Problem of elimination of undesirable thermal flows at thermophysical measurements

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Abstract
The undesirable thermal flows at thermophysical measurements arise as a consequence of the non-existence of perfect thermal insulators. These flows can be minimalized through suitable thermal shielding. The use of the thermal shielding enables to increase the precision of the thermophysical measurements. In the paper some special cases of the thermal shielding are introduced and analyzed.

1. Introduction

At measurement of the thermophysical parameters of samples a task arises to eliminate or, at least to minimalise undesirable breakaway of heat into surrounding of a sample, it means to eliminate undesirable thermal flows. A perfect thermal insulator does not exist. A possible way out to achieve this goal can be a thermal shielding. Such shielding complicates situation for an experimentalist but, it enables to obtain more precise results. Further on, we describe a few special cases of the thermal shielding in measurements with selected temperature regime.
It is known a large number of measuring methods and set-ups. Many of them are described in papers [1 – 20]. J.Krempaský [4] estimated that there exist approximately 500 measuring methods.
The method sused for measuring thermophysical parameters of materials can be divided into steady state and dynamic ones. While the former use a steady state temperature field in side the sample, the latter use a dynamic temperature field. The dynamic methods can be characterized as follows. The temperature of the sample is stabilized and uniform. Then the dynamic heat flow in the form of a pulse or stepwise function is applied to the sample. The thermophysical parameters of the material can be calculated from the temperature response.
Dynamic methods represent a large group of techniques with various geometrical arrangements [21-38]. The following method belong to this group: Transient hot wire [33], Transient hot strip [34], Pulse transient, Step-wise transient, Hot plate transient, Hot disc transient, Gustafsson probe [35], Dynamic plane source [36], Extended dynamic plane source (EDPS) [37] and Laser flash method [38].
The measuring procedure consists of theory and experiment. The theoretical model of the experiment is described by the partial differential equation for the heat transport. The temperature function is a solution of this equation with boundary and initial conditions corresponding to the experimental arrangement. The experiment consists in measuring the temperature response and fitting the temperature function over the experimental points. Using the least squares procedure following thermophysical parameters can be estimated: thermal diffusivity a, thermal conductivity λ and specific heat capacity c.
Obtaining theoretical requirements (conditions) for some measuring method – it is one step on the route to the realization of a measurement.
A creation of proper conditions for its realization is the next important aspect of a measurement. Among them is also an elimination or restriction of heat losses in the measurement. Heat losses could devalue results of a measurement. Heat losses may be caused by a conduction, convection as well as radiation. Radiation is significant mainly in measurements at high temperatures.
A shielding method was used for an elimination of heat losses at first in calorimetry [39, 40]. Adiabatically shielded calorimeters were mainly used in measurements of specific heat capacities of various substances, namely metals [40, 41, 42].

Simple methods of shielding were first used in measurements of thermal conductivity, mainly in stationary and quasi-stationary methods of the measurement [17,18]. Heat losses manifest themselves mostly in long-time measurements (in stationary as well as quasi-stationary measurements). A distinctive case of non-stationary methods is the method of a constant increase in temperature [8,12,43]. The method of a constant increasing of temperature is a especially case of non stationare methods. Heat losses are less important in short-time measurements.
The reliability of every measurement confirms a quantitative statement of its uncertainty [44-46]. The sources of uncertainty in thermophysical methods can be divided in to two parts. The first part is caused by the deviations from the theoretical model. The second part is created by uncertainties of input parameters measurements.

