
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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Published in Switzerland

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Foreword

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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]).

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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 \times 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 = $10\ \Omega$
$V_{3\omega}$	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 $\omega = V_0/(R + R_{REF} + R_{CAL})$
P	power dissipated in specimen resistor = $I^2 R$
dR/dT	temperature coefficient of resistance

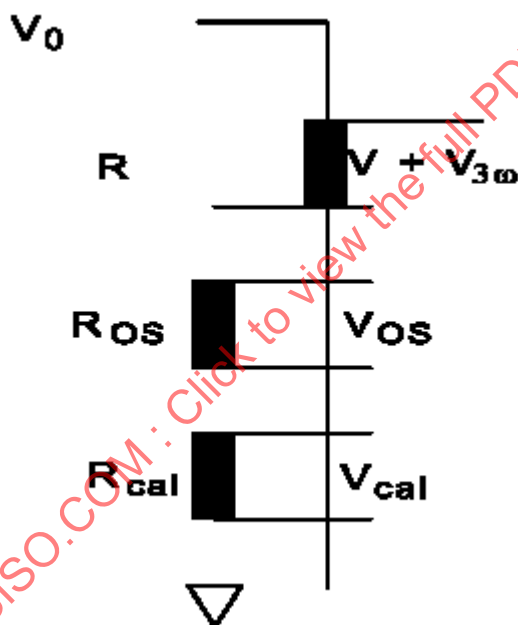


Figure 1 — Nomenclature

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 comparator, 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 $10\ \Omega$ and $100\ \Omega$.

