



1. DESCRIPTION OF THE INSTRUMENT

The FOX50 instrument consists of the parallel round plates assembly with guard insulation cylinders, and a body where all electronics is contained including keypad, and liquid-crystal display (LCD). Schematic design of the Instrument's measuring part is shown on Fig.1. The upper plate is stationary and lower one can move up and down by pneumatic mechanism. Digital thickness readout sensor (not shown) monitors the position of lower plate. Once the lower plate is moved down, a sample can be placed between the two plates. Each time the stack is closed, the average thickness of the sample is determined within ± 0.025 mm (± 0.001 " resolution).

High-output Heat Flow Meters (HFMs, or transducers), developed in LaserComp, Inc., are bonded to the surfaces of both plates. The HFMs are made of hundreds of small thermocouples, so they provide high sensitivity and integration of the signals. Transducer's overall thickness is about 1 mm (~ 0.04 ").

Type E thermocouples are bonded in the center of each transducer. The thermocouples provide accurate readings of the both plates' temperatures. The same type external thermocouples also may be used to measure temperatures directly at the sample's surfaces.

24-bit Analog-to-Digital Converter (ADC) converts analog signals of the thermocouples and the heat flow meters to the digital domain with $0.6\mu\text{V}$ resolution, Resolution of the temperature measurements is $\sim 0.01^{\circ}\text{C}$ ($\sim 0.02^{\circ}\text{F}$).

Each plate has a powerful thermoelectric (Peltier) element, which is controlled independently. Other sides of the elements are cooled down by water flow (at $\sim 18^{\circ}\text{C}$ i.e. $\sim 64^{\circ}\text{F}$ or less), so temperatures of the plates can be independently maintained at any temperature from -10°C (14°F) to 110°C (230°F) with $\pm 0.02^{\circ}\text{C}$ ($\sim 0.04^{\circ}\text{F}$) stability.

The "brain" of the all FOX instruments' electronics is a powerful Digital Signal Processor (DSP). The DSP controls all aspects of the instrument operation.

One of the most important of the DSP's tasks is to perform a multistage PID algorithm to control temperatures of both plates. Correcting control signals are sent periodically to the Power Output Board, which provides the proper power to the Peltier elements of each plate.

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The DSP carries out all necessary actions (using special software written in Assembler language):

- control of pneumatic mechanism and determination of the sample's thickness;
- multiplexing ADC inputs;
- accurate PID control and stabilization of the temperatures of both plates;
- determination whether the thermal equilibrium has been reached or not using Equilibrium Criteria (see Section 8 "Thermal Equilibrium Criteria"), calculation of results, and then switching to the next entered temperature setpoint;
- transmission of the data to the personal computer through RS-232 serial interface.



