Designation: F 95 – 89 (Reapproved 2000)

Standard Test Method for Thickness of Lightly Doped Silicon Epitaxial Layers on Heavily Doped Silicon Substrates Using an Infrared Dispersive Spectrophotometer[1]

This standard is issued under the fixed designation F 95; 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.

INTRODUCTION

In editions of this test method published through 1987, the title and scope of the method required that the epitaxial layer and substrate be of the same conductivity type. This requirement was dropped, allowing the epitaxial layer and substrate to be of opposite conductivity type in the revision first published in 1988, subject to the continuing requirements of minimum allowed resistivity of the epitaxial layer and maximum allowed resistivity of the substrate. This same revision changed specifications on dispersive instruments from wavelength values to wave number values, where appropriate. A brief description of the theory of this test method is given in Appendix X1.

Automated test systems, utilizing Fourier-Transform Infrared Spectrophotometry (FT-IR) are widely used for epitaxial layer thickness measurements. Because such instruments are normally supplied with proprietary software for measurement analysis, detailed procedures for the use of such instruments are not included in this test method. However, for information purposes, estimates of single instrument repeatability and multiinstrument reproducibility, based on a 1986/1987 multilaboratory comparison of FT-IR instrument measurements are given in Note 6 and Appendix X2.

Automated test systems, utilizing Fourier-Transform Infrared Spectrophotometry (FT-IR) are widely used for epitaxial layer thickness. Because such instruments are normally supplied with proprietary software, detailed procedures for the use of such instruments are not included in this test method.

1. Scope

1.1 This test method[2] provides a technique for the measurement of the thickness of epitaxial layers of silicon deposited on silicon substrates. A dispersive infrared spectrophotometer is used. For this measurement, the resistivity of the substrate must be less than 0.02 Ω·cm at 23°C and the resistivity of the layer must be greater than 0.1 Ω·cm at 23°C.

1.2 This technique is capable of measuring the thickness of both *n*- and *p*-type layers greater than 2 µm thick. With reduced precision, the technique may also be applied to both *n*- and *p*-type layers from 0.5 to 2 µm thick.

1.3 This test method is suitable for referee measurements.

1.4 *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 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 932 Practice for Describing and Measuring Performance of Dispersive Infrared Spectrophotometers[4]

F 84 Test Method for Measuring Resistivity of Silicon Slices with an In-Line Four-Point Probe[5]

3. Terminology

3.1 *Definitions:*

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[1] This test method is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

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[2] DIN 50437 is an equivalent method. It is the responsibility of DIN Committee NMP 221, with which Committee F-1 maintains close technical liaison. DIN 50437. Testing of Inorganic Semiconductor Materials: Measurement of the Thickness of Silicon Epitaxial Layers by the Infrared Interference Method, is available from Beuth Verlag GmbH Burggrafenstrasse 4-10, D-1000 Berlin 30, Federal Republic of Germany and Vol 10.05.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] *Annual Book of ASTM Standards*, Vol 10.05.

3.1.1 *epitaxial layer—in semiconductor technology*, a layer of semiconductor material having the same crystalline spacing as the host substrate on which it is grown.

3.1.2 *index of refraction*—the relative index of refraction is defined by Snell's law as the ratio of the sine of the angle of incidence to the sine of the angle of refraction. The angles are measured between the surface normal and the infrared beam. The value of this index for the wavelength range from 6 to 40 µm is 3.42 relative to air for silicon having resistivity greater than 0.1 Ω·cm.

3.1.3 *interface*—the boundary between the substrate and the epitaxial layer as determined by this technique.

3.1.4 *substrate—in semiconductor technology*, a wafer that is the basis for subsequent processing operations in the fabrication of semiconductor devices or circuits.

3.1.4.1 *Discussion*—The devices or circuits may be fabricated directly in the substrate or in a film of the same or another material grown or deposited on the substrate.

4. Summary of Test Method

4.1 The reflectance of the specimen is measured as a function of wave number using an infrared spectrophotometer. The reflectance spectrum of a suitable specimen exhibits successive maxima and minima characteristic of optical interference phenomena. The thickness of the epitaxially deposited layer is calculated using the wave numbers of the extrema in the reflectance spectrum, the optical constants of the layer and the substrate, and the angle of incidence of the infrared beam upon the specimen.