Dimensions in millimetres

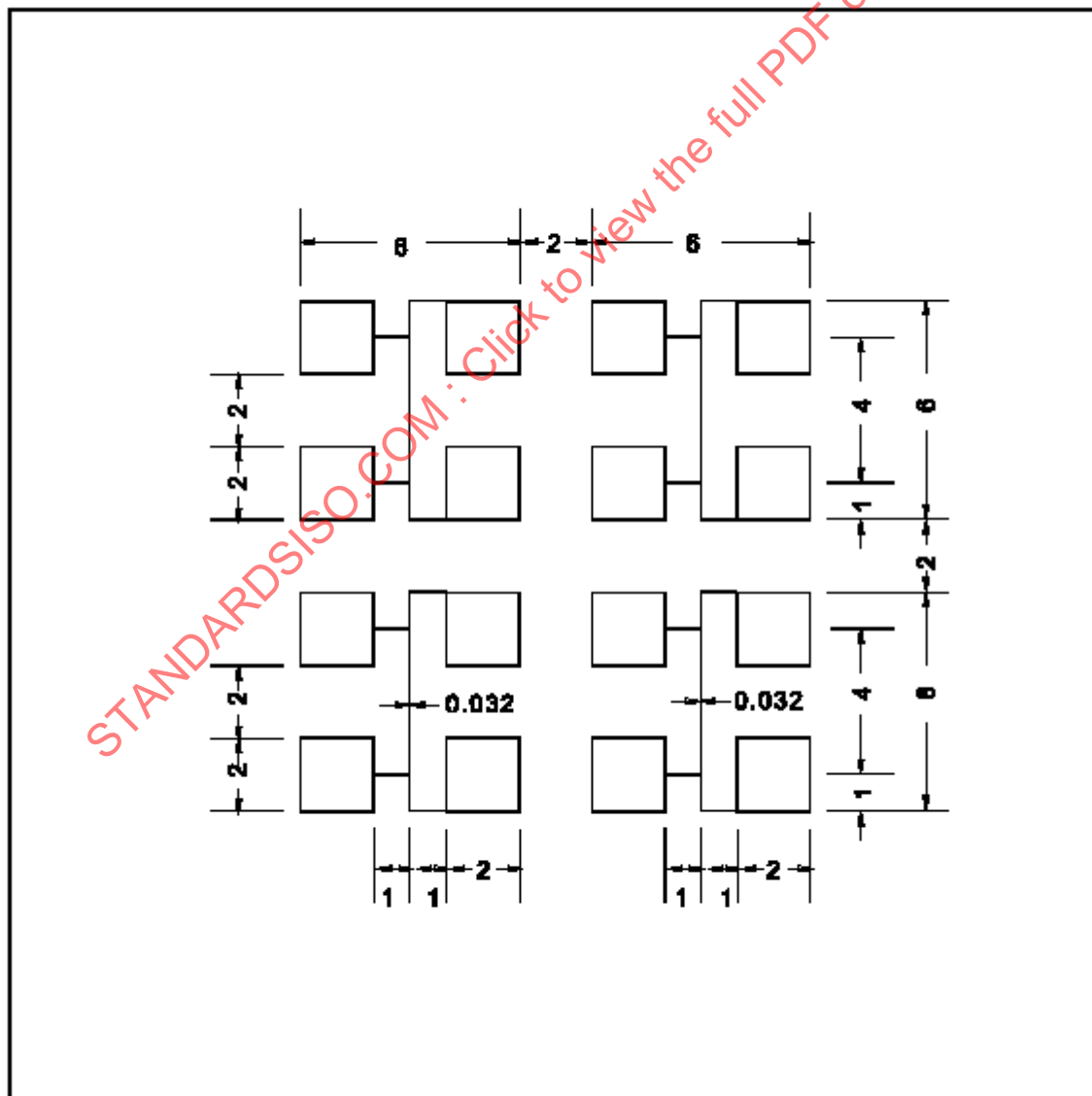


Figure 2 — Recommended photomask pattern for 3-Omega Method

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 $T_{\text{SP}}(20)$ 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.

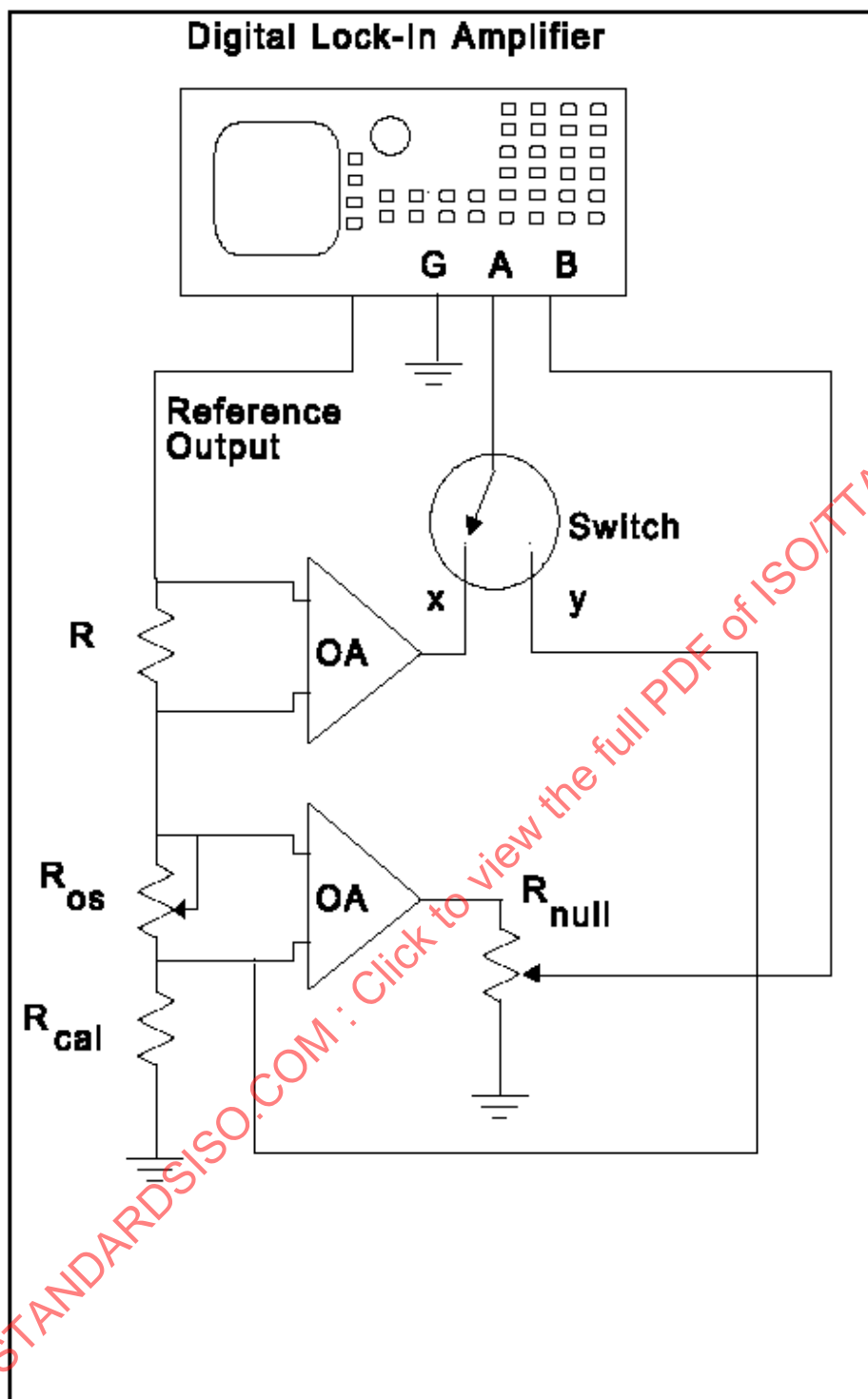
5.6 Wait 20 min for specimen temperature to stabilize.