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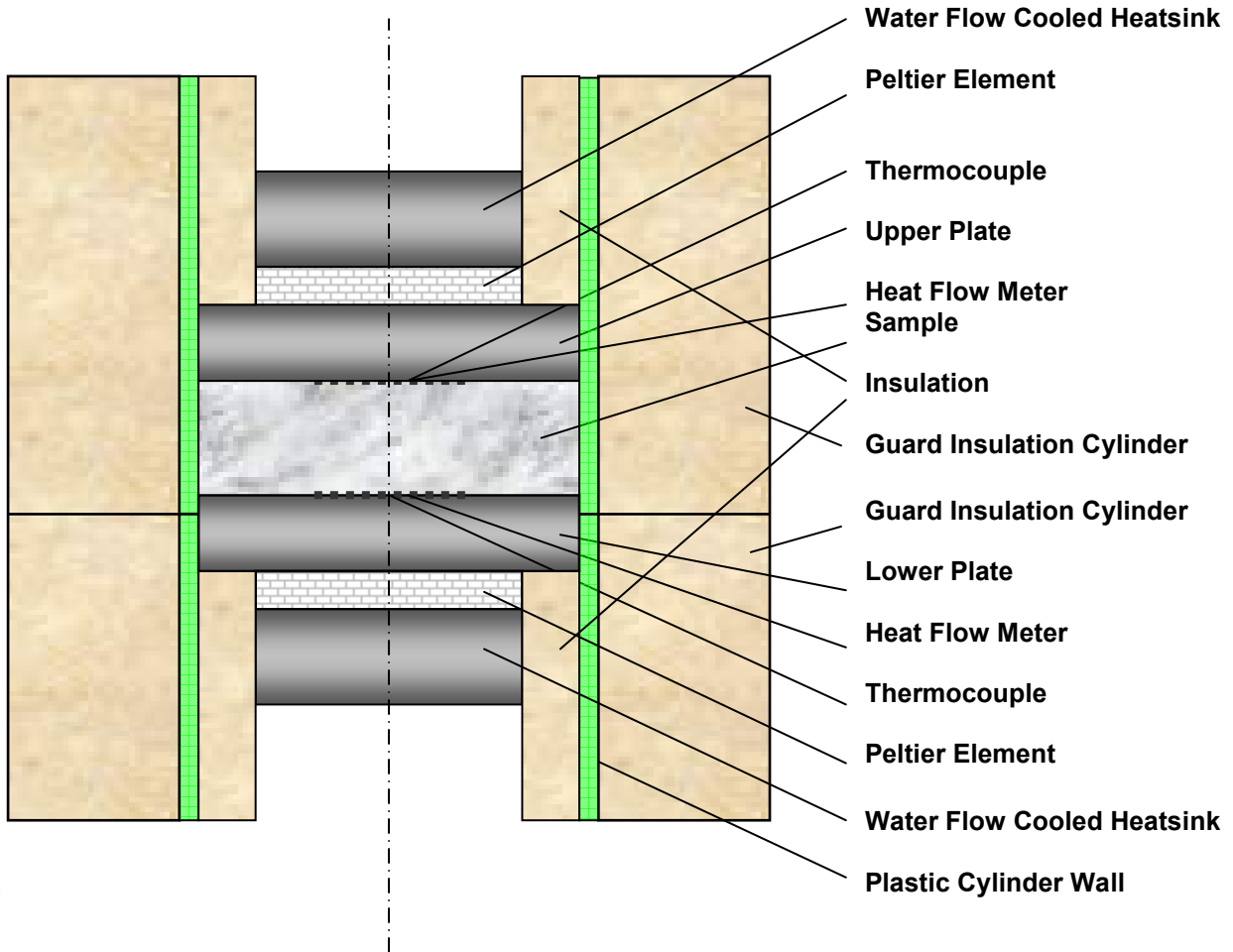


Fig. 1. Schematic diagram of FOX50 Heat Flow Meter Instrument for Thermal Conductivity Measurements (not all parts are shown). LaserComp, Inc.

2. THEORY OF THE TWO-THICKNESS METHOD

The general principle of the FOX heat flow meter instruments is based on one-dimensional equation for Fourier-Biot law:

$$q = -\lambda (dT/dx)$$

where q is heat flux (W/m^2) flowing through the sample, λ is its thermal conductivity ($W m^{-1} K^{-1}$) of the sample, dT/dx is temperature gradient ($K m^{-1}$) on the isotherm flat surface in the sample.

If a flat sample is placed between two flat isothermal plates maintained at different temperatures, eventually a uniform one-dimensional temperature field establishes within all the sample's volume (size of the plates is supposed to be much larger than thickness of the sample). The temperature gradient within the sample is equal to the difference between temperatures of its surfaces ΔT ($\Delta T = T_{hot\ surface} - T_{cold\ surface}$) divided by its thickness Δx , because in this case average temperature gradient dT/dx is equal to $-\Delta T/\Delta x$.

Thermal resistance of the flat sample R_{sample} is equal to its thickness Δx [m] divided by its thermal conductivity λ [$W m^{-1} K^{-1}$]:

$$R_{sample} = \Delta x / \lambda \quad [m^2 K W^{-1}] \quad (1)$$

In case of high thermal conductivity sample the temperatures of its surfaces are not equal to the temperatures of the instrument's plates because the thermal contact resistance is not much smaller than sample's thermal resistance, so:

$$T_{cold\ plate} < T_{cold\ surface} < T_{hot\ surface} < T_{hot\ plate}$$

$$\delta T + \Delta T_{sample} + \delta T = \Delta T_{plates}$$

The thermal contact resistance R is equal to temperature difference δT [K] between two contacting surfaces divided by heat flux q [W/m^2]:

$$R = \delta T / q \quad [m^2 K W^{-1}] \quad (2)$$

and depends on the types of adjoining materials, their surface roughness, and the interface pressure. Although the subject has been studied for a long time, still very little is known about the complex mechanism of heat transfer at the contact between two bodies [1,2].

