Chapter 6

The Review of Some Commonly Used Methods and Techniques to Measure the Thermal Conductivity of Insulation Materials

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Additional information is available at the end of the chapter

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Abstract

The use of insulation materials is considered as one of the most effective means of conserving energy in various fields. Thermal insulation materials enable systems to achieve energy efficiency. Many different thermal insulation materials have been developed to reduce heat flow by limiting conduction, convection, and/or radiation while performing one or more functions. These functions may vary in the context of thermal design, numerical simulations, and a wide range of engineering problems, such as determining the heat loss, temperature field, isolation, and cooling conservation, and in a variety of other technologies. One of the most effective ways to identify and determine performance is effective thermal conductivity. The thermal measurement performance is usually evaluated in a temperature and signal gradient for single or combined homogeneous/heterogenous materials. The two main categories of thermal conductivity measurement techniques are steady-state methods and transient methods. The aim of this chapter is to present various measurement methods and to investigate their suitability for method purposes. This chapter presents new and accurate experimental techniques and methods for measuring the thermal conductivity of several most commonly used insulation materials. Some of these methods are commonly used in the field for measuring the thermal property of insulation materials. On the other hand, different insulation measurement practices are presented depending upon the overall structures. The analysis predicting the thermal conductivities of insulation materials is also discussed.

Keywords: heat transfer, insulation material, mathematical model, measurement method, tests, thermal conductivity

1. Introduction

The development of technologies leads to changes in demand. Insulation materials are created in several forms including porous form, blanket or batt form, rigid form, natural form, foamed structure, and reflective structure. Fiber and polymer products are the most commonly used types of thermal insulation. Many studies have examined the impact of various parameters on the thermal performance of insulation materials. Extensive investigations have focused on heat transfer in these materials in the context of their numerous and varied applications. The thermal conductivity in these applications is one of the most important challenges facing thermal, mechanical, material, and civil engineers. In various fields, the accuracy of different techniques for evaluating thermal conductivity and other properties is widely debated as a fundamental parameter. As a result of the wide range of thermal properties of insulation materials, there is no single measurement method for all thermal conductivity measurements [1].

The accuracy of manufacturers' claimed values for thermal properties is sometimes questionable since the thermal data for certain materials are often incomplete and lacking in important information. When values for insulation types are quoted, manufacturers do not always report the density and temperature of the materials tested. In general, the "effective" thermal conductivities of the materials depend on the constituents and/or voids present in the different characteristics of their structures as well as the density and temperature of the material.

In the field of thermal insulation technology, many developments during the last two decades have enhanced the accuracy of measurement techniques as well as the current understanding of the principles of heat transfer through different materials. These techniques are thus differentiated mainly by the range of thermal conductivity, the range of material types, measurement time, measurement accuracy, specimen type, and temperature range.

In the study, a survey concerning available experimental techniques for the measurements is conducted. The main focus is on descriptive measurement methods, and their ranges of the thermal conductivity and temperature are determined. The objective is to analyze a measurement apparatus designed to determine effective thermal conductivity of insulation materials. The other aim of this chapter is to figure out the models for the effective thermal conductivity of insulation materials. The prediction of the property has been determined using experimental and analytical models in different studies. The accuracy of any method and model is limited by physical properties and other factors. However, measurement and modeling of thermal conductivity are difficult and require high precision in the determination of the various parameters involved in the calculations. To analyze thermal behavior of materials, the methods and the models must be clearly known and defined.

Insulation materials such as natural or man-made materials differ concerning the material structure and the range of use. To develop insulation materials in an economically and environmentally friendly manner, it is essential to have knowledge and control over their thermal conductivities. The properties can vary with temperature, pressure, and composition, affecting the transfer of heat. To answer the following questions, thermal conductivity must be known [2].

- How is a particular insulation material performing?
- How does performance change with the weather and different conditions, and how can the performance be improved?
- What is the optimum insulation for technologies/systems operating under different temperature, gas, or pressure conditions?
- How can a system be designed to achieve the required efficiency, and what are the best materials to use?
- What is the heating/cooling load of a building and a structure?
- How can cryogenic tanks be insulated in the best possible way?
- How can the heat transfer from an electronic component be improved?

There are a number of methods to measure thermal conductivity. In general, there are two basic techniques for measuring thermal conductivity: steady-state methods and transient or non-steady-state methods [1, 3]. Each of these methods is suitable for a limited range of materials, and they are based on the fundamental laws of heat conduction and electrical analogy. Steady-state methods have been traditionally used since they are mathematically simpler. There is an important distinction between steady-state and transient techniques [4, 5]. Transient heat transfer methods are capable of directly determining thermal diffusivity, whereas steady-state methods are considered to be more accurate than transient methods for testing dry materials [6].

The **steady-state technique** records a measurement when a tested material's thermal state reaches complete equilibrium [5]. A steady-state condition is attained when the temperature at each point of the specimen is constant and the temperature does not change with time. A disadvantage, however, is that it generally takes a long time to reach the required equilibrium [4, 5]. The method involves expensive method apparatus since a well-designed experimental installation system is usually needed. Nevertheless, it is the primary and most accurate measurement method.

The **non-steady-state or transient** technique records a measurement during the heating process. The method determines thermal conductivity properties by means of transient sensors. These measurements can be made relatively quickly, which garners an advantage over steady-state techniques [4, 5, 7]. For this reason, numerous solutions have been derived for the transient heat conduction equation by using one-, two-, three-dimensional geometries [7]. Transient methods generally employ needle probes or wires [4].

Compared to electrical and thermal transport, the ratios of thermal conductivities of the best conduction and insulation conditions are significative and determinative magnitudes. Therefore, instruments for thermal property identification are often designed only for specific kinds of materials or temperature ranges. **Table 1** presents a comparison of the most common methods of thermal conductivity measurement [7]. Measurement systems can also be divided into three categories based on the operating temperature of the apparatus: (1) room temperature operation (20–25°C), (2) below room temperature operation (down to about –180°C), and

(3) high-temperature operation (up to 600°C or above) [8]. A given measurement system is often optimized for one of these temperature ranges.

Method		Temperature range	Uncertainly	Materials	Positive	Negative
Steady-state methods	Guarded hot plate	80–800 K [7], −180–1000°C [9], 80–1500 K [10]	2% [7] and 0.0001–2 W/(m K) [9] 2–5% and 0.0001–1 W/(m K) [10]	Insulation Materials [7] and solid, opaque, insulators [10]	High accuracy	Long measurement, large specimen size, low conductivity materials
	Cylinder	4–1000 K [7]	2% [7]	Metals [7]	Temperature range, simultaneous determination of electrical conductivity	Long measurement
	Heat-flow meter	−100–200°C for normal [7] 90–1300 K for axial heat flow and 298–2600 K for radial heat flow [10]	3–10% [7] 0.007–1.0 W/(m K) [9] 0.5–2% and 10–500 W/(m K) (axial) and 3–15% and 0.01–200 W/(m K) (radial) [10]	Insulations, plastics, glasses, ceramics [7] Some metals, rocks, polymers [9] Metals and Solids [10]	Simple construction and operation	Measurement uncertainly, relative measurement
	Comparative	20–1300°C [7] 0–1000°C [9]	10–20% [7] and 0.2–200 W/(m K) [9]	Metals, ceramics, plastics [7]	Simple construction and operation	Measurement uncertainly, relative measurement
	Direct heating	400–3000 K [7, 10]	2–10% [7] 2–5% [10] and 10–200 W/(m K) [10]	Metals [7] Wires, rods, tubes of electrical conductors [10]	Simple and fast measurements, simultaneous determination of electrical conductivity	Only electrically conducting materials
	Pipe method	20–2500°C [7] and 50–800°C [9]	3–20% [7] and 0.02–2 W/(m K) [9]	Solids [7] calcium silicates,	Temperature range	Specimen preparation, long measurement