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.



Key

- OA = two operational amplifiers (AD524), set for unity gain
- G = earth (ground)
- A, B = voltage inputs
- R_{NULL} = nulling resistor consisting of a 10 turn potentiometer, resistance 10 k Ω , in series with a fixed 50 k Ω resistor that is connected to ground
- R_{OS} = offset resistor consisting of a 10 turn potentiometer, resistance 5 k Ω
- R_{CAL} = calibration resistor, fixed resistance of 10,0 \pm 0,1 Ω
- SW = single-pole double-throw switch

Figure 3 — Electrical circuit

- 5.11** Set the switch to position y. Measure V_{CAL} and record this value in Table 1, col. 1.
- 5.12** Set V_0 to 2,0 V.
- 5.13** Wait 20 min for specimen temperature to stabilize.
- 5.14** Repeat steps 5.6 to 5.10, except enter the values of V and V_{CAL} in Table 1, col. 2.
- 5.15** Set the lock-in to read the voltage at 3ω .
- 5.16** Measure $V_{3\omega}$ and enter the value in Table 1, col. 2.
- 5.17** Set T_{SP} to 60 °C and wait for the temperature to stabilize. Record the temperature value in Table 1 as $T_{\text{SP}}(60)$ to within 0,1 °C.
- 5.18** Repeat steps 5.2 through 5.10 except record the indicated values in Table 1, col. 3.
- 5.19** Repeat steps 5.11 through 5.14 except record the indicated values in Table 1, col. 4.
- 5.20** Repeat steps 5.15 and 5.16 except record the respective values of $V_{3\omega}$ in Table 1, col. 4.

6 Calculations

- 6.1** For each column of Table 1, calculate I , P and R using the formulae

$$I = V_{\text{CAL}} / R_{\text{CAL}}$$

$$R = V / I$$

$$P = I^2 R$$

Insert the calculated values in the corresponding locations in Table 1.

- 6.2** For the initial temperature, extrapolate R to zero input power using the formula

$$R(20) = R_2 - P_1 \frac{R_2 - R_1}{P_2 - P_1}$$

Insert the value of $R(20)$ into Table 1.

- 6.3** For the second temperature, extrapolate R to zero input power using the formula

$$R(60) = R_4 - P_4 \frac{R_4 - R_3}{P_4 - P_3}$$

Insert the value of $R(60)$ into Table 1.

- 6.4** Calculate the temperature of measurement for col. 2 using the formula

$$T_2 = T_{\text{SP}}(20) + [R_2 - R(20)] \frac{T_{\text{SP}}(60) - T_{\text{SP}}(20)}{R(60) - R(20)}$$

and insert the value into Table 1.

- 6.5** Calculate the temperature coefficient of the specimen resistance from the formula below and record the value in Table 1.

$$\frac{dR}{dT} = \frac{R(60) - R(20)}{T_{\text{SP}}(60) - T_{\text{SP}}(20)}$$

6.6 Compute ΔT_b .

6.6.1 Using a text editor, generate a data input file with the name "input.dat" using the format of A.2 by substituting the appropriate data from Table 1, col. 2.

6.6.2 Using the executable version of the program in A.1, compute the bare substrate thermal signal, ΔT_b , and record the value in Table 1.

NOTE The program contains formulae based on handbook data for the temperature dependence of the thermal conductivity and thermal diffusivity of Si. If another substrate material is desired, modify the appropriate lines in the program. The formulae are located in lines 90 and 96 of program OMEGA3.