Electric signal Q [μV] in heat flow meter instruments is proportional to the heat flux q [W/m^2], which is equal to temperature difference ΔT divided by sum of thermal resistance of the sample $\Delta x/\lambda$ and two thermal contact resistances $2R$:

$$Q = q / S_{cal} = \Delta T / [(\Delta x/\lambda + 2R) S_{cal}] \quad [\mu\text{V}] \quad (3)$$

All heat flow meter instruments must be calibrated. During the calibration the calibration factor S_{cal} is determined for each of the heat flux transducers (of upper and lower plates) using a standard material with well-known thermal conductivity $\lambda_{cal}(T)$. The S_{cal} [$\text{W m}^{-2} \mu\text{V}^{-1}$] is a proportionality factor between the heat flux q [Wm^{-2}] and the transducer's output signal Q [μV] - see Eq.3.

In case of samples having large thermal resistance (thermal insulators like Extruded Polystyrene, for example) the sample's thermal resistance $R_{sample} = \Delta x/\lambda$ is many times larger than the thermal contact resistance $2R$, so the latter can be neglected without loss of accuracy. Approximate formula for electric signal Q [μV] of regular heat flow meter instruments is simpler:

$$Q \cong \Delta T / [(\Delta x/\lambda) S_{cal}] \quad [\mu\text{V}] \quad (3a)$$

In case of samples having small thermal resistance (thin samples with fairly high thermal conductivity $> 0.3 \text{ W/mK}$) the thermal contact resistance on the sample's surfaces should not be neglected and the more accurate Eq.3 should be used. The thermal contact resistance $2R$ may even exceed the sample's thermal resistance $\Delta x/\lambda$ and may be a cause of considerable error if it is not taken into account.

Two unknown values (λ and $2R$) can be determined by using two specimens of the same material and different thickness Δx_1 and Δx_2 . Then we have system of two equations for heat flow transducer signals Q_1 and Q_2 containing two unknown values (we assume that the thermal contact resistances at the two surfaces for both samples are equal):

$$Q_1 = \Delta T / [(\Delta x_1/\lambda + 2R) S_{cal}] \quad (3.1)$$

$$Q_2 = \Delta T / [(\Delta x_2/\lambda + 2R) S_{cal}] \quad (3.2)$$

where ΔT is temperature difference, λ is sample's thermal conductivity, $2R$ is sum of two thermal contact resistances, Q_1 and Q_2 are signals from the heat flow transducers.



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During a Two-Thickness calibration run the FOX50 instrument's calibration factor S_{cal} and the thermal contact resistance $2R_{cal}$ on the standard's surfaces are determined. Standard with known thermal conductivity λ_{cal} should be used (i.e. Pyrex) - see sub-section "Formulas for Two-Thickness Calibrations".

During a Two-Thickness test run the sample's thermal conductivity λ and thermal contact resistance $2R_{test}$ on the sample's surfaces are determined – see sub-section "Formulas for Two-Thickness Tests".

Recommendation: Thicknesses of the two samples used for calibrations or for tests should be as different as possible, because error of calculations is inversely proportional to the *difference* of the thicknesses.

Two-thickness measurements also are used at National Institute of Standards and Technology (NIST) to get reliable thermal conductivity values of Pyroceram by Guarded-Hot-Plate apparatus [3].