Method		Temperature range	Uncertainly	Materials	Positive	Negative
				mineral and refractory fiber blankets) [9]		time
Transient Methods	Hot wire, hot strip	20–2000°C [7], −40–1600°C for hot wire and −50 to 500°C for hot strip [9] 298–1800 K [10] for hot wire	1–10 % [7] 0.001–20 W/(m K) for hotwire and 0.1–5 W/(m K) for hot strip [9] 5–15% [10] and 0.02–2 W/(m K) for hotwire [10]	Liquids, glasses, solids of k_{low} [7] refractory materials [9, 10] and plastics, granules, powders for hotwire [9] and glasses, foods, ceramics for hot strip [9]	Temperature range, fast, accuracy	Limited to low conductivity materials
	Hot-disk (TPS technique)	30–1200 K [7]	-	liquids, pastes, solids and powders	very short time accuracy, different thermal properties simultaneously	the range of 0.005 and 500 W/(m K) (conducting or insulating material)
	Laser flash	−100–3000°C [7] and 100–3300 K [10]	3–5% [7] 1.5–5 % [10] and 0.1–1500 W/(m K) [9, 10]	solids, liquids, and powders [7] and liquid metals, polymer, ceramics [9, 10]	Temperature range, most small specimen, fast, accuracy at high temperature	Expensive, not for insulation materials
	Photothermal (PT), Photoacoustic	30–1500 K [7] −50–500°C [9] and 200–800 K [10] for PT	Not sufficiently known [7], 1–10 % [10] 0.1–200 W/(m K) for PT [9, 10]	Solids, liquids, gases, thin films [7], small parts of most solid [9,10]	Usable for thin films, liquids, and gases	nonstandard, knowledge about accuracy

Table 1. Comparison of measurement methods for detecting the thermal conductivity [7, 9, 10]

For measuring thermal conductivity, there are four main types of measurement setups: the guarded hot plate (GHP), the heat-flow meter (HFM), the hot wire, and laser flash diffusivity. The usage of these tools/methods differs in technique, material type, intended specimen size, measurement time, capability, and measurement methodology [5].

To analyze heat transfer behavior of insulations, a guarded hot plate or a heat-flow meter is usually used. The hot-wire and flash method use special devices for consolidated insulation specimens. The laser flash method is often employed for highly conductive ceramics, metals, and some composites [2]. The thermal conductivity of large specimens of refractory material is measured by using hot-wire systems [2]. **Figure 1** provides a comparison of measurement methods and material types for the ranges of thermal conductivity [2].

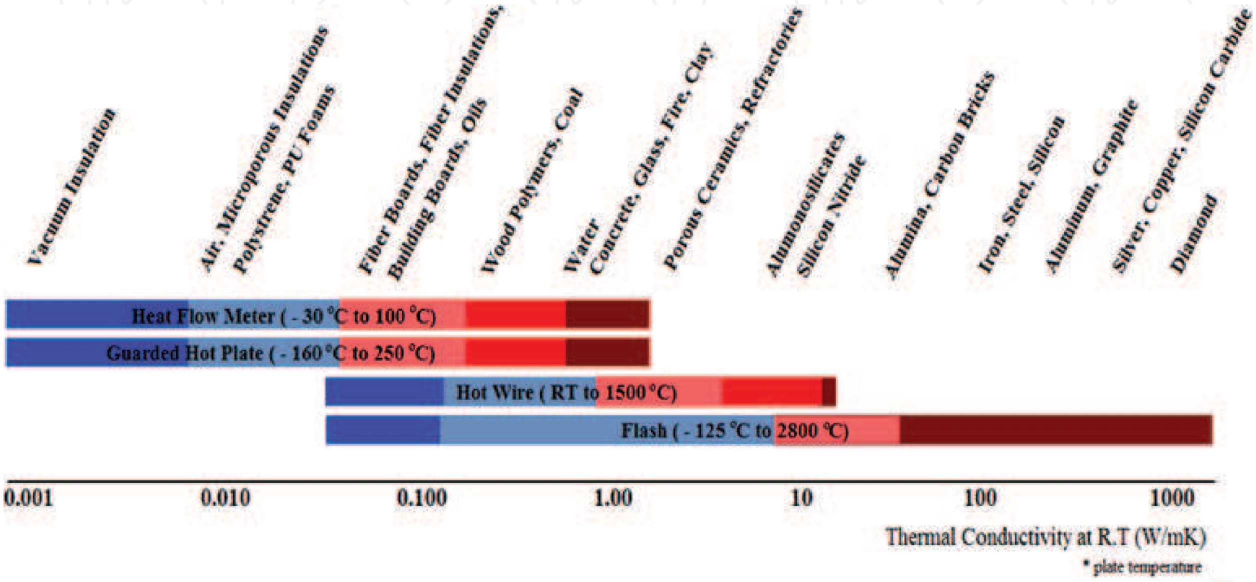

Figure 1. Comparison of measurement methods and material type for the ranges of thermal conductivity [2].

2. Steady-state methods

Steady-state methods apply Fourier's law of heat conduction to measure thermal conductivity. The solution to the problems with the different steady heat-flow methods is to convert the heat transfer problem to a one-dimensional problem, thus simplifying the mathematics. The calculations change for the models of an infinite slab, an infinite cylinder, or a sphere. The typical specimen geometry, the configuration of a measurement system, and the magnitude of the thermal conductivity are used to distinguish between different types of thermal conductivity measurements. The thermal magnitude of the measuring object is determined by the following measuring techniques using the direction of the heat flow, the conservation of the heat flow, and an auxiliary layer having a known thermal property.

2.1. Guarded hot plate

The guarded hot plate, also known as the Poensgen apparatus [11], is the most commonly used and most effective method for measuring the thermal conductivity of insulation materials. The

GHP relies on a steady temperature difference over a known thickness of a specimen and its primary purpose is to control the heat flow through the material. One disadvantage is that establishing a steady-state temperature gradient through a specimen is time-consuming when using the GHP and other steady-state techniques. Other potential disadvantages are that the temperature gradient must be relatively large, the specimen width must be large, and also that the contact resistance between the thermocouple and the specimen surface poses a major source of error [12]. Although Reference [12] cites large specimen size as a potential disadvantage, size is usually not a serious issue [8].

The experimental setup of the guarded hot plate employs a steady-state heat transfer between a hot plate and a cold plate. However, the accuracy of this method is questionable, interlaboratory comparisons of GHP calculations have revealed discrepancies among 20 different GHPs used at different times [5]. The individual results of these 20 GHPs diverged significantly from reference values, ranging from +13 to −16% [5, 11].

Despite these disadvantages, the standardized GHP method is the ideal apparatus for researchers and scientists in the field of insulation testing and it is considered an absolute measurement method. The practical applicability requires careful consideration of the array content: (a) attaining steady-state conditions; (b) the unidirectional heat flow in the area under analysis, the temperatures of the hot and cold surfaces, and the specimens' thickness; and (c) other factors influencing the unidirectional heat flow [8].

Another advantage is that the GHP method is standardized in countries such as the United States (ASTM C 177-63), Great Britain (B.S. 874:1965), and Germany (DIN 52612) [13]. The details of this method are provided by the American Society for Testing Material (ASTM) Standards associated with the method and/or materials [1]. The details of this standard are partly based on the difficulty of attaining steady-state conditions [11], accurately adjusting the temperatures in conventional plates (guarded, hot, and cold), and design conditions.