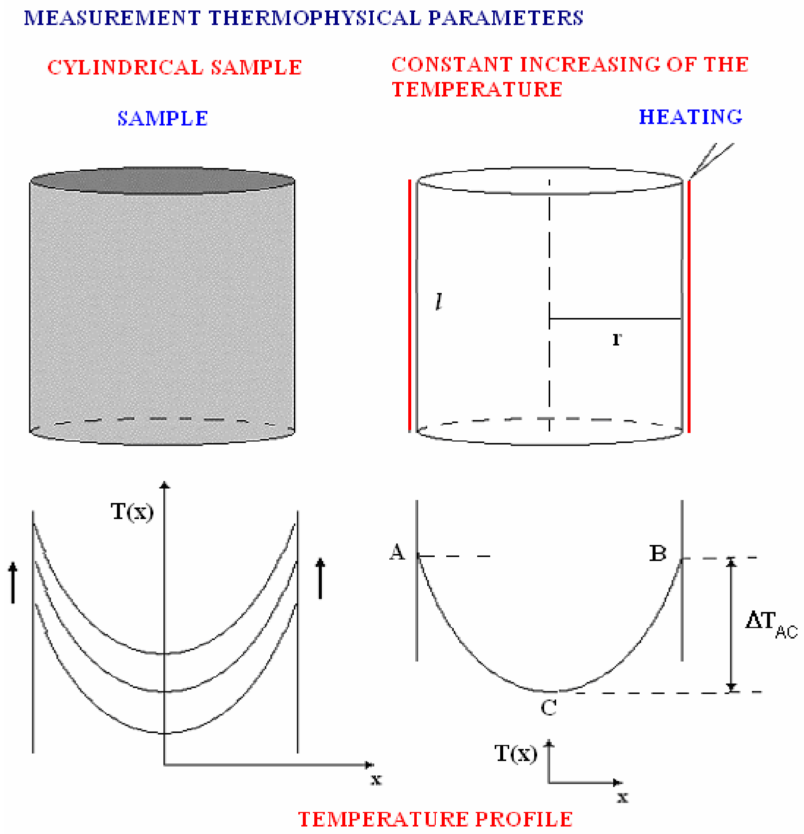

Fig. 1

2. Thermal shielding at steady increasing temperature methods

At steady increasing temperature method the rate of temperature increase is the same everywhere within a sample so that derivation of temperature relative to the time τ is constant [1-8]. Then it holds

$$\frac{dT}{d\tau} = const$$

Such temperature regime usually erases in a sample if a thermal insulated sample is heated with a thermal source of constant power (after some onset time).

2.1 Cylindrical sample heated at its border surface

The scheme of experimental setup is depicted in Fig. 1. A face of sample is cylindrical. Its surface is heated by a stove of constant heat power. At a constant rate of increasing temperature at the cylindrical sample surface arises a parabolic temperature profile $T(x)$. Minimum of temperature is at the axis of the cylinder. If we know the difference ΔT_{AC} of the temperature T_A at the surface and that one T_C at the cylinder axis, and knowing the temperature rate $\dot{T}$ and power P then we can determine the diffusivity a_C of the sample as well as specific heat c_C. Naturally, we need to find out to this aim dimensions of the sample. Moreover, if the density of that sample ρ_C is known these data permit us to determine the thermal conductivity λ_C of sample.

