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Measurement of thermal conductivity of thin films on silicon substrates

Mesurage de la conductivité thermique des films minces sur substrat de silicium

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

To respond to the need for global collaboration on standardization questions at early stages of technological innovation, the ISO Council, following recommendations of the ISO/IEC Presidents' Advisory Board on Technological Trends, decided to establish a new series of ISO publications named "Technology Trends Assessments" (ISO/TTA). These publications are the results of either direct cooperation with prestandardization organizations or ad hoc Workshops of experts concerned with standardization needs and trends in emerging fields.

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Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

VAMAS, the Versailles project on Advanced Materials and Standards, was formed in 1982 at the Economic Summit of the G7 Heads of State as a way to support world trade in products dependent on advanced materials technologies by providing the technical basis for harmonized measurements, testing, specifications, and standards. Through its technical programmes, VAMAS emphasizes international collaborative pre-standards measurement research in support of development of national and international standards by standards development organizations such as ISO.

ISO/TTA 4 was prepared by VAMAS and published under a memorandum of understanding concluded between ISO and VAMAS.

Introduction

The purpose of this document is to propose a standard procedure for measuring the thermal conductivity of insulating thin films on silicon substrates. Based on a recent interlaboratory comparison, a recommendation is made for the adoption of the three-omega method as a standard measurement method. A procedure for the three-omega method is proposed for measuring the thermal conductivity of a thin, electrically insulating film, on a substrate having a thermal conductivity significantly greater than the thermal conductivity of the film. Annex B contains a review of several measurement methods that have been used to measure the thermal conductivity of such films (see reference [1]).

Measurement of thermal conductivity of thin films on silicon substrates

1 Scope

1.1 A standard procedure for the three-omega method is proposed for measuring the thermal conductivity of a thin, electrically insulating film, on a substrate having a thermal conductivity significantly greater than the thermal conductivity of the film. This method is applicable to a film on a silicon substrate with the following characteristics:

a) the film is electrically insulating;

b) the film has a thermal conductivity that is less than one tenth the thermal conductivity of silicon;

c) the film is uniform in thickness and the thickness lies in the range 0,25 µm to 1 µm;

d) the maximum dimensions of the film are limited by the sizes of the preparation and measurement apparatus;

e) the minimum dimensions of the film are limited by the minimum size of the circuit element that can be placed on the film surface.

NOTE A specimen approximately 15 mm by 25 mm is of an appropriate size although specimens as small as 10 mm × 10 mm are usable.

1.2 The method is directly applicable to films of silicon dioxide on silicon wafer substrates.

1.3 The method may be applicable to insulating films on other high-thermal conductivity substrates provided that the parameters of the substrate material are substituted for the parameters of silicon used in this method and the associated computer program.

1.4 The method is applicable to measurements near room temperature.

2 Symbols

See Figure 1.

f	frequency of excitation voltage
ω	angular frequency of excitation voltage = $2\pi f$
Λ	thermal conductivity of substrate
Λ_f	thermal conductivity of film
w	width of specimen resistor
t	film thickness
L	length of specimen resistor
V_0	excitation voltage at ω
V	voltage at ω across the specimen
R	mean resistance of the specimen

ΔR variation at 2ω of the specimen resistance

V_{OS} voltage at ω across offset resistor

R_{OS} resistance of offset resistor

V_{CAL} voltage at ω across calibration resistor

R_{CAL} resistance of calibration resistor = 10 Ω

$V_{3\omega}$ voltage at 3ω across specimen

T_{SP} temperature setting

T mean temperature of the specimen, also known as the measurement temperature

ΔT temperature variation of specimen at 2ω

ΔT_b bare substrate thermal signal

I electrical current at ω = $V_0/(R + R_{REF} + R_{CAL})$

P power dissipated in specimen resistor = I^2R

dR/dT temperature coefficient of resistance

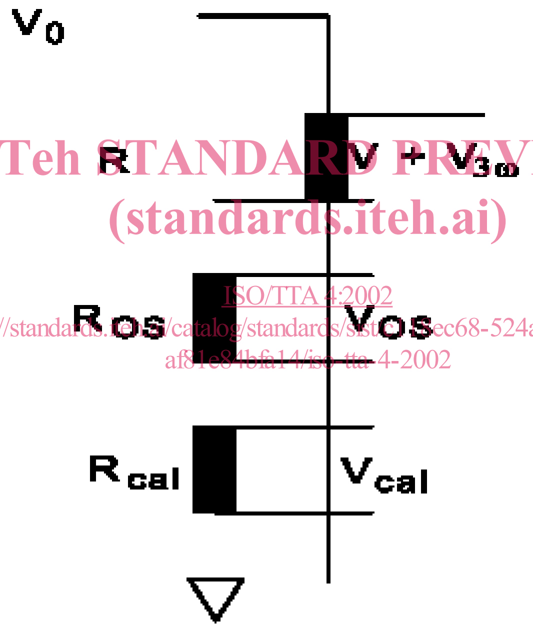

Figure 1 — Nomenclature

3 Specimen preparation and characterization

3.1 Apparatus

3.1.1 Photomask, a mask for photolithography used in manufacture of integrated electronic circuits. See Figure 2 for the recommended configuration. It is recommended that the photomask contain several duplicates of the desired circuit elements in case a deposited element is not usable.