2.1.1. The construction of the guarded hot plate

The guarded hot-plate measurements are analyzed on the fundamental of the heat transfer in the infinite slab geometry. Since specimen dimensions are finite, unidirectional heat flow is achieved through the use of guard heaters. The temperature of a thermal guard is maintained at the same temperature as its adjacent surface (which is considered as an auxiliary heater/heat sink), in order to prevent heat loss from the specimen and heat source/heat sink, and as a result, unidirectional heat flow is attained [1]. After a steady state is reached, the heating and cooling plates have stable temperatures. Then the thermal conductivity can be determined based on the heat input, the temperature difference through the specimen, the thickness of the specimen, and the size of the metered area of heat transfer. Steady-state conditions may change with respect to specimen type, specimen size, and mean temperature [14]. The GHP is most suitable for dry homogeneous specimens [15], but it is unsuitable for materials in which there is a potentiality for moisture migration [16].

The guarded hot-plate setup is comprised of cold plates, a hot plate, a system of guard heaters, and thermal insulation. Hot plate is electrically heated and the cold plates are Peltier coolers or liquid-cooled heat sinks. The configuration is arranged symmetrically, with guarded hot plates located on the sides while the heater unit is sandwiched between two specimens or a single specimen and an auxiliary layer (**Figure 2**). The different types of guarded hot-plate apparatus are shown in **Figure 2**. In the single-sided system state, the heat flow passes through one specimen, while the top of the main heater acts as an insulating guard, thus ensuring an adiabatic environment [8].

In the two specimen apparatus, the main advantage is that heat loss from the hot plate can be controlled more effectively due to the symmetrical arrangement of the specimen on each side of the heater. Unlike the single-specimen method, the symmetrical setup can be used for investigating solid materials. For measuring the conductivity of nonsolid materials, it is necessary to heat the specimen from the top in order to avoid convection [7].

The electrical heating is placed into the plates in a certain shape or form, such as a square or a circular shape. The guarded plate (ring), the central plate (metered area), and the auxiliary heater can all be arranged in this manner. The apparatus must test two specimens simultaneously in the form of a slab with a standard size (such as 300 mm × 300 mm or different sizes). A fixed heat rate must be applied by an electric heater. This arrangement produces a heat flow across the two specimens, flowing outward toward two plates chilled by a Peltier or a liquid cooling system.

These heat measurements are recorded by differential thermocouples, which are instruments that control a flat electrically heated metering area that is surrounded on all lateral sides by a guard heater section. The heated section provides the planar heat source applied to the hot face of the specimens [8]. Heat is supplied to the metered area (the central heater) at an assigned heat power rate. The temperature of the guard heater is maintained at the same temperature as the metered section by using a control system. The adjacent thermal guard surfaces and/or plates are held at the same temperature range, and ideally no heat leakage occurs from the source, the specimen, or the boundaries. This is aimed at ensuring a one-dimensional thermal

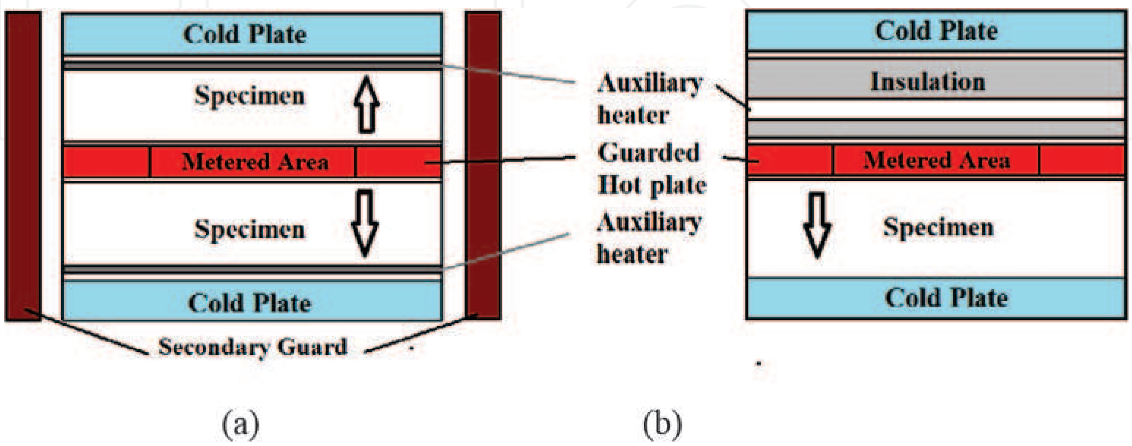

Figure 2. The apparatus of guarded hot-plate method for thermal conductivity measurement. (a) two specimen with/without auxiliary heaters and secondary guards, (b) single-specimen [7].

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heat flow in the actual and practical test section, corresponding solely to the central metered heater. In addition, the apparatus is surrounded by thermal insulation, as well as guard heaters. And, the hot/cold metal parts are positioned between the heaters/cooling plates and each specimen. The parts matched the same frame design are adjacent to the related side (hot or cold) temperature sensors [13]. A data acquisition system is connected to the temperature sensors and the electrical power supply devices, which are in turn controlled by a closed-loop control system.

2.1.2. Principle of operation

The specimens of the homogeneous material with the same thickness are interposed between the hot guard heaters and the cold plates. For two specimen apparatuses, the auxiliary heaters may be placed above and below the specimens. A well-defined, user-selectable temperature difference is established between the hot and the cold plates. The power rate input in the hot plate with metered area A is measured when thermal equilibrium is reached at steady-state conditions. When the control system is used, the plate temperatures reach stability.

It is assumed that the measured heat power rate is transferred across the specimen due to guarded heaters. After thermal equilibrium has developed and the heating and cooling plates are kept in stable temperatures, the thermal conductivity can be calculated from the input values. The input values are the heat power Q, the temperature differential across the specimen ($T_{hot} - T_{cold}$), the specimen thickness (Δx), and the heat transfer area (center metered area, A). The thermal conductivity is computed by measuring the quantity of heat input under the steady-state temperature profile in the entire specimen [1, 3, 14]. From the measured input values, the effective thermal conductivity can be calculated using the following unidirectional steady-state heat transfer equation:

$$k_{eff} = \frac{Q}{2}\frac{\Delta x}{A.\Delta T} \quad (1)$$

where the heat flow Q is obtained by measuring a power P (or half power for two specimen) generated in an electrical heater. The heat conduction equation for homogenous isotropic materials without using internal heat generation is given for the steady state in Eq. (1). These methods depend on Fourier-Biot law of heat conduction [1, 3, 14]. Its modified equation forms can be used for one-dimensional steady heat flow across different sizes, such as plate, cylinder, and sphere.

For the shapes in cylinder forms, radial heat-flow steady-state methods are observed. The specimen completely encloses the heating source in this method, eliminating end losses. The lateral effects are assumed to be insignificant either because the ratio of length to diameter of the test apparatus is large or because guard heaters are used. It is assumed that the surface of the central heater at a diameter r_1 and the outer specimen surface at diameter r_2 reach the same temperature after the steady state is established. The thermal conductivity can be determined based on “the heating power, the length of the cylinder, the temperature differential between two internally located sensors, and their radial position" [16]. Because of practical application difficulties, the cylinder (and sphere) method is not popular. Nevertheless, this method is applied and used to measure thermal conductivity using the cylinder shape method.

The guarded hot-plate method under a vacuum is based on an absolute measurement method for research and therefore requires no calibration standards. Furthermore, this can be seen as an absolute measurement, regardless of vacuum conditions. The plate system is placed in a vacuum medium. The measurements can be carried out under a vacuum as well as under atmospheric or defined pressure levels. The system requires a symmetry and two specimens for each test. With a guarded heater and/or thermal insulation, a relative uncertainty of 2% for thermal conductivity measurements of can be achieved. Each plate and the guard ring/heater are connected to a separate control system with temperature sensor(s) and an assigned power supply.