5. Apparatus

5.1 *Double-Beam Infrared Spectrophotometer*—This apparatus shall utilize monochromatic infrared light of known and variable wave numbers. This light shall be reflected from the specimen and the reflectivity shall be recorded as a function of wave number. It is essential that the wave numbers indicated by the apparatus be carefully calibrated. Calibration accuracy shall be determined in accordance with Practice E 932E 932, using polystyrene lines at 1601.6 and 648.9 cm^{-1}. The calibration accuracy shall be 0.05 µm. The useful range of wave number is 1670 to 250 cm^{-1}. The precision and thickness capabilities stated in Section 11 were established using data in the range 900 to 300 cm^{-1}. In general, thinner specimens require a broader wavelength range than thicker ones.

5.2 *Specimen Holder*— The specimen holder shall be so constructed that no damage can be inflicted by the holder on the epitaxial layer.

5.3 *Masking Aperture*— The size of the masking aperture shall be such as to restrict the illuminated area on the specimen surface to a value sufficiently small to eliminate the effect of thickness fluctuations, without impairing detection of the reflected light. The masking aperture shall be constructed from a nonreflecting material such as matte-surface graphite.

6. Test Specimen

6.1 The specimen surface shall be highly reflective, free from large-area imperfections, and free of passivating layers except native oxides. The specimen surface may be cleaned prior to measurement by any technique which does not affect the specimen polish or the layer thickness.

7. Preparation of Apparatus

7.1 Establish the maximum allowable scan speed as follows:

7.1.1 Choose a specimen with a substrate resistivity between 0.008 and 0.02 Ω·cm, (see Scope) and an epitaxial layer of such thickness that an observable minimum occurs at a wave number less than 400 cm^{-1}.

7.1.2 Choose a suitable masking aperture.

7.1.3 Place the specimen on the instrument and record the spectrum of a minimum wave number less than 400 cm^{-1} using the slowest scan speed available.

7.1.4 Record the position of the minimum.

7.1.5 Increase the scan speed in steps and record the position of the minimum for each scan speed.

7.1.6 Allowable scan speeds are those which show a shift of the minimum of less than ±1 cm^{-1} relative to the position of the minimum as determined at the slowest scan speed.

8. Procedure

8.1 Handle the specimen carefully to avoid surface damage to the thin epitaxial layer.

8.2 Place the specimen over the aperture mask to expose the desired location to the beam.

8.3 Obtain a reflection spectrum similar to that shown in Fig. 1. Do not attempt a calculation of layer thickness if the peak amplitude to noise amplitude ratio is less than five.