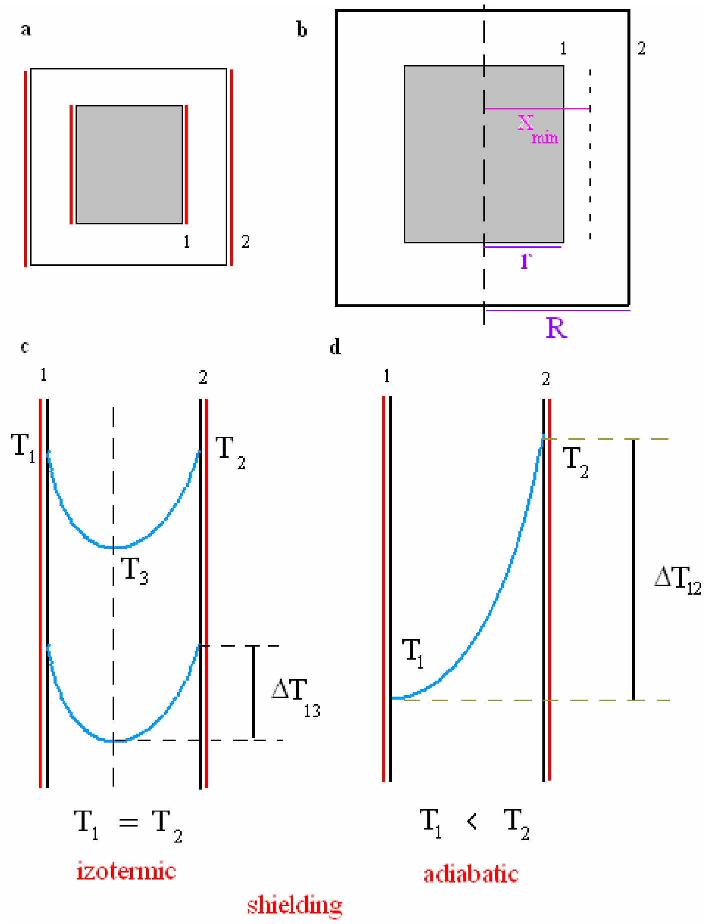

Fig. 2

Then formulae are valid for upper mentioned quantities

$$a_c = \frac{r^2}{2\Delta T_{AC}} \frac{dT}{d\tau}, \qquad \lambda_c = a_c c_c \rho_c, \qquad c_c = \frac{P}{m_c \; dT/d\tau}$$

where m_C is mass of the cylindrical sample.

Up to now a thermal loss was ignored at the upper described measurement method. Practically, the thermal loss has to be protected; it means to eliminate undesirable thermal flows.

2.1.1 Radial thermal flow shielding

Loss of heat of a cylindrical sample passing through lower and upper circular areas will be not yet considered. The thermal loss across boundary cylindrical surface of a sample in a given setup is minimalized throughout thermal shielding. It consists of a thermal insulating layer

between surface 1 and 2 and heated metallic screen in form of a cylindrical tube surrounding the thermal insulating layer.
The shielding can be isothermic or adiabatic.

Isothermic shielding

In a case of an isothermic shielding the temperature T_2 is identical with the temperature T_1 of the external cylindrical surface of the sample every time. In this way radial thermal flow is protected across insulating layer and at the first sight it seams that the thermal loss is excluded completely. Actually, the things are a little different. We want to pointed out that the heating stove placed at the boundary sample surface heated also a part of insulation, because at the regime of increasing temperature the temperature of isolation also increases. From this viewpoint it is important to determine what part of insulation is heated by the stove of the sample and what part is heated by the stove of the screen. This knowledge permits more precisely to determine the heat supplied by the stove to the sample alone.