The guarded hot-plate and cylinder method exemplify a measurement principle that has been optimized for different ranges of thermal conductivity. The guarded hot-plate method can be used to test the thermal properties of nonmetals such as thermal insulation materials, polymers glasses, and ceramics, as well as liquids and gases in the temperature range between about 80 and 800 K [7]. The thermal conductivities of metals (approximately up to 500 W/(m K) in a temperature range between about 4 and 1000 K) can be tested via the cylinder method of employing axial heat flow. The GHP method is appropriate for these kinds of metal because the determination of the temperature difference is the main challenge when measuring materials with high thermal conductivity (e.g., metals). In these kinds of tests, the contact resistances between the specimen and the heater or the cold plate must be considered [7].

2.2. Heat-flow meters

The use of heat-flow meters is based on substantially the same principles as other measurement techniques, but it is not identical to them [13, 17, 18]. The main disadvantage of the guarded hot-plate methods is that they are very time consuming, as stated above. In contrast, heat-flow meters are accurate and fast apparatuses, and the operation of these apparatuses is easy for measuring the thermal conductivity of low-conductivity materials [2]. The method is based on the improvement in accuracy and speed of the measurement. The maximum temperature limits are approximately 200°C for the heat-flow meter method [7] and about 100°C in practical applications [2].

The heat-flow meter is described in various standards for tests. The heat-flow meter design resembles to setup of the single-specimen guarded hot-plate apparatus. The basic idea of the heat-flow meter is deducing the heat flux based on the measurement of a drop in temperature throughout a thermal resistor. The way for heat flux measurement is carried out either by using a certified well-known reference specimen or a heat flux sensor.

The specimen is placed between two plates held in different temperatures, with one being heated and the other plate being cooled, as shown in **Figure 3.** Instead of using a main heater as in the guarded hot-plate method, heat flux transducers are used to measure the heat flow through the specimen. The heat flux is determined in a current with the measurement of a voltage drop through an electrical resistor. Sensors provide an electrical output signal. The measured signal and the change in thermovoltage is proportional to the drop in temperature drop occurring throughout the plate.

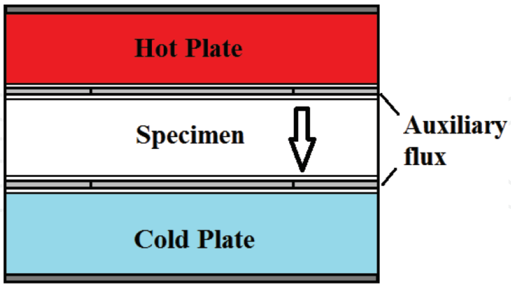

Figure 3. Schematic design of heat-flow meter basically.

Heat flux sensors are most often composed of a series of connections of thermocouples spanning a thermal resistor, e.g., a thin ceramic or plastic plate [7]. A second heat flux is occasionally applied at the cold plate is in order to measure radial heat loss and also to reduce the time needed for the measurement. This reduction in time poses an advantage for this method when measuring of insulation materials. The temperature of the plates is measured and adjusted to the desired set point when reaching a constant value. Steady-state conditions occur when the amount of heat flux is equal at each point of the layered system. After thermal equilibrium is established, the test is determined under the conditions. In order to calculate thermal conductivity, the steady-state temperatures, the specimen's thickness, the specimen's metered area, and the heat flux input to the hot plate are used. The heat flux output is usually calibrated with various reference standards, e.g., in a guarded hot-plate apparatus.

Compared to the guarded hot-plate method, which is an absolute method, the heat-flow meter method is comparative and thus can be considered to be a relative method. Insulation materials and polymers ($k < 0.3$ W/(m K)) are usually tested via the heat-flow meter method, and sometimes it is used for glasses and ceramics and for other materials with thermal conductivities lower than about 5 W/(m K) [7]. For insulation materials at about room temperature, the measurement uncertainties are approximately 3%, and at high temperatures the uncertainties are between 10 and 20% [7].

Steady-state stability can be reached in short times, resulting in productivity gains. The stable case, however, is valuable for providing repeatability, the long-term consistency of a specimen.

2.3. Direct heating method

Two disadvantages of steady-state methods are the lengthy time requirements and the difficulty of determining heat loss, especially at high temperatures. These disadvantages can be overcome by the direct heating method, which can be used for electrically conductive materials such as metals.

The specimen, such as a wire, pipe, or rod, is placed in a vacuum chamber, clamped between two heat sinks cooled by liquid, and the specimen is heated up to temperatures in the range of 300–4000 K [7]. **Figure 4(a)** portrays the schematic of the design of the direct heating method. Voltage drops and temperatures are measured: in the middle of the rod and on each end of the rod. From these three measurements obtained in the direct heating method, the thermal conductivity and the specific electric resistivity *k* is given as [7]

$$k\frac{V_h A}{I_h l} = \frac{(V_3 - V_1)^2}{4\left[2T_2 - (T_1 + T_3)\right]} \tag{2}$$

where l and A are the length and cross-sectional area of the specimen. I_h and V_h are heating current and voltage drop.

2.4. Pipe method

The pipe method takes advantage of a radial heat flow in a cylindrical specimen. A core heater, which is a tube, rod, or wire, is inserted into the central axis of the pipe-shaped specimen. There are heaters at both ends of the specimen. The combination of the specimen and heaters is surrounded by thermal insulation and then a water jacket or a liquid-cooled heat sink. **Figure 4(b)** shows the schematic and components of the pipe method. End guard heaters can be used to minimize axial heat loss, and also increasing the specimen's ratio of length to

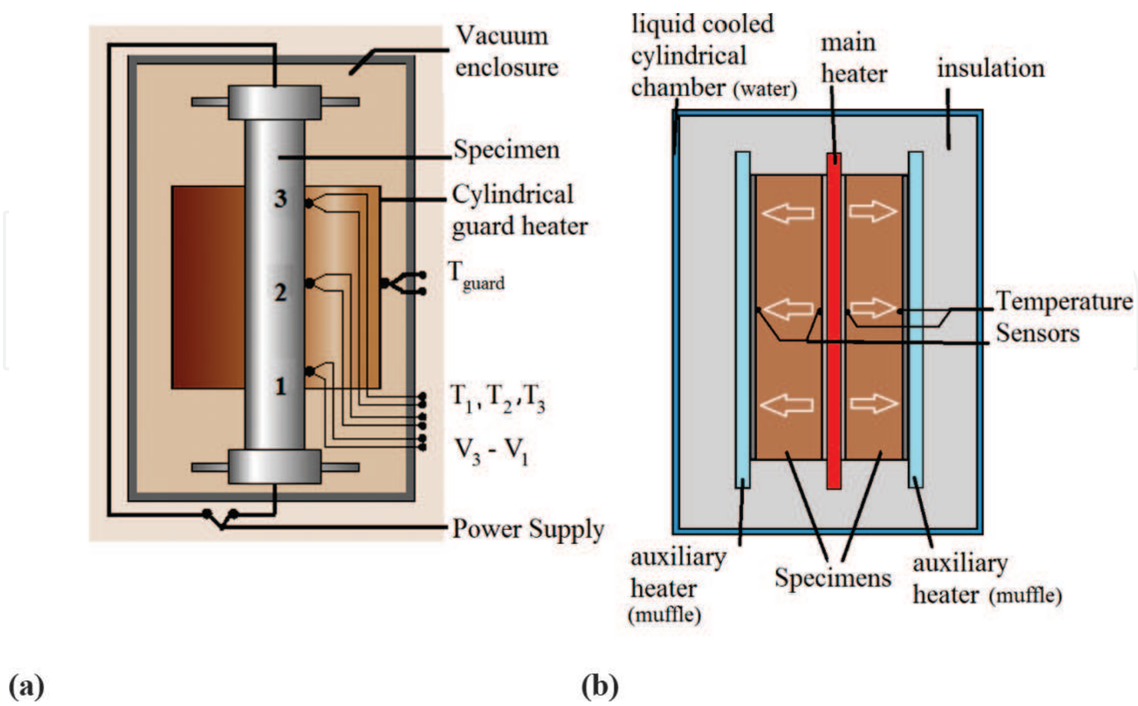

Figure 4. (a) The schematic design of the direct heating method [7], (b) the schematic design of the pipe method.