3.1.2 Clean room.

3.1.3 Spin coater.

3.1.4 Cleaning solution.

3.1.5 Deionized water.

3.1.6 Photoresist.

3.1.7 Photoresist developer.

3.1.8 Acetone.

3.1.9 Aluminium etch.

3.1.10 Vacuum deposition chamber, equipped for deposition of aluminium and set up to hold the specimen.

3.1.11 Ultraviolet exposure system.

3.1.12 Baking ovens.

3.1.13 Plasma etcher.

3.1.14 Assorted plastic cups and tweezers.

3.1.15 Optical comparitor for line width measurement.

3.1.16 Ellipsometer for thickness measurement.

3.1.17 Electrically conducting silver paste.

3.2 Determination

Measure the film thickness using the ellipsometer and record the value as t in Table 1.

3.3 Circuit element preparation

3.3.1 Using the cleaning solution, clean the specimen using an established cleaning procedure.

3.3.2 Place the specimen in the vacuum deposition chamber and deposit an aluminium film approximately 300 nm thick onto the film surface of the specimen. Remove the specimen from the chamber.

3.3.3 Spin coat the metallized surface of the specimen with photoresist.

3.3.4 Bake the specimen in an oven at 95 °C for 25 min.

3.3.5 Soak the specimen in water for 2 min.

3.3.6 Mount the specimen and the photomask in the ultraviolet exposure system.

3.3.7 Expose the specimen to ultraviolet radiation through the photomask for the time appropriate to the exposure system.

3.3.8 Develop the specimen in the photoresist developer diluted with water (volume ratio of photoresist developer to water = 1:4) for 30 to 45 s.

3.3.9 Bake the specimen in an oven at 125 °C for 25 min.

3.3.10 Plasma etch the specimen in oxygen for 45 s to remove any scum on the specimen surface.

3.3.11 Etch the specimen in the aluminium etchant at 50 °C for 30 to 45 s.

3.3.12 Soak in acetone for about 1 min to remove remaining photoresist.

3.3.13 Rinse in deionized water and dry in air.

3.3.14 With the optical comparitor, measure the line width of the circuit element produced. Record this value as w in Table 1.

Use a blank table, Table 1 displays a set of representative data.

NOTE The line width will usually differ significantly from the line width of the photomask.

3.3.15 Record in Table 1 the length of the metal strip between the two voltage pads shown in the photomask design as L.

NOTE The value for L used in the photomask design in Figure 2 is satisfactory for the purposes of this method.

3.3.16 Using the silver paste, attach fine copper wires to the four electrical pads of the circuit element chosen to be used in the experiment.

3.3.17 Check for electrical continuity in the circuit element with a volt-ohm-resistance meter between all pairs of electrical pads. If an open circuit condition exists or a short circuit condition exists, choose a different circuit element and repeat this step in the procedure with this new circuit element. If none of the circuit elements is satisfactory, reject the specimen as unacceptable for measurement.

The resistance between the voltage pads (the two left pads of a circuit element shown in Figure 2) should be between 10 Ω and 100 Ω.