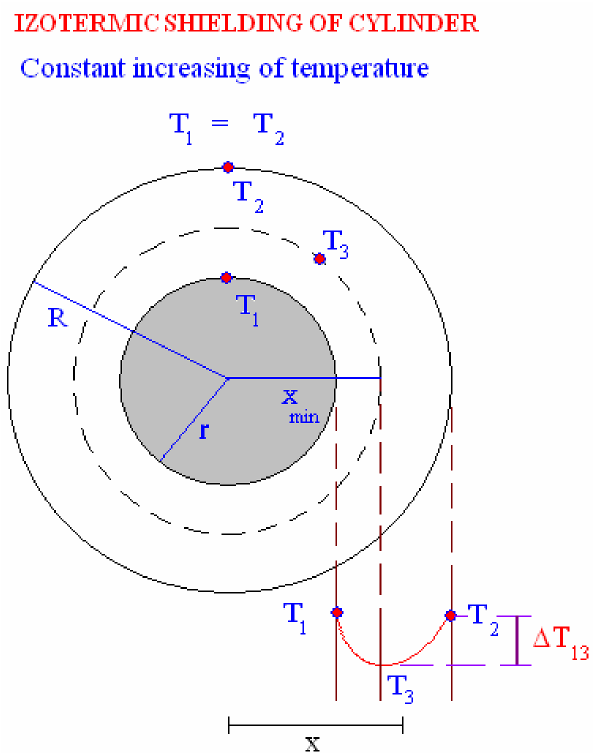

Fig. 3

At the constant temperature rate of the system by isothermic shielding the temperature T_1 of the external sample surface is equal to the temperature of the screen T_2 anytime. These temperatures uniformly increase. At the upper described regime a more complex temperature profile is created across the insulating layer because in deep of insulator the temperature is retarded in comparison with temperatures $T_1 = T_2$ at the surfaces. In this way created temperature profile is shifted uniformly to the higher temperature. It looks like as a process in measured sample alone. The shape of the temperature function in insulator is more complex (and not exactly parabolic) that one in the sample. See Fig. 2c and Fig. 3.
Analysis of this problems leads to the conclusion that the temperature minimum in isolating layer surrounding cylindrical sample lies at the distance x_{min} from the cylinder axis, where

$$x_{\min} = \sqrt{\frac{R^2 - r^2}{2\ln\frac{R}{r}}} \qquad (1)$$

This result one can obtain as follows: one determines thermal flow passing through cylindrical surface with radius x in interval $r < x < x_{\min}$. This heat is used to the heating of an insulating cylindrical layer with radius from x up to $x_{\min}$. By analogy, one determines thermal flow passing through cylindrical surface with radius x from interval $x_{\min} < x < R$, considering that corresponding heat crossing this surface is used for heating of the insulating cylindrical layer with radius lying in interval of $(x_{\min}, x)$. Integration of two formulae obtaining this way leads to two expressions for temperature difference between points 1 and 2; 2 and 3 respectively. At an isothermic shielding values of those differences have to be equal (identical) because the temperature at the points 1 and 2 are equal ($T_1 = T_2$)

Analyzing this problem one can achieves following analytical expression for temperature profile $T(x,\tau)$ in insulating layer ($x \in (r, R)$)

$$T(x,\tau) = \frac{1}{4a}\frac{dT}{d\tau}\left(x^2 - x_{\min}{}^2 - 2\,x_{\min}{}^2 \ln\frac{x}{x_{\min}}\right) + T(x_{\min}, \tau) \qquad (2)$$

where for x_m relation (1) is still valid. Term $T(x_{\min}, \tau)$ represents the minimal temperature of isolation at the time τ. It holds $T(x,\tau) = T(x) + k\tau$. The term $T(x)$ expresses an intrinsic temperature profile of isolation which is uniformly shifted to the higher temperatures due to the condition

$$\frac{dT}{d\tau} = const$$

As temperature minimum is at the cylindrical surface with radius x_m temperature gradient is zero at this surface. Through this cylindrical surface doesn't pass anymore thermal flow at a measurement. It implies that the cylindrical part of insulation of radius between r and $x_{\min}$ is heated by the stove located on the specimen. The other outer part of the insulation (its radius is in interval $(x_{\min}, R)$ is heated by outer shielding stove.

A thermal power P_C submitted to the specimen is smaller than the stove power P on the specimen. We can write

$$P_C = P - c\, m_{13}\frac{dT}{d\tau}$$

where c is a specific heat of isolation and m_{13} - mass of the corresponding inner part of isolation. Mass m_{iv} can be determined from known density ρ_{i} of isolation and volume of the insulator's cylindrical layer (inner radius *r,* outer - $x_{\min}$).

With respect to (2) we can write for the temperature differences

$$\Delta T_{13} = \Delta T_{23} = \frac{1}{4a}\frac{dT}{d\tau}\left(r^2 - x_{\min}{}^2 - 2\,x_{\min}{}^2 \ln\frac{r}{x_{\min}}\right)$$

where $\Delta T_{13} = T_1 - T_3 = T_2 - T_3 = \Delta T_{23}$, considering the temperature T_1 at the surface of the sample is equal as the temperature at the screen T_2. T_3 is the temperature at $x_{\min}$ in isolation where the temperature possesses its minimum.

