	0.1082	0.916	0.00810	0.01389	0.0956	0.4451	0.9016
	0.00278	0.1158	0.975	0.00858	0.01472	0.1019	0.4840	0.9801
2	0.00270	0.114	0.837	0.0084	0.0142	0.100	0.446	0.839
	0.00260	0.109	0.816	0.0080	0.0137	0.096	0.424	0.799
	0.00269	0.113	0.836	0.0085	0.0142	0.100	0.435	0.856
3	0.0026	0.1105	0.809	0.0082	0.0138	0.0967	0.420	0.814
	0.0026	0.1102	0.788	0.0082	0.0140	0.0962	0.409	0.788
	0.0028	0.1120	0.880	0.0086	0.0140	0.0971	0.436	0.884
4	0.00267	0.113	0.816	0.00837	0.0142	0.0990	0.413	0.802
	0.00269	0.113	0.815	0.00841	0.0142	0.0985	0.413	0.804
	0.00268	0.110	0.816	0.00838	0.0142	0.0993	0.414	0.804
5	0.00267	0.115	0.844	0.00881	0.0149	0.110	0.445	0.838
	0.00265	0.115	0.845	0.00900	0.0148	0.114	0.444	0.848
6	---	0.13	0.91	0.091	0.10	0.12	0.46	0.90
	---	0.13	0.90	0.09	0.10	0.12	0.47	0.91
	---	0.13	0.90	0.09	0.10	0.12	0.49	0.91
7	0.003	0.12	0.84	0.008	0.014	0.11	0.44	0.85
	0.003	0.12	0.85	0.008	0.014	0.11	0.43	0.856
	0.0028	0.12	0.85	0.0075	0.014	0.115	0.440	0.85

A2.4.2 Temperature correction of resistivity value calculations were checked for low and high range reference specimens from all laboratories based on their stated ambient temperatures during measurement and their stated ambient temperature resistivity values for these specimens. All laboratories except 3, 7, and 10 clearly performed this step to within round off error amounting to less than 0.1%. Laboratory 3 listed the ambient temperature for all measurements, but provided no record of ambient temperature resistivity values used for the reference specimens. Laboratory 7 made temperature corrections in the wrong direction, but operated close enough to 23°C that the maximum error from this source was 1.4%. Laboratory 10 made temperature correction errors of up to 5% but without a consistent pattern in magnitude or direction; the worst of these errors was for reference specimens above 0.8 Ω·cm.

TABLE A2.4 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of Low Range Specimens, Laboratories 8 through 11

Laboratory Number	Slice Identification				Test			
	1928	8	58	17	T	U	2051	V
8	0.00300	0.1104	0.827	0.00830	0.01420	0.09734	0.410	0.8243
	0.00299	0.1105	0.828	0.00839	0.01419	0.09753	0.412	0.8291
	0.00299	0.1106	0.829	0.00839	0.01419	0.09742	0.412	0.8280
9	0.00293	0.105	---	0.0084	0.0143	0.096	0.334	---
	0.00289	0.106	---	0.0083	0.0143	0.096	0.327	---
	0.00290	0.106	---	0.0083	0.0143	0.096	0.343	---
10	0.0030	0.1097	0.870	0.0081	0.0139	0.0985	0.415	0.861
	0.0030	0.1096	0.865	0.0083	0.0139	0.0964	0.415	0.861
	0.0030	0.1099	0.869	0.0082	0.0140	0.0967	0.416	0.871
11	0.0030	0.1120	0.8260	0.0084	0.0143	0.0984	0.4041	0.8138
	0.0030	0.1122	0.8333	0.0085	0.0144	0.0997	0.4104	0.8281
	0.0030	0.1125	0.8365	0.0085	0.0144	0.0994	0.4139	0.8326

A2.4.3 The most common problems with linearity performance, that is, staying within ±5% of the resistivity value obtained by four-probe measurement values was noted at the high end of the low resistivity range and at the low end of the high resistivity range.