NOTE 1—The interference pattern may be obscured or illegible if the

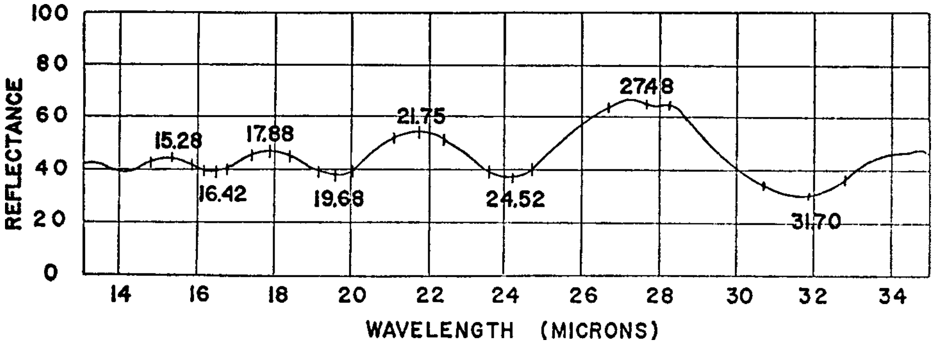

FIG. 1 Typical Reflection Spectrum for n-Type Specimen

TABLE 1 Phase (Shifts ($\phi/2\pi$) for *n* -Type Silicon

Wave-length, µm	Resistivity, Ω·cm														
	0.001	0.002	0.003	0.004	0.005	0.006	0.007	0.008	0.009	0.010	0.012	0.014	0.016	0.018	0.20
2	0.033	0.029	0.028	0.027	0.027	0.026	0.025	0.024	0.023	0.022	0.020	0.019	0.017	0.016	0.021
4	0.061	0.050	0.047	0.046	0.045	0.043	0.041	0.039	0.038	0.036	0.034	0.031	0.029	0.027	0.025
6	0.105	0.072	0.064	0.062	0.060	0.057	0.055	0.052	0.050	0.048	0.044	0.042	0.039	0.036	0.033
8	0.182	0.099	0.083	0.078	0.075	0.071	0.067	0.064	0.016	0.059	0.054	0.051	0.047	0.043	0.040
10	0.247	0.137	0.105	0.095	0.090	0.084	0.079	0.075	0.071	0.069	0.063	0.059	0.055	0.051	0.047
12	0.289	0.183	0.132	0.115	0.106	0.098	0.091	0.084	0.081	0.078	0.072	0.067	0.062	0.057	0.053
14	0.318	0.225	0.164	0.137	0.124	0.113	0.104	0.097	0.092	0.087	0.080	0.074	0.069	0.064	0.059
16	0.339	0.258	0.197	0.163	0.144	0.129	0.117	0.109	0.102	0.097	0.088	0.082	0.075	0.070	0.065
18	0.355	0.283	0.226	0.189	0.166	0.146	0.131	0.121	0.113	0.107	0.096	0.089	0.082	0.076	0.070
20	0.368	0.303	0.251	0.214	0.188	0.165	0.147	0.134	0.124	0.117	0.105	0.096	0.088	0.081	0.075
22	0.378	0.319	0.272	0.236	0.209	0.183	0.163	0.148	0.136	0.127	0.113	0.104	0.095	0.087	0.081
24	0.387	0.333	0.289	0.255	0.229	0.202	0.179	0.162	0.148	0.138	0.122	0.111	0.101	0.093	0.086
26	0.394	0.344	0.303	0.272	0.246	0.219	0.196	0.177	0.161	0.150	0.131	0.119	0.108	0.099	0.091
28	0.401	0.353	0.316	0.286	0.261	0.235	0.211	0.191	0.175	0.161	0.141	0.127	0.115	0.104	0.096
30	0.406	0.362	0.326	0.298	0.275	0.250	0.226	0.206	0.188	0.173	0.150	0.135	0.121	0.110	0.101
32	0.411	0.369	0.336	0.309	0.287	0.263	0.240	0.219	0.210	0.185	0.160	0.143	0.128	0.116	0.106
34	0.415	0.375	0.344	0.319	0.297	0.274	0.252	0.232	0.213	0.197	0.170	0.151	0.135	0.122	0.112
36	0.419	0.381	0.351	0.327	0.307	0.285	0.263	0.243	0.225	0.209	0.180	0.160	0.143	0.129	0.117
38	0.422	0.386	0.357	0.335	0.315	0.294	0.273	0.254	0.236	0.220	0.191	0.167	0.150	0.135	0.123
40	0.425	0.391	0.363	0.341	0.323	0.302	0.283	0.264	0.246	0.230	0.200	0.178	0.158	0.141	0.128

thickness of the epitaxial layer varies by more than 4 % over the masking aperture, or if the interface impurity concentration profile does not approximate a step function.

8.4 Determine the wave number of each extremum in the reflection spectrum by averaging the intercepts of the reflection spectrum and a horizontal line 3 % of full scale below a maximum or above a minimum. This procedure reduces the ambiguity encountered when the extrema are broad.

NOTE 2—A more correct procedure for locating the position of the extrema on layers less than 2 µm thick is to draw the tangent envelops to the spectrum and determine the intersection of the envelopes with the interference spectrum.[6] However, this procedure is apparently more difficult to perform since its use in a round-robin test (see 11.3) resulted in reduced rather than improved precision.

8.5 Measure the resistivity of the substrate in the area of the thickness measurement on the side opposite the epitaxial layer using the four-probe method of Test Method F 84F 84.

9. Calculation

9.1 Determine the orders for the maxima and minima observed using the following equation:

$$P_2 = m\lambda_1/(\lambda_1 - \lambda_2) + 1/2 - (\phi_2\lambda_1 - \phi_{22}\lambda_2)/2\pi(\lambda_1 - \lambda_2) \quad (1)$$

where:

P_2 = order of the extremum associated with λ_2,

λ_1 = 10 000/υ_1,

λ_2 = 10 000/υ_2,

m = difference in the orders of the extrema considered, and

ϕ_{21} and ϕ_{22} are the phase shifts suffered by the ray reflected at the interface for λ_1 and λ_2 respectively.

9.1.1 Obtain the phase shifts ϕ_{21} and ϕ_{22} from Table 1 or Table 2 . Round off the calculated order, P_2, to an integer for a maximum and a half integer for a minimum. After calculating one order, assign the orders to the remaining extrema in descending order with increasing wavelength as shown in Fig. 1.

9.2 Calculate the thickness using the following equation:

$$T_n = [P_n - 1/2 + (\phi_{2n}/2\pi)]\cdot\lambda_n/2(n_1^2 - \sin^2\theta)^{1/2} \quad (2)$$

where:

T_n = epitaxial layer thickness,

n_1 = index of refraction of the epitaxial layer ($n_1 = 3.42$ for silicon),

θ = angle of incidence of the beam upon the epitaxial layer, and

the other symbols have the same meaning they had in Eq 1. Use the same units for the thickness as for the wavelength. Calculate T_n for all of the observed maxima and minima and calculate the average value of T.