This difference at the constant temperature increase becomes unchanged (while diffusivity stays unchanged).
This account enables correction of the stove power connected with heating of inner insulating part. In this way the measurement of sample thermal parameters is more precise.
For the temperature difference $\Delta T_{13} = \Delta T_{23}$ it holds also

$$\Delta T_{13} = \frac{1}{4a}\frac{dT}{d\tau}\left(R^2 - x_{\min}{}^2 - 2\, x_{\min}{}^2 \ln\frac{R}{x_{\min}}\right)$$

Adiabatic shielding
Shielding is called adiabatic if thermal flow throughout entire outer surface of a sample (including a stove at its surface) is zero. Such a state can be achieved when within the isolation a temperature profile will be created with a minimum of the corresponding temperature function $T(x)$ resting just at the cylindrical surface of the sample. This state shows Fig. 2d.
Thermal flow analysis of a similar type as in the case of an isotermic shielding showed that this case occurs when the temperature of the screen T_2 is of some amount ΔT_{21} higher than the temperature T_1 at the sample surface

$$\Delta T_{21} = \frac{1}{4a}\frac{dT}{d\tau}\left(R^2 - r^2 - 2r^2 \ln\frac{R}{r}\right)$$

A way out is to express thermal flow across a cylindrical surface with the radius x inside interval $r < x < R$ and a fact that corresponding heat isolation of the thickness $r - x$
Inside isolation one can express the temperature profile in isolation as

$$T(x,\tau) = \frac{1}{4a}\frac{dT}{d\tau}\left(x^2 - r^2 - 2r^2 \ln\frac{x}{r}\right) + T(r,\tau)$$

In the case of an adiabatic shielding a heating stove at a sample transmits to the sample whole thermal power. There is not need for correction.

2.1.2 Shielding of cylindrical bases

In method of steady increase with heating at the surface of a cylinder the temperature profile $T(x)$ inside a homogeneous sample is parabolic as mentioned earlier. While, the thermal loss is neglected across both basic areas of a cylinder there will be a similar parabolic distribution of the temperature at the basic circular areas. A shielding of the basic areas will be harder than the shielding of the enveloping cylindrical surface. We are going to describe two suitable methods of the thermal shielding.

Method No 1
This method employs two or more sliced screen (disc 1) constructed in such way that its effective diffusivity a_{eff} is nearly the same as the diffusivity of the sample. Practically, after first measurement of the sample parameters with application of a shielding the screen will be adapted to the new measured value of the quantity *a*.
For two-layer screen can be proved following formula

$$a_{ef} = \frac{\lambda_1 h_1 + \lambda_2 h_2}{c_1 \rho_1 h_1 + c_2 \rho_2 h_{21}}$$

where h_1 and h_2 denote the thickness of screening layers. The meaning of all other symbols is apparent. This relation one can easily spread onto multilayer screens.
Multilayer screen is less adaptable and as fare as we don’t know parameters of each layer it can be prepared only by experience. If it is correctly done, at the analogical increase of the temperature on the sample periphery the temperature profile inside will satisfy the temperature profile on the cylindrical sample base.
Of course, the temperature regime inside the screen disturbs particularly venting of heat into surrounding. This state one can improve by a double shielding. It means by using of two analogical screens with an isolation layer in between. In this way in the screen which is attached to the sample base a thermal profile approaches better to the perfect one.
This fact can be reasoned via theoretical model and corresponding calculation methods of numerical mathematics.