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diameter can achieve the same purpose. The thermal conductivity is obtained by measuring the radial heat flow ϕ [7]:

$$k = \frac{\phi ln\left(d_2 / d_1\right)}{2\pi\left(T_1 - T_2\right)} \tag{3}$$

This technique has been modified for use with a variety of solids, ranging from insulation materials (20 mW/(m K)) to metals (200 W/(m K)) and evaluating for temperatures between room temperature and 2770 K [7]. This technique can be modified to collect data over time for the simultaneous measurement of thermal conductivity and thermal diffusivity, and these kinds of transient methods are gaining increased interest.

Advantages of the steady-state methods to other methods are as follows:

- simple mathematical expression,
- absolute and primary method for low conductivity specimens,
- acceptable time consumption,
- partially suitable for powdered, granular, or solid forms,
- uncertainties of 1–2% for insulations near room temperatures,
- acceptable small test specimens (except for concentric sphere).

Disadvantages of the steady-state methods to other methods are as follows:

- complexity of the apparatus giving high accuracy,
- uncertainties of 10% or higher to the conditions,
- time consuming,
- immeasurable error due to contact resistance,
- difficulty of measuring geometrically shaped specimens (concentric cylinder or concentric sphere),
- heat losses from especially in parallel plate and concentric cylinder methods,
- difficulty of measurement of heat-flow value for two specimens,
- use error of specimens containing moisture.

3. Transient methods

The advantages of transient methods are mainly distinguished by the short amount time needed, so that various thermal values can be determined in the measurement process.

Therefore, this method is based on a signal measurement and an acceptably small temperature differential. The transient technique is measured by evaluating the feedback response after a signal is transmitted to the specimen for heat generation in the specimen. Therefore, test time is obtained in a few minutes or a subsecond time intervals for transient methods. This method is also more appropriate for high moisture content materials because of the signal and response in the specimen. In many cases, it is possible to replace the temperature measurements at two opposite surfaces with a measurement as a function of time at only one position on the specimen [7].

Among transient methods, the hot-wire and the laser flash methods are commonly used for measuring the thermal conductivity of different materials provided in **Table 1**. A modification of the hot-wire method is the hot-strip or disk technique, which can be applied to solid nonelectrically conducting materials in order to measure the thermal diffusivity and conductivity [12].

3.1. Hot-wire method

This method is a transient technique based on recording the rise in temperature at a defined distance from the heat source. The hot-wire technique is a good method to determine the thermal conductivity of liquids. In the hot-wire method, the specimen preparation is simplified by the use of a heat source, except in the case of solids. When solids are being tested, the wire is situated between two equally sized homogeneous specimens, as shown in **Figure 5**. The hot wire is embedded in small channels, because it is important to ensure sufficiently low contact resistance between solid specimens and the heating wire [7]. For this reason, the hot-wire method is avoided in favor of an increasingly popular variation, the hot-strip method, which is used for measurements on solids.

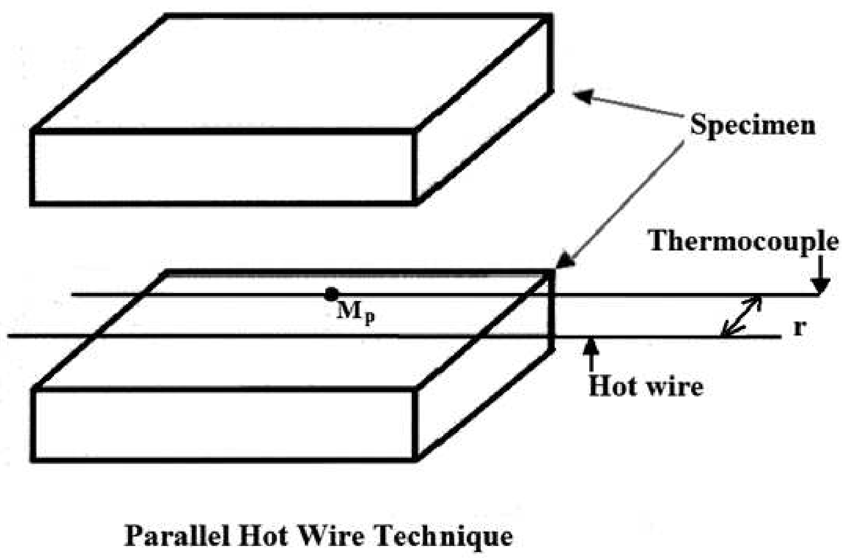

Figure 5. The schematic principle and the design of the hot wire.

In the standard transient hot-wire technique (**Figure 5**), the wire system (often made of platinum) is recorded as two functions of a temperature sensor and heater [7]. One modification of this technique is the probe method, which uses a probe instead of a wire. This probe configuration is useful whenever the specimen conductivity is calculated based on the response of a probe inserted into the specimen. A similar theory underlies both the non-steady-state line heat source (hot wire) method and the probe method for the measurement of thermal conductivity. Both methods are practical for measuring the thermal conductivity of "biological materials, insulations, rocks, ceramics, foods, soils, and glass over a wide range of temperatures" [9]. Thus, the probe method is applied to low-conductivity materials in powder or semirigid form. A closely controlled furnace is used to produce the base temperatures for the tests of the thermal properties of the specimen, soils, in situ [10].

Thermal conductivity is calculated by comparing the plot of the wire temperature versus the logarithm of time, as long as density and capacity are given or measured. The hot-wire method is also capable of measuring the thermal conductivity of gases as well as refractories, such as insulating bricks, fibrous, or powder materials [10]. The technique can be adapted for measuring the properties of liquids and plastics of relatively low thermal conductivity [8]. It is practical for foams, fluids, and melted plastics, but it is impractical for solids [5].

The probe containing the heater and the thermocouple measures instantaneous changes in temperature. Once a predetermined amount of current travels through the heater for a limited period of time, the temperature change of the heater's surface is determined in a characteristic form. After the heat begins to flow from the probe to the specimen side, it reaches the outer surface of the specimen side. When the rise slows down or stops altogether due to heat losses into the environment, the rate of rise with time becomes constant. The thermal conductivity can be calculated based on the linear portion of the temperature versus time curve [8].

The thermal conductivity of the line heat source or probe methods is determined as in Ref. [19].

$$k = \frac{q}{4\pi\left(T\left(t\right) - T_0\right)} ln\left(\frac{4t\alpha}{r^2C}\right) \tag{4}$$

neglecting convection and radiation heat losses. Where q is the heat flow per unit length of the source and ln(C) = 0.5772 is Euler's constant. r is the wire radius and α is the sample thermal diffusivity. Thus, the thermal conductivity can be calculated based on the temperature rise at two different times (or the slope of the temperature rise compared to the logarithm of time) and from the strength of the heat source [16].