NOTE 3—When several extrema are available, somewhat better precision than that reported in Section 11 will result if the longer wavelength points are excluded from the calculation.

9.3 *Sample Calculation*—Typical data and a calculation of epitaxial layer thickness for an *n*-type specimen, using the reflection spectrum shown in Fig. 1, are as follows:

9.3.1 The measured value of substrate resistivity was 0.014 Ω/cm.

9.3.2 Determine wavelength of first and last extrema; $\lambda_1 = 31.70$ µm and $\lambda_2 = 15.28$ µm.

9.3.3 Read appropriate phase shifts from Table 1: $\phi_{21}/2\pi = 0.142$ and $\phi_{22}/2\pi = 0.079$.

9.3.4 Find from Fig. 1 the difference in the orders of the extrema considered: $m = 3.5$.

9.3.5 Substitute in Eq 1 and solve for P_2:

$$P_2 = 3.5\,(31.70)/(31.70 - 15.28) + 1/2 - [31.70\ (0.142) - 15.28\ (0.079)]/(31.70 - 15.28) = 6.80 + 0.50 - 0.20 = 7.10 \approx 7$$

9.3.6 Substitute in Eq 2 and solve for T_2:

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[6] Schumann, P. A., "The Infrared Interference Method of Measuring Epitaxial Layer Thickness," *Journal Electrochemical Society*, Vol 116, 1969, p. 410.

TABLE 2 Phase Shifts ($\phi/2\pi$) for *p* -Type Silicon

Wave-length, µm	Resistivity, Ω·cm															
	0.001	0.0015	0.002	0.003	0.004	0.005	0.006	0.007	0.008	0.009	0.010	0.012	0.014	0.016	0.018	0.020
2	0.036	0.034	0.033	0.033	0.033	0.034	0.034	0.033	0.032	0.031	0.030	0.028	0.027	0.025	0.024	0.024
4	0.067	0.060	0.057	0.055	0.055	0.055	0.055	0.054	0.052	0.050	0.049	0.045	0.043	0.040	0.038	0.037
6	0.119	0.091	0.082	0.076	0.074	0.073	0.072	0.071	0.068	0.066	0.064	0.059	0.056	0.053	0.050	0.049
8	0.200	0.140	0.114	0.099	0.094	0.091	0.089	0.086	0.083	0.080	0.077	0.072	0.067	0.064	0.060	0.059
10	0.261	0.199	0.158	0.127	0.115	0.110	0.105	0.102	0.097	0.093	0.089	0.083	0.078	0.073	0.070	0.068
12	0.300	0.247	0.205	0.160	0.140	0.130	0.123	0.117	0.111	0.106	0.101	0.094	0.088	0.083	0.078	0.076
14	0.327	0.282	0.244	0.194	0.167	0.152	0.141	0.133	0.126	0.119	0.113	0.104	0.097	0.091	0.087	0.084
16	0.346	0.307	0.274	0.226	0.195	0.175	0.161	0.151	0.141	0.132	0.126	0.115	0.106	0.100	0.094	0.091
18	0.361	0.327	0.297	0.253	0.221	0.198	0.182	0.168	0.157	0.146	0.138	0.125	0.116	0.108	0.102	0.099
20	0.373	0.342	0.315	0.274	0.243	0.220	0.202	0.186	0.173	0.160	0.151	0.136	0.125	0.117	0.100	0.106
22	0.383	0.354	0.330	0.292	0.263	0.240	0.220	0.204	0.188	0.175	0.164	0.147	0.134	0.125	0.117	0.113
24	0.391	0.365	0.342	0.307	0.279	0.257	0.238	0.220	0.204	0.189	0.177	0.158	0.144	0.133	0.125	0.120
26	0.398	0.374	0.352	0.320	0.294	0.272	0.253	0.236	0.219	0.203	0.190	0.169	0.153	0.142	0.132	0.127
28	0.404	0.381	0.361	0.331	0.306	0.285	0.267	0.250	0.233	0.217	0.203	0.180	0.163	0.150	0.140	0.134
30	0.409	0.387	0.369	0.340	0.316	0.297	0.279	0.262	0.245	0.229	0.215	0.191	0.173	0.159	0.148	0.141
32	0.414	0.393	0.376	0.348	0.326	0.307	0.290	0.273	0.257	0.241	0.227	0.202	0.182	0.167	0.155	0.148
34	0.418	0.398	0.381	0.355	0.334	0.316	0.299	0.284	0.268	0.252	0.238	0.213	0.192	0.176	0.163	0.155
36	0.421	0.403	0.387	0.362	0.341	0.324	0.308	0.293	0.277	0.262	0.248	0.223	0.201	0.185	0.171	0.162
38	0.425	0.407	0.391	0.368	0.348	0.331	0.316	0.301	0.286	0.271	0.258	0.232	0.211	0.193	0.178	0.169
40	0.428	0.410	0.396	0.373	0.354	0.338	0.323	0.309	0.294	0.280	0.266	0.241	0.219	0.201	0.186	0.176