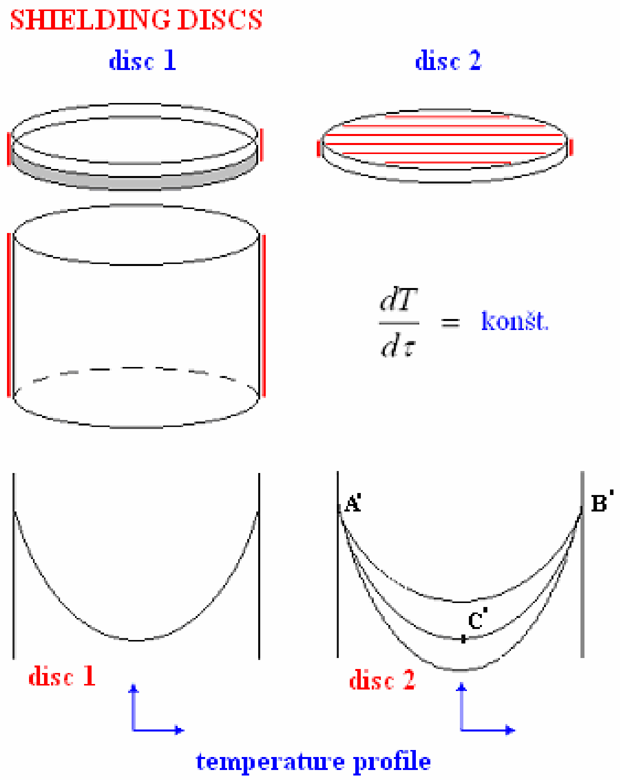

Fig. 4

Method No 2

A better possibility for shielding basic surfaces of a cylinder offers a screen (disc 2) with planar heat source at its circular surfaces. Diffusivity for such a screen has to be less than is the diffusivity of a sample. Without applying of heating the temperature profile of a screen at an increase of temperature would not correspond to the profile in sample. Instantaneous temperature at the middle of the screen by increasing of temperature would be lower than the temperature at the middle of cylindrical sample. A planar heating of the screen permits to fit a temperature profile of screen so that it would correspond to the sample profile exactly adapting power of a stove (changing the current). Assuming, the screen is regularly heated over all circular area.
At intrinsic measurement with the use of shielding one inserts between the sample base and the screen a suitable insulating layer. Under optimal conditions average gradient of temperature in orthogonal direction to the isolation layer will be zero. In this way the thermal losses are eliminated to a great extent.

It is possible to prove mathematically that trough power adjusting of the stove on the circular screen makes it possible to adjust temperature profile of the screen to that one of the cylindrical stove. Such screen possesses varieties of shielding properties. This is great advantage in comparison with two-layer screen.
It can be imagine quasi-adiabatic shielding of the cylinder bases. In that case the thermal profile of the screen would be shifted to higher temperature compare to the sample profile Required temperature difference between temperature profile of the screen and that one of the sample will be determined by (3) – will be introduced latter – despite the other situation wit comparison to the case of a platelike sample.
We notice that difference between screen temperature at its circuit and the temperature at its center is given as

$$\Delta T_{A'C'} = \frac{1}{4a_t}\left[\frac{dT}{d\tau} - \frac{2P_t}{C_t}\right]R^2$$

where P is a power of electric stove, λ_t, a_t are thermal conductivity and diffusivity of the shielding layer respectively. C_t is heat capacity of the screen. At equality of analogical temperature differences on the sample and the screen the temperature profiles will be also identical.
The parabolic temperature profile creating in the screen by the superficial heater is represented by parabola

$$T(x,\tau) = \frac{1}{4a_t}\left(\frac{dT}{d\tau} - \frac{2P_t}{C_t}\right)x^2 + T(0,\tau)$$

The power P_t of electric screening stove is managed in such manner in order that it corresponds to the sample profile. To this aim it suffices to regulate (by a change of current on superficial stove of the screen) temperature at the screen center with that one in the middle of measured sample.

3. Shielding in a case of a spherical specimen

We denote radius of a spherical sample as *r*, and outer radius of an isolation layer as *R*.
At isothermic shielding with a regime of steady temperature increase (surfaces of the sample and that one of the screen having identical temperature) minimum of the temperature inside an isolation layer is at the spherical surface which radius is $x_{\min}$ where

$$x_{\min} = \sqrt[3]{\frac{Rr\,(R+r)}{2}}$$

Knowledge of this value enables to correct power of a stove heating sample. Thereby, determination of sample specific heat becomes more precise. By derivation this formula we precede the same way as it was at the case of a cylindrical insulating layer.
Thermal profile in isolation by an isotermic shielding gives the relation

$$T(x,\tau) = \frac{1}{6a}\frac{dT}{d\tau}\left[R^2 - x^2 + 2x_{\min}^3\left(\frac{1}{R} - \frac{1}{x}\right)\right] + T(R,\tau)$$