The use of a stabilized electrical power supply ensures that the heat source produces a constant output. In order to eliminate the interference of axial conduction via the large-diameter current supply, leads are connected to the ends of the hot wire, and also two hot wires of differing lengths are utilized in a differential mode [7]. The other variations of the hot-wire method are the cross wire and the parallel-wire techniques. In the parallel technique, the heater and temperature sensor are separated from each other. In the cross-wire technique, the heating wire is in contact with the thermocouple [20]. The parallel-wire method is advantageous when applied to anisotropic materials and for materials in the magnitude of a thermal conductivity above 2 W/(m K) [20]. The parallel-wire technique can be used for thermal conductivities below 20 W/m K. The cross-wire technique can be used to measure thermal conductivities below 2 W/m K [2].

Another use of the method is a steady-state pipe method having a cylindrical specimen geometry and containing radial heat-flow measurements [7, 21, 22]. This technique is considered to be a transient radial flow technique, so isotropic specimens are required. Although the pipe method has the disadvantage of deviations from the radial symmetric temperature field with respect to the wire method, adequate mathematical model and evaluation procedure can compensate for this disadvantage [7, 23, 24].

3.2. Hot-disk method

The transient plane source (TPS) technique is a recent development of the hot-strip method. It is also known as the Gustafsson probe or the hot-disk method. The technique is designed to measure both thermal conductivity and thermal diffusivity. The advantage of transient technique to steady-state technique is that the effect of the contact resistance is eliminated in the analysis. This method ensures accurate measurements over a thermal conductivity range from 0.005 to 500 W/(m K) over temperatures from 30 to 1200 K [25]. The TPS technique is used for measuring the thermal conductivity of insulation materials and electrically conducting materials [12]. The main advantages of the hot-disk measurement are that it produces results quickly (usually in under 10 min), and that different sensor sizes can be used to accommodate different specimen types. Furthermore, the hot disk requires using specimen sizes that are usually much smaller than those used in other techniques [12].

The hot-strip method is very similar to the hot-wire technique with the exception of an extended strip. The strip is a metal foil between two specimens or a thin metal film on the surface of a plane specimen. Materials studied so far with this system include metals, alloys, ceramics including high conducting ceramics like Aluminium nitride (AlN), high critical temperature materials, minerals, polymers, composites, glass, fabrics, paper, glass wool, foam, powder, biomaterial, and liquids, as well as materials with anisotropic thermal properties [25].

The hot-disk method utilizes a sensor in the shape of a double spiral of nickel covered material. The TPS sensor consists of a number of concentric circles that are made into a double spiral so that the current will travel from one end to the other. A thin polymer coating material is used as electrical insulation and sensor protection on the spiral. The coating materials are most commonly Kapton for measuring temperature ranges between 30 and 450 K, Mica for higher temperatures of up to 1200 K [25], and Teflon. The sensor acts as both a heat source and a thermometer. The source and the thermometer are used to determine the changes in the temperature of the specimen and the increase in the time-dependent temperature, respectively. The sensor is sandwiched between two pieces of the specimen, as shown in **Figure 6**. During testing, a current travels through the nickel spiral and causes an increase in temperature. The generated heat dissipates throughout the specimen on either side. By comparing the temper-

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ature versus the time response in the sensor, thermal conductivity or diffusivity can be calculated accurately [26, 27].

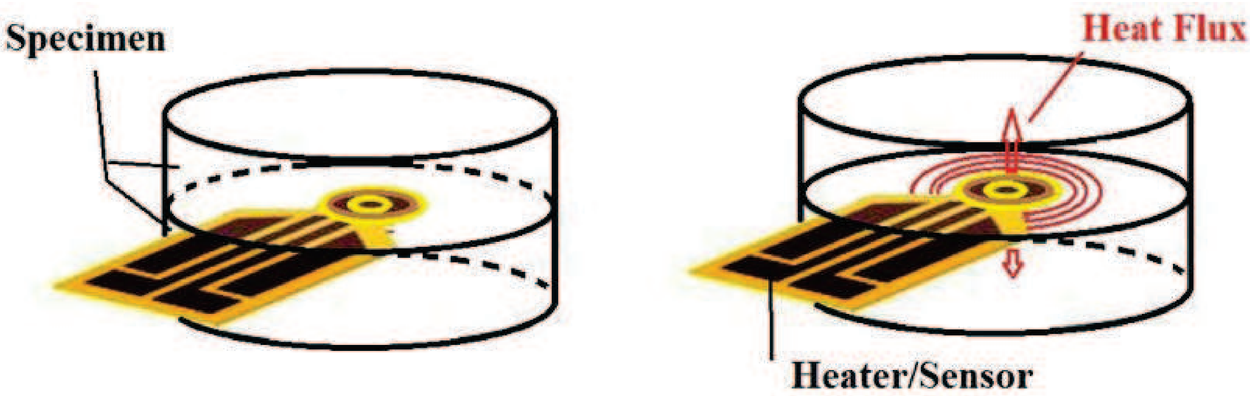

Figure 6. The schematic and principle of the hot disk.

The hot-wire and guarded hot-plate techniques considerably require both larger specimens and a precisely known thickness. While the guarded hot-plate method is a time-consuming method for requiring a temperature gradient across the specimen, the hot-disk method provides instant and direct measurement and the reading is obtained in a short amount of time. In contrast, the guarded hot plate is inherently subject to error due to contact resistance between the thermocouple and the specimen surface. Even at different conductivity levels, a negative influence is unavoidable with the guarded hot-plate method. The hot-disk method, on the other hand, only gathers data regarding from heat diffusion in material for its calculations [26]. As a result, the test procedure of the steady-state method (i.e., the guarded hot plate) is superior to the hot-wire method for testing nonisotropic materials despite the long duration of those steady-state measurements, the higher costs of the steady-state apparatus due to the high reliability in the entire temperature range investigated [28]. It is also in the agreement between the thermal measurements and practical application of insulations [28]. Thus, the hot-wire method should not be employed for determining the thermal conductivity of nonisotropic materials (fiber mats), for which this method is totally ineffective in ranges with a low extinction coefficient, i.e., low bulk densities [28]. On the other hand, one study of the isotropic materials demonstrated that the transient hot-wire method proved to be most dependable due its high reliability and low amount of effort, time, and cost required [28]. For glass fiber, the measured conductivities by hot-disk analyzer are 20 and 12% higher than the claimed values for the lower and higher densities, respectively [12]. For the lower density, the conductivity determined for the rock wool with the hot-disk method harmonizes with the claimed value; the measured value is 8% higher than the claimed conductivity for the higher density [12]. The TPS technique can be used to study liquids, solids, pastes, and powders (electrically conducting or insulating). The TPS technique can provide the values without the interference of thermal contact resistance, without prolonged measurement times, and without extensive specimen preparation [26].

3.3. Laser flash method

The laser flash method is the most commonly used method for ascertaining the thermal properties of solids. The method can investigate to properties of glasses, metals, and ceramics without significant limitations due to uncertainties of the achievable measurement [7]. The property can be measured in a temperature range between −100 and about 3000°C.

In this method, the thermal diffusivity α is determined, and if given the specific heat capacity and the density of a material, the thermal conductivity can be calculated by using the following equation (Eq. (6)) under adiabatic conditions:

$$\alpha = 0.138\frac{d^2}{t_{1/2}} \rightarrow \alpha = {}^{k}\!/_{\varrho c_p} \quad (5)$$

An instantaneous heat pulse is generated by the laser energy. The thermal diffusivity is calculated based on the thickness d of the specimen (typically 2 mm) and the time $t_{1/2}$ [20]. This value represents the time required for the back side surface temperature to reach a value equal to half its maximum value.

In the method, a laser pulse is send to the front side of a specimen, and the temperature change on the back side is measured. The method is conducted through heating a specimen with a short laser pulse of 1 ms width on the front side of the specimen. The temperature increase at its rear side is measured and determined. **Figure 7** shows the schematic and the principle of the method.