6.7 Calculate the total thermal signal using the formula below and record the value in Table 1.

$$\Delta T = 2V_{3\omega} \frac{dT}{dR} \frac{R}{V}$$

6.8 Calculate the thermal resistance of the thin film (units, $10^{-9} \text{m}^2 \text{K} \cdot \text{W}^{-1}$) using the formula below and record the value in Table 1.

$$R_T = \frac{(\Delta T - \Delta T_b)}{P} L_w - 23 \times 10^{-9}$$

NOTE The value 23×10^{-9} is the estimated thermal resistance due to interfaces in the specimen and is the value derived in reference [1].

6.9 Calculate the thermal conductivity of the film using the formula below and record the value in Table 1.

$$\lambda = \frac{t}{R_T}$$

7 Uncertainty

The standard uncertainty of a measurement is estimated to be $\pm 10\%$. A reanalysis of round robin data by the round robin participants would permit a better determination of the uncertainty. The deviation of the sample data shown in Table 1, obtained by one laboratory, gives a value $1,28 \text{ W} \cdot \text{m}^{-1} \text{K}^{-1}$, which deviates from the expected bulk value of $1,37 \text{ W} \cdot \text{m}^{-1} \text{K}^{-1}$ by $0,09 \text{ W} \cdot \text{m}^{-1} \text{K}^{-1}$ or $6,7\%$.

8 Test report

The report shall contain the following information:

- specimen identification;
- substrate material;
- film material;
- film thickness;
- temperature of measurement;
- film thermal conductivity.

Table 1 — Example of representative data

	Col. 1	Col. 2	$P = 0$	Col. 3	Col. 4	$P = 0$
L (m)		$4,00 \times 10^{-3}$				
w (m)		$28,45 \times 10^{-6}$				
t (m)		$0,488 \times 10^{-6}$				
f (Hz)		332,6				
T_{SP} (°C)	$T_{SP}(20) = 19,2$			$T_{SP}(60) = 60,0$		
V_{CAL} (V)	$7,126 \times 10^{-2}$	$1,778 \times 10^{-1}$		$6,732 \times 10^{-2}$	$1,685 \times 10^{-1}$	
V (V)	$1,925 \times 10^{-1}$	$4,824 \times 10^{-1}$		$2,065 \times 10^{-1}$	$5,188 \times 10^{-1}$	
I (A)	$7,126 \times 10^{-3}$	$1,778 \times 10^{-2}$				
R (Ω)	$R_1 = 27,01$	$R_2 = 27,13$	$R(20) = 27,27$	$R_3 = 30,67$	$R_4 = 30,79$	$R(60) = 30,93$
P (W)	$1,372 \times 10^{-3}$	$8,58 \times 10^{-3}$		$1,390 \times 10^{-3}$	$8,74 \times 10^{-3}$	
T (°C)		$T_2 = 20,8$				
$V_{3\omega}$ (V)		$3,59 \times 10^{-5}$				
dR/dT ($\Omega \cdot K^{-1}$)		$8,96 \times 10^{-2}$				
ΔT_b (K)		$1,46 \times 10^{-2}$				
ΔT (K)		$4,51 \times 10^{-2}$				
R_T (m^2KW^{-1})		$3,82 \times 10^{-7}$				
λ ($W \cdot m^{-1}K^{-1}$)		1,28				

Annex A

Computer programs

A.1 Fortran file for computation of ΔT_b

```

c      program omega3
c      version 1, 01-Nov-2001
c      Calculate real temperature variation at a given frequency
c      for a silicon plate heated by a thin metal strip.
c      The calculation can be applied to plates composed of
c      material other than silicon if the thermal conductivity
c      and thermal diffusivity of that material are substituted
c      in the indicated lines below.
c      This is used for analyzing data obtained by the 3 omega
c      method
c
c      library routine 'dqagi' used for integration
c      -----
c
c      A data file named 'input.par' written in ASCII is needed
c
c      A sample file is given in section A2. Just substitute data
c      in the appropriate lines. Double precision is used.
c
c      Function Fcomplex is written for a two layer system. The
c      main program defines one of these layers as having zero
c      thickness and having the same material parameters as
c      the other layer.
c      -----
c      Nomenclature (not necessarily the same as in the standard)
c      D      =diffusivity
c      k      =conductivity
c      L      =substrate thickness
c      lnth=heater length
c      b      =heater half width
c      b2     =heater full width
c      w      =angular frequency at 2 omega
c      f      =fundamental frequency
c      f2     =frequency at 2 omega
c      q      =power input to specimen
c      T      =measurement temperature
c      tr     =substrate temperature signal at 2 omega
c      -----
c      define integration parameters for dqagi
c      double precision, external :: fr
c
c      double precision, parameter :: bound=0.d0
c      double precision, parameter :: epsabs=1.d-6
c      double precision, parameter :: epsrel=1.d-12
c      integer, parameter :: inf=1
c      integer, parameter :: limit=1000
c      integer, parameter :: lenw=limit*4
c      integer, parameter :: idim=2