The value of maximal temperature difference in isolation - between the temperature at the border of spherical isolating layer and the minimal temperature inside isolation is

$$\Delta T_{13} = \frac{1}{6a}\frac{dT}{d\tau}\left[R^2 - x_{\min}^2 + 2\,x_{\min}^3\left(\frac{1}{R} - \frac{1}{x_{\min}}\right)\right]$$

Temperature profile in spherical insulator layer at an adiabatic shielding is

$$T(x) = \frac{1}{6a}\frac{dT}{d\tau}\left[x^2 - r^2 + 2r^3\left(\frac{1}{x} - \frac{1}{r}\right)\right] + T_1$$

By an adiabatic shielding difference of temperature at the inner and outer surface of the isolation has to obey following formula

$$\Delta T_{21} = \frac{1}{6a}\frac{dT}{d\tau}\left[R^2 - r^2 + 2r^3\left(\frac{1}{R} - \frac{1}{r}\right)\right]$$

In a case of an adiabatic shielding the whole stove power is heating sample and so no correction of its power is needed.
Diffusivity a_s of a spherical sample (sphere) by a method of a steady increase of temperature assuming that sphere is heated by a thermal sour at its outer spherical surface - is determined as

$$a_S = \frac{r^2}{6\Delta T_S}\frac{dT}{d\tau}$$

where ΔT_S denotes the temperature difference between temperatures at outer surface of the sphere and that one at center.
For the other thermal parameters it holds

$$c_S = \frac{P}{m_S \frac{dT}{d\tau}}$$

$$\lambda_S = a_S c_S \rho_S$$

where their meaning is clear. By adiabatic shielding the power P needs no correction; by isothermic one its correction is necessary since inner part of isolation is also heated.

4. Shielding in a case of platelike samples

By shielding a (planeparalel) plate (Fig. 5) the isolation layer ISOL is platelike**.** Let us h denotes its thickness. A temperature profile in isolation by isothermic shielding ($T_1 = T_2$) is parabolic and symmetric. At a constant rate of temperature increase the minimum of temperature is at the plane dividing the insulator layer into two equal parts. As we choose x-axis according to the Fig. 2 it is the (vertical) plane at $x_{\min} = h/2$.
This way, one half of insulator plate is heated by the sample, which permits to correct power of a stove at the sample.
Maximal value of temperature difference in isolation is equal to

$$\Delta T_{13} = \frac{h^2}{8a}\frac{dT}{d\tau}$$

The temperature profile in insulator layer by an isothermic isolation of the platelike sample is expressed by the formula

$$T(x) = \frac{h^2}{2a}\frac{dT}{d\tau}\left(x - \frac{h}{2}\right)^2 + T_3$$

or

$$T(x) \;=\; \frac{h^2}{2a}\frac{dT}{d\tau}\left(x^2 - hx\right)^2 + T_1$$

An adiabatic shielding of the platelike sample at regime of steady temperature increase requires keeping temperature of the screen higher of amount

$$\Delta T_{21} = \frac{h^2}{2a}\frac{dT}{d\tau} \qquad (3)$$

than is the surface temperature of the sample.

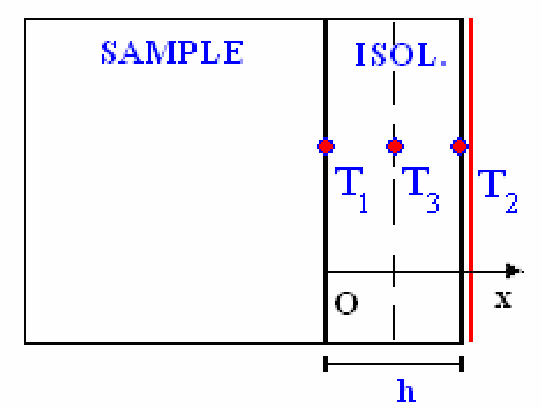

Fig. 5

The temperature profile inside insulator layer using an adiabatic isolation determines the function