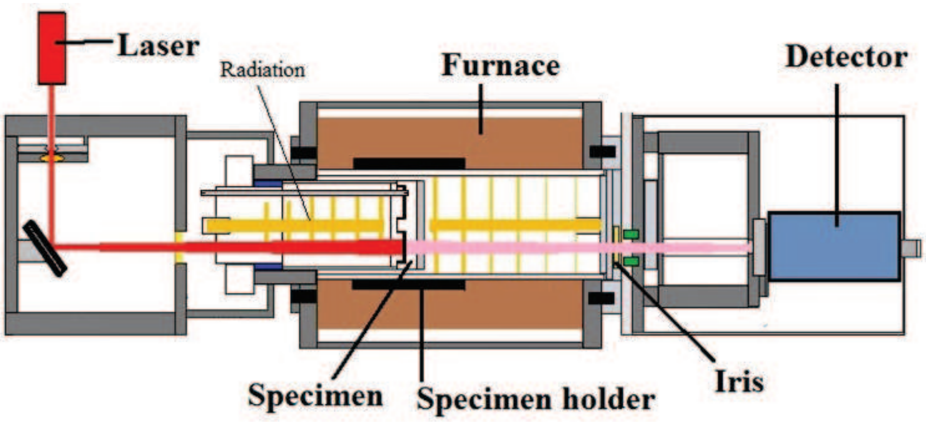

Figure 7. The schematic and principle of the laser flash method [2].

There have been several developments in the method since its introduction by Parker et al. (1961) [29]. The some modifications have been developed to determine directly the thermal conductivity by performing measurement of the specific heat capacity.

The laser flash method has the advantage of involving neither temperature nor heat-flow measurements for the determination of a thermal property. The measurement of the thermal diffusivity is calculated based on the relative temperature change as a function of time only. The main result of this fact is that even at high temperatures the relative measurement uncertainties in the 3–5% range can be achieved [30–32].

3.4. The 3-ω method

The method called the 3-ω method is commonly used for measuring the thermal conductivity of thin films and solid materials. The range of thermal conductivity is changed of 0.20–20 W/(m K), and the range can be extended to 77– 900 K in literature. The method is a similar method to that of the hot wire. While the hot-wire method is a time domain transient technique, the 3-ω method has the advantage of being time independent because it measures electrical signals in a specific frequency domain [33]. An AC current with frequency ω of angular modulation is passed through the wire [34]. The wire is used simultaneously as a heater and a thermometer. The heat generated by this process diffuses into the specimen. Since the electrical resistance of the metal heater is proportional (linear) to the temperature, the temperature oscillation can be measured indirectly by measuring the associated 3ω voltage [34]. Because the current is driven at a frequency ω and the resistance changes at a frequency 2ω, a voltage at 3ω results [34]. In this method, a thin electrically conductive wire is patterned on the specimen, as shown in **Figure 8** [35].

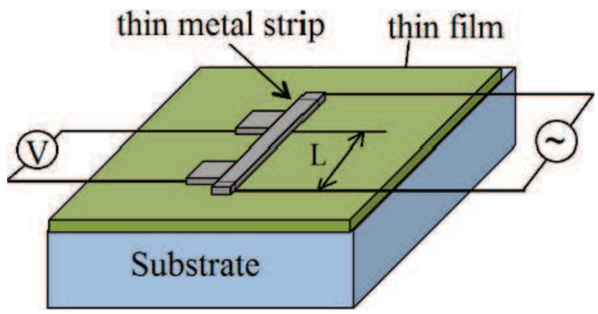

Figure 8. The schematic of the 3-ω method for thin film [35].

Both basic mechanisms generally affect in-plane (*x*) and cross-plane (*z*) transport differently, so that the thermal conductivity of related materials is usually anisotropic. The method measures the average thermal conductivity in the in-plane and cross-plane directions. The combination between the heater wire width and the thin film thickness determines the measurement sensitivity according to the in-plane and cross-plane thermal properties of the film [16].

3.5. Fitch method

The Fitch method developed by Fitch is used to measure the materials of low thermal conductivity by using a plane source of heat. This method consists of two components: a heat source and a heat receiver. The heat source is a vessel filled with a constant temperature liquid that functions as a sink. The heat receiver is a sink in the form of a copper plug insulated all sides but the one facing the vessel [36]. The roles of the heat source and the heat receiver can be changed if the vessel is at a temperature lower than that of the copper block. The specimen is interposed between the vessel and the open face of the plug. The sample is firstly in thermal equilibrium with the copper block as shown in **Figure 9**. The vessel is brought into contact with the specimen under a temperature differential. The temperature history of the copper block and the temperature of the bottom of the vessel are measured by thermocouples. It is assumed to have a uniform temperature distribution. A change of time and the temperature are measured, and the thermal conductivity of specimen is calculated using the following equation [37]:

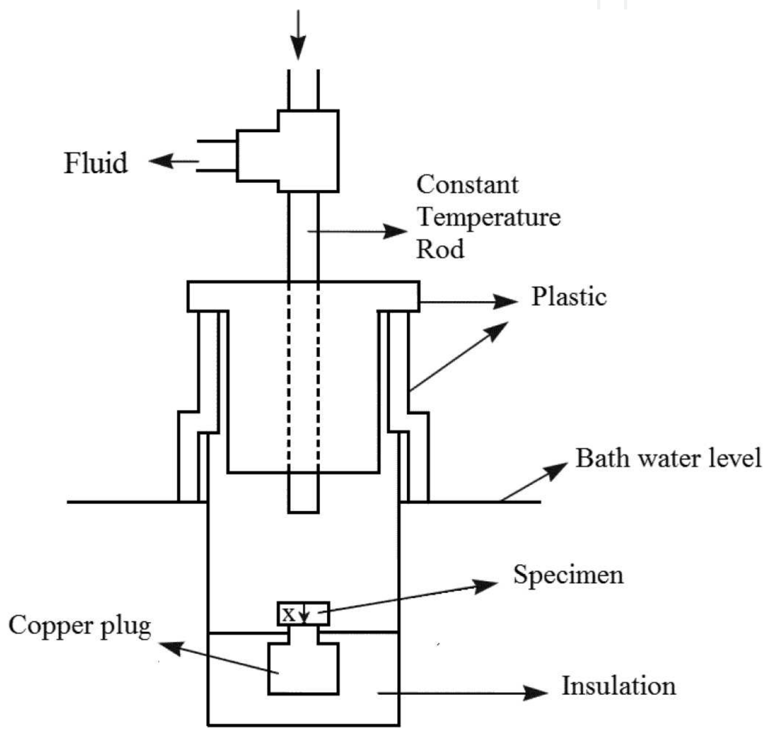

Figure 9. The schematic of the Fitch method [37].

$$k = \frac{\Delta x . m_c . c_{pc}}{A.t} ln \frac{(T_0 - T_\infty)}{(T - T_\infty)} \quad (6)$$

where c_{pc}, Δx, and A are the heat capacity of the copper block, and the thickness and heat transfer area of the specimen. The heat transfer from the copper block to insulation, the heat storage in the specimen, and the surface contact resistance are assumed to be negligible under a linear temperature profile.

This method is generally used for measuring the conductivity of foods and specially small-sized specimens. But the Fitch method is not practical for use at high temperatures. In this method, either a line heat source or one or more heat sources are used [16].

3.6. Photothermal methods

The principle of this method is based on ascertaining the light-induced change in the thermal state of a material in solid, liquid, or gaseous state. After the light is absorbed by a specimen, the changes in temperature, pressure, or density are determined and measured. There are methods in which the specimen is placed in contact with and without the detection system [7].