A2.5 Two analyses of these data were performed, the first of which provides the basis for the precision statement in 11.1.1. Each is based on the average of four readings taken for each day's instrument setup but with each day's average taken as a separate entry since the setups for each day are assumed to be independent.

TABLE A2.5 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of High Range Specimens, Laboratories 1 through 7

Laboratory Number	Reference				Slice Identification			Test				
	8	58	9	54	U	43	V	6	W	56	X	Y 2
1	0.1099	0.825	11.31	30.46	0.1000	0.4253	0.8358	8.034	10.73	20.81	27.61	80.01
	0.1100	0.819	11.14	30.16	0.1013	0.4190	0.8257	7.951	10.73	20.46	27.17	77.05
	0.1104	0.828	11.37	30.72	0.0986	0.4278	0.8331	8.057	10.73	20.83	27.49	79.12
2	0.112	0.813	11.1	29.2	0.102	0.419	0.803	7.82	10.4	19.8	26.0	70.6
	0.110	0.806	11.0	29.2	0.101	0.418	0.798	7.76	10.3	20.2	26.2	73.6
	0.113	0.820	11.1	29.0	0.102	0.426	0.812	7.88	10.3	20.6	26.2	70.6
3	0.1181	0.789	10.70	27.8	0.108	0.410	0.799	7.58	10.06	18.38	25.6	73.0
	0.1178	0.794	10.76	28.1	0.108	0.408	0.804	7.59	10.12	19.6	26.0	79.1
	0.1182	0.809	10.49	26.2	0.108	0.410	0.810	7.50	9.90	18.0	24.4	62.5
4	0.112	0.824	11.1	29.3	0.099	0.419	0.814	7.82	10.3	20.9	26.1	74.5
	0.109	0.815	10.9	29.6	0.097	0.414	0.807	7.87	10.1	20.6	26.0	75.4
	0.110	0.817	10.9	29.9	0.097	0.414	0.807	7.83	10.1	20.4	26.3	71.7
5	...	...	11.6	32.4	...	...	...	8.48	10.7	22.1	27.7	84.0
	...	...	11.5	32.2	...	...	...	8.40	10.8	22.4	28.3	80.6
	...	...	...	...	...	...	...	...	...	...	...	...
6	0.13	0.90	11.68	28.8	0.12	0.46	0.91	8.45	11.11	20.9	26.9	85.7
	0.13	0.91	11.68	29.2	0.12	0.46	0.92	8.45	11.06	21.0	27.2	85.5
	0.13	0.90	11.64	28.9	0.12	0.46	0.92	8.44	11.07	20.9	26.9	85.6
7	0.12	0.843	11.19	30.05	0.11	0.435	0.85	8.05	10.7*	18.36	27.3	77.8
	0.125	0.842	11.15	30.5	0.11	0.43	0.85	8.06	10.68	19.62	27.5	77.4
	0.12	0.843	11.16	30.3	0.11	0.43	0.85	8.07	10.70	18.95	27.5	74.8

A2.5.1 Laboratories Passing the $\pm 5\%$ Linearity Requirement for All Reference Specimens On the Appropriate Instrument Range:

A2.5.1.1 Table A2.7 gives the grand average, percent standard deviation and number of contributing measurements for the low range specimens; a pooled standard deviation based on degrees of freedom is also reported for Specimens 43 and 2051. Table A2.8 gives the same parameters for the high resistivity range specimens with pooled values of standard deviation being reported for Specimen Pairs 43 and 2051 and for 56 and 1055. Each day's data from a laboratory passing the linearity requirement was taken as a separate contributing measurement under the assumption that each day's setup and qualification of the instrument was independent of the rest. No significant trend of multilaboratory reproducibility of average value with resistivity is seen on either instrument range. Based on this analysis, multilaboratory reproducibility is stated to be better than 9 % (3S %) for specimens up to 30 $\Omega \cdot \text{cm}$, to be <15% (3S%) for specimens between 30 and 90 $\Omega \cdot \text{cm}$ and to be about 18% (3S%) for specimens between 90 and 125 $\Omega \cdot \text{cm}$. The latter two values are derived from specimens well beyond the highest resistivity reference specimen available.