Typical value of thermal diffusivity $a = \lambda / C_p \rho$ of materials like glasses or stones is about $(3-7) \cdot 10^{-7} \text{ m}^2\text{s}^{-1}$ ($C_p \rho$ is volumetric specific heat, C_p is specific heat at constant pressure, ρ is density). So, Fourier number (dimensionless thermal similarity parameter used in studying heat flow problems) $Fo = at / (\Delta x / 2)^2$ is about 7-16 per hour for 1" (25.4 mm) thick sample. So, it takes a fairly long time (not less than 0.5 hour for one-inch sample) to reach full temperature equilibrium to have $Fo \gg 1$. Experimental check showed that *average* value of two heat flow meters signals reaches equilibrium much faster than their *individual* values.

Formulas for Two-Thickness Calibration

Calibration done using two standards of the same material with different thicknesses Δx_1 and Δx_2 ($\Delta x_1 < \Delta x_2$) and known values of thermal conductivity $\lambda(T)$ (e.g. Pyrex 7740 or Pyroceram 9606 [4]) will provide correct values of the instrument's calibration factor S_{cal} excluding thermal contact resistance.

Thermal contact resistance $2R$ and calibration factor S_{cal} can be calculated using the solution of the system of Equations 3.1 and 3.2 where the known value of the standard's thermal conductivity λ_{cal} is used:

$$2R_{cal} = (\Delta x_2 Q_2 - \Delta x_1 Q_1) / [\lambda_{cal} (Q_1 - Q_2)] \quad [m^2 K/W] \quad (4)$$

$$S_{cal} = \Delta T \lambda_{cal} (Q_1 - Q_2) / [Q_1 Q_2 (\Delta x_2 - \Delta x_1)] \quad [W / (m^2 \mu V)] \quad (5)$$



Because physical properties of the heat flow transducers change with temperature, calibrations at several temperatures are necessary to get the temperature dependent calibration factor $S_{cal}(T)$. Thermal conductivity values of calibration standard materials at different temperatures are presented in Table 1:

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Table 1. Thermal conductivity values used for FOX50 instrument calibration

Temperature, °C	Pyrex 7740 (Powell, NBS)	Pyroceram 9606 (TPRC)	Vespel DuPont
0	1.058	4.149	0.365
10	1.073	4.091	0.368
20	1.087	4.036	0.371
30	1.102	3.985	0.374
40	1.116	3.937	0.377
50	1.131	3.892	0.380
60	1.145	3.851	0.383
70	1.160	3.811	0.386
80	1.175	3.775	0.389
90	1.189	3.741	0.392
100	1.203	3.710	0.396

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Dimension of the calibration factor is $W m^{-2} \mu V^{-1}$ or $W m^{-2} mV^{-1}$. A reciprocal value is used in some laboratories.

Each of the two transducers has its own temperature, so the calibration factor values should be referred to the transducers' actual temperatures. Two separate sets of the calibration factors (for upper and lower transducers) are measured during the calibration run.

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Formulas for Two-Thickness Tests

Tests (measurements of thermal conductivity) using two same material samples of different thickness Δx_1 and Δx_2 ($\Delta x_1 < \Delta x_2$) provide correct values of the thermal conductivity $\lambda(T)$ excluding the thermal contact resistance.



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The thermal contact resistance $2R$ and thermal conductivity λ can be calculated using solution of the system of Equations 3.1 and 3.2 where known value of instrument's calibration factor S_{cal} is used:

$$R = (\Delta x_2 Q_2 - \Delta x_1 Q_1) \Delta T / [Q_1 Q_2 S_{cal} (\Delta x_2 - \Delta x_1)]; \quad [m^2 K/W] \quad (6)$$

$$\lambda = S_{cal} Q_1 Q_2 (\Delta x_2 - \Delta x_1) / [\Delta T (Q_1 - Q_2)]; \quad [W/(m K)] \quad (7)$$

The tests can be performed at various mean temperatures within the temperature range of the calibrations, using the same ΔT and direction of the heat flow.

Checking two-thickness tests versus two-thickness calibrations using standard materials (Vespel test versus Pyrex calibration and vice versa, Pyroceram test versus Vespel calibration and vice versa, and Pyrex test versus Pyroceram calibration and vice versa) show that FOX50 instruments produce reliable thermal conductivity data.