$$T(x) \;=\; \frac{h^2}{2a}\frac{dT}{d\tau}x^2 + T_1$$

5. Shielding at measurement of caliducts

Thermal looses of caliducts are usually measured in a stationary regime with qualificative boundary conditions [47]. The temperature is given in the nucleus of a caliduct (temperature of the central metallic tube) and the temperature at the external caliduct surface. During a measurement stationary temperature profile is created within caliduct isolation of a radial type. That one would be disturbed (without shading) near the marginal regions of a caliduct (Fig. 6a). In order to exclude that distortion shielding of border surfaces is necessary.

A few possibilities for shielding of caliduct ends shove Fig. 6. In the case shoved in Fig. 6b a marginal part of the caliduct is employed with a separated part of central tube, which is heated to the desired temperature by a separated stove under control. Temperature field disturbances owing to heat losses throughout end areas in inner part of caliduct - where measurement is executed – will not appear.

To the shielding of the ends can be used also the other type of material (Fig. 6c), than is insulator material of caliduct. It cannot be either a thermal isolating material. It is due to fact, that a steady temperature profile at equal boundary condition will be the same in different materials. Even thought, if a material possesses better thermal conductivity the shielding will be also better and a screen may be narrower. Preferable possibility offers a multilayer screen in which metallic and insulating layers alternate (Fig.6d). Temperature profile in conduction layers of a multilayer screen improves gradually in direction to the caliduct and shielding is superior. Disturbances caused by surrounding affect significantly the external layers of the screen. Central part of the screen is heated to the desired temperature by a separated stove under control.

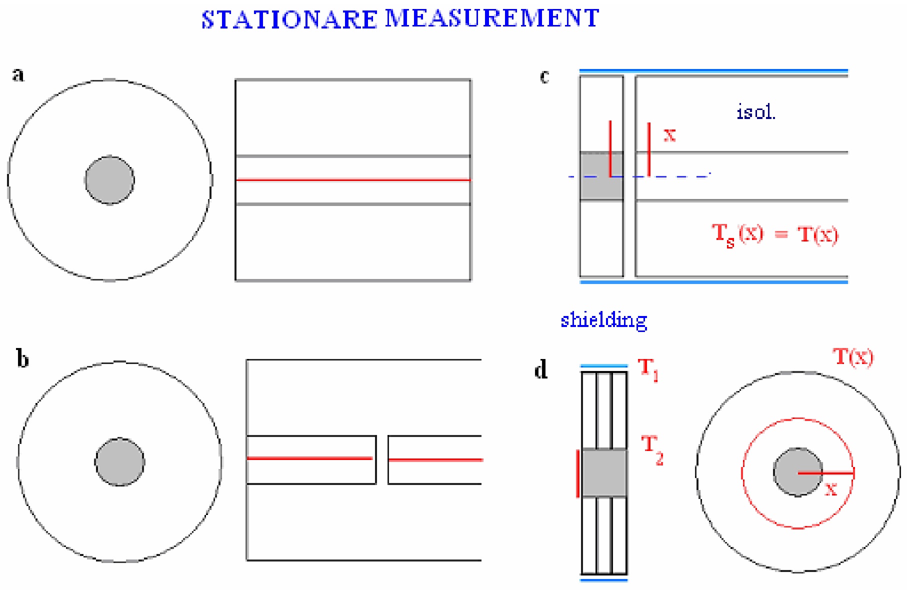

Fig. 6

The temperature field in separate screen layers one can determine using numerical methods under some reasonable simplifications.

6. Conclusion

This article suggests many methods of elimination undesirable thermal flows at thermophysical measurements using temperature shielding. It is going on some kinds of experimental setup applying a steady increase temperature method. It deals with shielding in a case of stationary regime at the laboratory measurement of caliduct losses. Temperature shielding enables to increase precision of the thermophysical measurements.

7. Acknowledgement

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