Measuring the thermal diffusivity measurement via the photothermal method is based on the concept of modulating between the heating of a specimen surface and the temperature phase on the opposite sides of the specimen and includes a frequency function. This technique can be modified by having both surfaces heated simultaneously with a single modulation frequency and taking a measurement of the phase difference between surface signals [20]. The schematic principle of a photoacoustic method is also provided in Reference [38].

4. Standards for thermal insulation measurement

Every measurement of insulation materials has a definition, a standard, and a test realization. The tests are determined by means of experiments for the definition as closely as possible. The experiments are usually performed by standards. When the test realization is obtained, the measurement devices provide its property value. The national and international laboratories use various standards associated with insulation materials. The standards' representation is a main standard to which other representations are compared. The international standards to general methods are listed as follows:

- ASTM C177/C177-13, standard test method for steady-state heat flux measurements and thermal transmission properties by means of the guarded-hot-plate apparatus.
- ASTM C518/C518-10/C518-15, standard test method for steady-state thermal transmission properties by means of the heat-flow meter apparatus.
- ASTM C335/335-10e1, standard test method for steady-state heat transfer properties of pipe ınsulation.
- ASTM C653-97(2012), standard guide for determination of the thermal resistance of low-density blanket-type mineral fiber insulation.
- ASTM C680-14, standard practice for estimate of the heat gain or loss and the surface temperatures of insulated flat, cylindrical, and spherical systems by use of computer programs.
- ASTM C687-12, standard practice for determination of thermal resistance of loose-fill building ınsulation.
- ASTM C1303/C1303M-15, standard test method for predicting long-term thermal resistance of closed-cell foam ınsulation.
- ASTM C1114-06(2013), standard test method for steady-state thermal transmission properties by means of the thin-heater apparatus.

- ASTM C1363/C1363-05/ C1363-11, standard test method for thermal performance of building materials and envelope assemblies by means of a hot box apparatus.
- ASTM C1667-15, standard test method for using heat-flow meter apparatus to measure the center-of-panel thermal transmission properties of vacuum insulation panels.
- ASTM C1696-14ae1, standard guide for industrial thermal insulation systems.
- ASTM C1774-13, standard guide for thermal performance testing of cryogenic insulation systems.
- ASTM D5470-06, standard test method for thermal transmission properties of thermally conductive electrical insulation materials.
- ASTM E1225-09/E1225-13, standard test method for thermal conductivity of solids by means of the guarded-comparative-longitudinal heat-flow technique.
- ASTM E1530-06/E1530-11, standard test method for evaluating the resistance to thermal transmission of materials by the guarded heat-flow meter technique.
- ASTM F433-02 (2009, 2014), standard practice for evaluating thermal conductivity of gasket materials
- ASTM D5334-08, standard test method for determination of thermal conductivity of soil and soft rock by thermal needle probe procedure.
- DIN EN 12667/12939, European standard for measurements of insulating materials using the heat-flow meter method or the guarded hot-plate technique.
- DIN EN 13163, European Standard for characterization of foam insulations for building applications using the heat-flow meter method or the guarded hot-plate technique.
- ISO 8301/8302, thermal insulation—determination of steady-state thermal resistance and related properties—heat-flow meter/guarded hot-plate apparatus.
- ISO 8894-1 (EN 993-14), refractory materials—determination of thermal conductivity—Part 1: hot-wire methods (cross-array and resistance thermometer).
- ISO 8894-2 (EN 993-15), refractory materials—determination of thermal conductivity—Part 2: hot-wire method (parallel)
- ASTM C1113/C1113M-09 (2013), standard test method for thermal conductivity of refractories by hot wire (platinum resistance thermometer technique).

The problem of determining the thermal conductivity of insulation material as a porous medium has been investigated by researchers. The literature contains a large number of standard models for predicting the thermal conductivities of homogeneous, heterogeneous, or composite materials materials [39], as well as methods of a measurement apparatus designed [40]. These models are largely dependent on mathematical models derived from the physical laws that govern the process (e.g., mass and energy balances, thermodynamics, etc.). The literature also present an experimental evaluation of the models and a comparison analysis of the existing experimental methods. Several researchers have proposed a number of models derived from a set of equations, usually based on a basic model, but many of these include empirical parameters [1]. The difference of thermal conductivity values between models and methods can be considered due to the lack of structural data. The structures include nonhomogeneity/homogeneity, the components, the differences in the direction of components, the condensation/evaporation of a liquid phase, the contact mechanism, the size and shape of particles/pores, the tortuosity of pores, mixed phases, shape factor, contact, orientation,thickness, reinforcement etc. [39, 41, 42].

Many thermal conductivity models found in the literature are based on one or more basic structural models, such as the series and parallel models, the geometric mean model, Maxwell models (different forms) and effective medium theory (EMT) models, and the Russell Model [39]. The choice of thermal conductivity models is generally discussed separately in the literature for each of these classes [39]. A detailed description for a bulk material with a porous medium can be expressed in terms of “ancestor,” “extra parameter,” “component knowledge,” and “heat transfer mechanisms” in various models [39]. Similar models are given in different forms and replace the other components instead of a component parameter. It has not been possible to know which expression is the most accurate for describing the thermal conductivity even in every model of two-phase components/mixes. Therefore, there is no single model or prediction procedure to be considered that is universally applicable. In most modeling exercises, the prediction of thermal conductivity also involves a trade-off between simplicity and accuracy. The accuracy of predicted thermal conductivities cannot be reasonably expected to be better than 5% [39–41]. Several models that have been proposed are created specifically for a particular material and contain material-content properties [39].The models need to be revised by clear parameters, particularly with respect to parameters in models (such as thermal conductivities, porosity, or volume fractions ratio of phases). It is concluded that, instead of new relations, the use of present mathematical relations with finite errors for specific materials and at specific intervals is important.

5. Conclusion

The present chapter presents a comprehensive overview of the research progress on the measurement methods of the thermal conductivity of materials. This study discusses the effectiveness of various methods for measuring the conductivity of insulation materials. The models show similarities and differences depending on whether it is a steady state. Some methods are similar or derivative methods regardless of whether the thermal conductivity model being used is similar. The first major conclusion that can be drawn is that these methods may not be applicable for all materials.

Thermal conductivity methods are broadly classified as either steady-state methods or transient methods. In principle, these methods are based on establishing a steady temperature and a signal gradient over a sample. The techniques measure a response as a signal or a temperature given in the sample. Therefore, these techniques are differentiated mainly concerning a range of thermal conductivities, a range of material types, a measurement time, a measurement accuracy, a specimen type, and a temperature range. Estimating low thermal conductivity may be determined with adjustable parameters in these methods.

There are various methods for determining thermal conductivity of insulation materials. The preferable methods are commonly the guarded hot-plate method, the heat-flow method, and then can be given by the hot wire/disk, laser flash methods. Under the condition of anisotropic and isotropic type, some methods are in good agreement with the claimed values. The thermal conductivity value from the method is higher or lower than the value from other methods, depending on the relationships between the heat loss distribution, the surface temperatures, the uniform heat flow over the material, and the signal and its response. However, the methods must be carefully selected according to the materials, such as nonisotropic materials, and the measurement times before applying the methods. It is concluded that choosing methods to use with finite errors for specific structures and at specific intervals is important. This point can provide an effective way and the accuracy and suitability of a variety of methods for users, manufacturers, and researchers in this field.

Many theoretical models, as well as methods, have been proposed by various researchers. The models are based on one or more basic structural models, such as the series and parallel models, the geometric mean model, Maxwell models (different forms), effective medium theory (EMT) models, and the Russell model. The models may not seem to be a clear explanation of the interior structure and behavior of porous materials. The second major conclusion that can be drawn from the studies is that in the future researchers need to standardize methods for insulators or conductors and for thermal conductivity values.

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