Tests Using Only One Sample

One-sample tests can also be performed if the value of thermal contact resistance $2R$ is known. It should be determined beforehand using two samples from the same material but of different thickness with the same quality of the surface. In this case the thermal conductivity λ of the sample can be calculated using following formula:

$$\lambda = \Delta x / [\Delta T / (S_{cal} Q) - 2R]; \quad [W/(m K)] \quad (8)$$

The tests can be performed at various mean temperatures within the temperature range of the calibrations, using the same ΔT and direction of the heat flow.

One-sample tests can also be performed using the thermal contact resistance value obtained during two-thickness calibration of the instrument, but accuracy of this data can be low, especially for samples having a small thermal resistance.

Nevertheless, checking one-sample tests versus two-thickness calibrations using standard materials (Vespel test versus Pyrex calibration and vice versa, Pyroceram test versus Vespel calibration and vice versa, and Pyrex test versus Pyroceram calibration and vice versa) show that FOX50 instruments produce reasonable thermal conductivity data. Thermal contact resistance in these tests was assumed to be the same as during the two-thickness calibrations.

FOX50 Errors Estimation

Errors of FOX50 Instrument can be estimated using well-known standard procedure and formulas for calibration factor S_{cal} (Eq.5) and thermal conductivity λ (Eq.7). Edge heat losses are assumed negligible comparing to main heat flow through sample (special analysis showed that *average* value of thermal conductivity is much less sensitive to the edge heat losses than *individual* plate's values).

Second lines (percentage) show approximate error values for each measured quantity:

1) Calibration factor:

$$S_{cal} = \Delta T \lambda_{cal} (Q_1 - Q_2) / [Q_1 Q_2 (\Delta x_2 - \Delta x_1)] \quad [W/(m^2 \mu V)] \quad (5')$$

Calibration factor error estimation: $\delta S_{cal} / S_{cal} =$

$$\begin{aligned} & \{ (\delta \Delta T / \Delta T)^2 + (\delta \lambda_{cal} / \lambda_{cal})^2 + [\delta (Q_1 - Q_2) / (Q_1 - Q_2)]^2 + \\ & \{ (\sim 0.2\%)^2 + (\sim 3\%)^2 + 2 * (\sim 2) * (\sim 0.5\%)^2 + \\ & + (\delta Q_1 / Q_1)^2 + (\delta Q_2 / Q_2)^2 + [\delta (\Delta x_2 - \Delta x_1) / (\Delta x_2 - \Delta x_1)]^2 \}^{1/2}; \\ & + (\sim 0.5\%)^2 + (\sim 0.5\%)^2 + 2 * (\sim 2) * (\sim 0.2\%)^2 \}^{1/2} = \\ & = \sim 3.27\% \end{aligned}$$

2) Thermal conductivity:

$$\lambda = S_{cal} Q_1 Q_2 (\Delta x_2 - \Delta x_1) / [\Delta T (Q_1 - Q_2)]; \quad [W/(m K)] \quad (7')$$

Thermal conductivity error estimation: $\delta \lambda / \lambda =$

$$\{ (\delta S_{cal} / S_{cal})^2 + (\delta Q_1 / Q_1)^2 + (\delta Q_2 / Q_2)^2 +$$



$$\begin{aligned} & \{ (\sim 3.27\%)^2 + (\sim 0.5\%)^2 + (\sim 0.5\%)^2 + \\ & + 2 [\delta(\Delta x_2 - \Delta x_1) / (\Delta x_2 - \Delta x_1)]^2 + (\delta \Delta T / \Delta T)^2 + 2[\delta(Q_2 - Q_1) / (Q_1 - Q_2)]^2 \}^{1/2}; \\ & + 2 * (\sim 2) * (\sim 0.2\%)^2 + (\sim 0.2\%)^2 + 2 * (\sim 2) * (\sim 0.5\%)^2 \}^{1/2} = \\ & \approx 3.5\% \end{aligned}$$

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We assumed here that samples' thicknesses and signals ratio is about 2 because the estimated error values slightly depend on the two samples' thickness difference or ratio.

So, value of the precision of the FOX50 Instrument is of the same order of the accuracy of the calibration standard materials which is typically about ~3% at moderate temperatures. FOX50 is comparative-type instrument, and, naturally, its precision does not exceed the accuracy of the calibration standard.

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