$$T_2 = (7 - 1/2 + 0.079)(15.28)(0.1477) = 14.85\ \mu\text{m}$$

NOTE 4— $\frac{1}{2}\,(n_1^{\,2} - \sin^2\ \theta)^{1/2} = 0.1477$ for $\theta = 30°$.

9.4 Tabulate ν_n, λ_n, $\phi_{2n}/2\pi$, P_n, and T_n for all the maxima and minima in Fig. 1 as shown in Table 3.

10. Report

10.1 Report the following information:
10.1.1 Identification of slices.
10.1.2 Substrate conductivity type,
10.1.3 Substrate resistivity,
10.1.4 Epitaxial layer conductivity type,
10.1.5 Estimated epitaxial layer resistivity,
10.1.6 Wave number range of infrared apparatus used,
10.1.7 Aperature size,
10.1.8 Wave number scan speed,
10.1.9 Location of measurement with sketch of specimen,
10.1.10 Wave numbers used, λ_n,
10.1.11 Orders of maxima and minima used P_n,
10.1.12 Thicknesses calculated, T_n, and
10.1.13 Average thickness value, T.

11. Precision and Bias

11.1 For *p*-type layers of thickness greater than approximately 2 µm, the measurement can be made with an interlaboratory precision, as defined in Practice E 177E 177, of ±(0.25µm + 0.025 T) (3S) where T is the layer thickness in micrometres. This precision was established in a round-robin experiment in which eight laboratories made one measurement on each of six layers with thickness in the range of 2.5 to 18 µm.

TABLE 3 Example of Thickness Computations (See Fig. 1)

n	λ_n	$\phi 2_n/2\pi$	P_n	T_n
1	31.70	0.142	3.5	14.71
2	27.48	0.124	4	14.71
3	24.52	0.113	4.5	14.89
4	21.75	0.103	5	14.79
5	19.68	0.095	5.5	14.81
6	17.88	0.088	6	14.76
7	16.42	0.082	6.5	14.75
8	15.28	0.079	7	14.85
Average	...	...	...	14.78

11.2 For *n*-type layers of thickness greater than approximately 2 µm, the measurement can be made with an interlaboratory precision of ±(0.25 µm + 0.005 T) (3S). This precision was established in a round-robin experiment in which eight laboratories made one measurement on each of six layers with thickness in the range from 2.5 to 15 µm.

11.3 For *n*- or *p*-type layers of thickness 0.5 to approximately 2 µm, the measurement can be made with an interlaboratory precision of ±(0.51 µm + 0.035 T) (3S). This precision was established in a round-robin experiment in which seven laboratories made one measurement on each of five layers with thickness in the range from 1 to 7 µm.

NOTE 5—Use of the procedure described in Note 2 for locating positions of extrema in Note 2 resulted in greater variability of the measurement.

11.4 The bias of this technique has not been established, and the relation to layer thickness as determined by other methods is not known.

NOTE 6—A sampling of six similar FT-IR systems has demonstrated single-system-operator-day precisions of ±0.1 µm (2S), as defined in Practice E 177E 177. No full round robin has been run; however, the mean value of thickness determined by each of these six systems has been shown to agree with the overall mean value to within ±5 %. The difference between the thickness as determined by this method and that determined by the automatic test systems exhibits a small linear dependence on thickness. As an example, for one system over the thickness range 3 to 30 µm on n/n^+ and p/p^+ wafers with resistivities between 0.002 and 0.014 Ω·cm, this difference is given by the relation:

$$T_s = 1.0026\ T - 0.283$$

where:
T_s = thickness value from automatic test system, µm, and
T = thickness value from this method, µm.

The factor 1.0026 and the term – 0.283 µm result from a least-squares fit to the data.

An extensive multilaboratory test was conducted in 1986/1987 to