```



```

c      double precision result, abserr, work(lenw), T
c      double precision f, f2, b2
c      integer neval, ier, last, iwork(limit)
c      character*30, label
c -----
c common variables
c      double precision w, b
c      double precision d(idim), k(idim), L(idim)
c      common w, d, k, L, b
c
c      double precision q, pi
c      double precision tr, lnth
c
1      format(A12)
2      format(A30)
3      format(6(1x,d13.6))
4      format(1x,A33,D12.4)
c -----
c      pi=4.d0*atan(1.d0)
c -----
c      open(1, 'input.par')
c
c      read (1,2)
c      read(1,2) label ; read(1,*) f
c      read(1,2) label ; read(1,*) q
c      read(1,2) label ; read(1,*) b2
c      read(1,2) label ; read(1,*) lnth
c      read(1,2) label ; read(1,*) L(1)
c      read(1,2) label ; read(1,*) T
c
c      f2=2*f
c      w=2.d0*pi*f2
c      b=b2/2.d0
c ++++++
c      The thermal conductivity of Si as a function of
c      temperature is computed in the line 90 below. If another
c      material is wanted, replace line 90
c
90      k(1)=1.685d0-8.73d-3*T+3.62d-5*T*T-9.0d-8*T*T*T
c
c      The thermal diffusivity of Si as a function of the
c      thermal conductivity of Si is computed in line 96 below.
c      If another material is wanted, replace line 96.
c
96      D(1)=0.093d0+0.268d0*k(1)+0.180d0*k(1)*k(1)
c ++++++
c      write(*,4)'thermal conductivity of Si, W/cm/K ', k(1)
c      write(*,4)'thermal diffusivity of Si, cm2/s ', D(1)
c      k(2)=k(1)
c      D(2)=D(1)
c      L(2)=0.d0
c
c -----
c      Calculate the temperature variation
c
c      calculate average temperature (real and imaginary) using
c      dqagi
c

```

```

      call dqagi(fr, bound, inf, epsabs, epsrel,
*result, abserr, neval, ier, limit, lenw, last, iwork, work)
      if (ier.gt.0) then
        call list(bound, inf, epsabs, epsrel,
*      result, abserr, neval, ier, limit, lenw, last, 'fr')
        end if
c
      tr=result*q/pi/lnth
c
      write(*,4) 'substrate thermal signal ', tr
c -----
      close(2)
      end
c
c *****
c      function fr(x)
c      real part of integrand for dqagi
      double precision x, fr
      complex*8 fcomplex
c
      fr=real(fcomplex(x))
      return
      end function fr
c
c *****
c      function fcomplex(x)
c
c      complex integrand for 2 layers in vacuum
c
c      common variables
      double precision w, b
      double precision d(2), k(2), L(2)
      common w, d, k, L, b
c
      double precision x
      complex*8 u(2), ea1, ea2
      complex*8 ci, gp, gm, ex1, ex2, bp, bm, ftr, fcomplex
c
      ci=(0,1.d0)
      u(1)=sqrt(x*x-ci*w/d(1))
      u(2)=sqrt(x*x-ci*w/d(2))
      gp=u(1)*k(1)+u(2)*k(2)
      gm=u(1)*k(1)-u(2)*k(2)
      ea1= 2.d0*u(1)*L(1)
      if (real(ea1).gt.160) then
        ex1=0.d0
      else
        ex1= exp(-ea1)
      end if
      ea2= 2.d0*u(2)*L(2)
      if (real(ea2).gt.160) then
        ex2=0.d0
      else
        ex2= exp(-ea2)
      end if
      bp=(gp*ex2 + gm)*ex1
      bm=(gm*ex2 + gp)
      ftr=(bm+bp)/(bm-bp)/u(1)/k(1)
      if(x.eq.0.d0) then
        fcomplex=ftr