Dimensions in millimetres

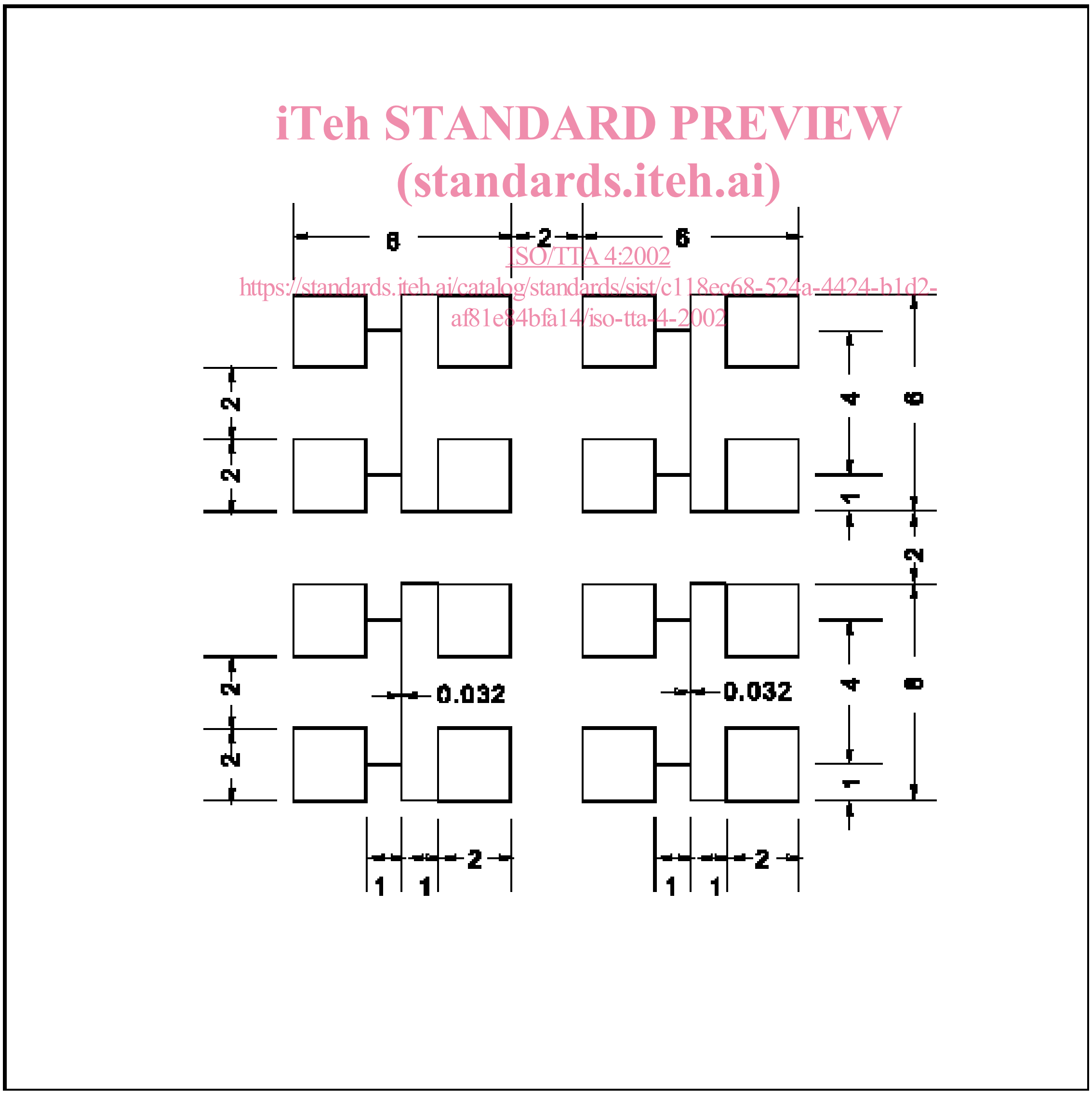

Figure 2 — Recommended photomask pattern for 3-Omega Method

4 Measurement apparatus

4.1 A variable temperature environmental chamber to hold the specimen. The chamber specifications are:

4.1.1 Temperature adjustable to 20 °C and 60 °C with an uncertainty of 0,1 °C or less.

4.1.2 A thermocouple to measure the temperature at the position of the specimen and a thermocouple readout. The uncertainty in the temperature measurement should be 0,1 °C or less.

4.1.3 Four electrical feed-throughs to accommodate two current leads and two voltage leads to the specimen.

4.2 An electrical measurement system as shown in Figure 3 consisting of the following components:

4.2.1 A digital lock-in amplifier for performing the electrical measurements. The specifications are:

4.2.1.1 A signal generator that generates a reference signal having an output voltage adjustable to 0,1 V and to 2,0 V at the frequency of 333 Hz.

4.2.1.2 Ability to measure single-ended input voltages and differential input voltages.

4.2.1.3 Settings capable of measuring signals at the fundamental frequency (f) and at the third harmonic ($3f$).

4.2.1.4 A voltage measurement uncertainty of 0,1 % or less.

4.2.2 A switch box for switching input A between measuring V_{CAL} (position y) and measuring V or $V_{3\omega}$ (position x).

4.2.3 Additional components as shown in Figure 3.

5 Measurement procedure

5.1 Mount the specimen in the environmental chamber making all of the necessary electrical connections.

5.2 Set T_{SP} to 20 °C and wait until the temperature stabilizes. Read the temperature on the thermocouple readout and record the value in Table 1 as $T_{SP}(20)$ to within 0,1 °C.

5.3 Set the lock-in frequency to 333 Hz. Set the time constant to 3 s and the roll-off to 24 db/oct. Set the lock-in to read the in-phase signal.

5.4 Set V_0 to 0,1 V.

5.5 Set R_{NULL} to an intermediate position.

5.6 Wait 20 min for specimen temperature to stabilize.

5.7 Set the switch to position x. Set the lock-in to read the differential input voltage.

5.8 Adjust R_{OS} until approximately zero voltage is measured. Maximize the gain on the lock-in and adjust R_{NULL} until a minimum voltage is obtained.

5.9 Lower the gain setting of the lock-in amplifier to the minimum setting. Switch the lock-in to single ended input.

5.10 Set the lock-in gain to a convenient range. Use the autogain feature if the lock-in is so equipped. Read V and record this value in Table 1, col. 1.