A2.5.2 Laboratories Passing the $\pm 5\%$ Linearity Requirement for Reference Specimens on Either Side of a Test Specimen But Not Necessarily for All Reference Specimens On That Instrument Range:

A2.5.2.1 This analysis generally gave more contributing measurements to the determination of multilaboratory reproducibility values (24 values each on Specimens 17, T, U, and X, and 27 measurements each on Specimens 6 and W). However, estimates of multilaboratory reproducibility values (1S%) were generally several tenths of a percent larger using this procedure rather than that preceding.

TABLE A2.6 Average Resistivity, Corrected to 23°C, for Each Day's Measurement of High Range Specimens, Laboratories 8 through 11

Laboratory Number	Reference				Slice Identification			Test					
	8	58	9	54	U	2051	V	6	W	1055	X	Y	2
8	0.1195	0.824	11.10	30.25	0.1096	0.407	0.8243	7.945	10.38	21.0	26.48	76.01	...
	0.1192	0.830	11.08	29.87	0.1091	0.405	0.8185	7.900	10.37	21.0	26.43	75.89	...
	0.1190	0.819	11.07	29.70	0.1091	0.405	0.8185	7.901	10.35	20.8	26.36	75.55	...
9	...	0.84	11.3	30.9	...	0.406	0.83	8.29	10.6	21.7	27.4	78.7	125.9
	...	0.84	11.2	30.7	...	0.401	0.827	8.24	10.5	21.6	27.2	78.7	126.5
	...	0.84	11.2	30.7	...	0.403	0.84	8.18	10.5	21.4	27.0	78.3	126.4
10	0.12	0.85	11.20	28.8	0.11	0.42	0.84	8.01	10.47	21.0	26.5	69.2	99.8
	0.12	0.85	11.12	29.0	0.11	0.42	0.84	7.97	10.43	21.1	26.4	69.2	97.0
	0.12	0.84	11.21	29.3	0.11	0.42	0.84	8.12	10.49	21.1	26.6	69.7	98.1
11	0.1119	0.818	10.97	29.63	0.0981	0.4042	0.8127	7.860	10.25	20.61	26.20	73.08	118.0
	0.1133	0.8326	11.26	30.40	0.0986	0.4116	0.8289	8.017	10.50	21.11	26.78	74.42	119.7
	0.1125	0.8362	11.28	30.57	0.0995	0.4122	0.8301	8.042	10.48	21.14	26.82	75.61	120.1

TABLE A2.7 Analysis of Results, Low Resistivity Range Specimens, from Second Multilaboratory Test; Laboratories 2, 4, 8, 9, 10, 11

Slice Identification	17	T	U	43	2051	V
Number of Measurements	18	18	18	6	9	15
Grand Average ($\Omega \cdot \text{cm}$)	0.00834	0.01417	0.0979	0.4242	0.4120	0.8302
Reproducibility (1S %)	1.6 %	1.3 %	1.5 %	3.2 %	0.9 %	2.8 %
Pooled Reproducibility					2.1 %	

TABLE A2.8 Analysis of Results, High Resistivity Range Specimens, from Second Multilaboratory Test Laboratories 1, 2, 4, 9, 11

Slice Identification	U	43	2051	V	6	W	56	1055	X	Y	2
Number of Measurements	15	9	6	15	15	15	9	6	15	15	12
Grand Average ($\Omega \cdot \text{cm}$)	0.0995	0.4201	0.4063	0.8203	7.977	10.42	20.51	21.26	26.70	75.4	117.1
Reproducibility (1S %)	1.8 %	1.2 %	1.2 %	1.6 %	2.1 %	1.9 %	1.7 %	1.9 %	2.2 %	4.2 %	6 %
Pooled Reproducibility		1.2 %					1.8 %				

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