```

```

        else
            fcomplex=ftr*sin(x*b)*sin(x*b)/x/x/b/b
        end if
c      print*, 'end of fcomplex'
        return
    end function fcomplex
c
c *****
    subroutine list(bound, inf, epsabs, epsrel,
        * result, abserr, neval, ier, limit, lenw, last, ri)
c
        double precision bound, epsabs, epsrel, result, abserr
        integer inf, limit, lenw, neval, ier, last
        character*2 ri
c
        print*, 'list parameters from dqagi call to ', ri
        print*, 'epsabs=', epsabs
        print*, 'epsrel=', epsrel
        print*, ' limit=', limit
        print*, 'result=', result
        print*, ' neval=', neval
        print*, '   ier=', ier
        print*, ' last=', last
        print*, ' '
        return
    end subroutine list
c *****

```

A.2 Data input file containing representative data

The data are written in double precision. Substitute appropriate values from Table 1.

```

'file input.par'
'fundamental frequency, Hz'
332.6d0
'power, W'
8.58d-3
'line full width, cm'
28.45d-4
'line length, cm'
0.4d0
'thickness Si, cm'
0.038d0
'measurement temp. T2, deg C'
20.8d0

```

A.3 Library routine dqagi.for

National Institute of Standards and Technology (NIST) Guide to Available Math Software.

Full source for module DQAGI from package CMLIB. This software can be downloaded from the web site <http://gams.nist.gov>.

Annex B

Various methods of measuring thin-film thermal conductivity

B.1 Introduction

The thermal properties of a thin film can play an important role in the performance of the film in applications sensitive to the transport of heat through the film. In the case of microelectronic devices, we need electrically insulating films that are good heat conductors; in the case of optoelectronic devices we may need transparent films that are good heat conductors. Good heat conduction facilitates the dissipation of heat generated in a device. Unfortunately, the films used are typically metal oxides, such as silicon dioxide, which are usually amorphous in structure and thus have low thermal conductivity. However, even though oxide films may have low thermal conductivity, their thermal resistance, defined as the film thickness divided by the thermal conductivity, can be made sufficiently small for satisfactory device performance. In order to model heat dissipation in the aforementioned devices, designers need to know the thermal conductivities of the thin film materials involved. This part is a review of several methods of measuring thin film thermal conductivity that had been used in a round robin (see reference [1]).

B.2 Methods

B.2.1 General

Various methods have been used to measure the thermal conductivities of thin film oxide materials. This assessment reviews many of these measurement procedures. Several of these methods have been used in a round robin for measuring the thermal conductivity of silicon dioxide films on silicon wafer substrates. The films had been produced by oxidation of the wafers.

B.2.2 Three Omega (3ω) Method

The 3ω method has been used extensively to measure the thermal conductivities of bulk and thin film dielectric materials [2]-[8]. A detailed description of the experimental method has been given by Cahill [4]. The name arises because the method depends on measurements at the third harmonic of a fundamental frequency. In the context of this report, the fundamental frequency and the first harmonic denote the same frequency.

The method employs a thin metallic strip in intimate contact with the thin film surface of the specimen. A typical strip is made of evaporated aluminium with dimensions 200 nm thick, 30 μm wide, and 4 mm long. The strip is usually applied by use of a microlithographic method used to produce electronic microcircuits although it may also be applied by evaporation of the metal through a fine mask or stencil. Figure 2 shows a diagram of a mask in the shape of the strip and Figure 3 shows the measurement circuitry.

An AC electrical current modulated at angular frequency ω , $I_\omega = I_0 \cos(\omega t)$, is induced to flow in the strip, where t is the time. Electrical power is dissipated in the strip, $P = I^2 R$ causing the strip to heat up. The heating has both a DC component that changes the average temperature of the specimen and an AC component at 2ω that generates thermal waves in the specimen.

Because the electrical resistance of the strip depends on the temperature, the resistance will be modulated at 2ω as well. Therefore, there will be an AC voltage drop across the ends of the strip, $V_{3\omega}$, measured at angular frequency 3ω , that is proportional to the AC temperature variation of the strip $T_{2\omega}$, at angular frequency 2ω . $T_{2\omega}$ will depend on the thermal conductivities, λ , of the underlying materials. Thus, it is possible to extract λ of the film from a measurement of $V_{3\omega}$ if one knows the thermal conductivity and thermal diffusivity of the substrate. Modulation frequencies from about 100 Hz to several kilohertz are appropriate for measurements; however, measurement frequencies should be far from harmonics of 60 Hz.