A NEW METHOD FOR MEASURING THE HEAT CONDUCTION COEFFICIENT OF THIN CERAMIC LAYERS AT HIGH TEMPERATURES

Wiktor Żyszkowski

Institute of Nuclear Research, Pl-05-400 Świerk, Poland

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Abstract—A new method is described for thermal conductivity measurements at high temperatures on poor thermal conductors such as thin ceramic layers. The essence of the method consists of using a calorimeter enclosed by a layer of the material under investigation. The calorimeter contains a pre-determined mass of the substance which melts when the calorimeter is suddenly immersed in a hot bath. The theoretical interpretation and the calculations of the heat conduction coefficient of the ceramic layer are based on the time duration of the change of phase which takes place in the calorimeter. Preliminary results are given for layers of SrZrO₃ made using the plasma spray technique.

NOMENCLATURE

- a thermal diffusivity
- B equivalent Biot number defined by equation (1)
- c_p, c_{pM} specific heat, and specific heat of the substance in the calorimeter, respectively
- $h_{\rm M}, h_{\rm M}^*$ specific-, and modified specific enthalpy of melting defined by equation (19), respectively
- Ja Jakob number, $c_p(T_M T_0)/h_M$
- k_c thermal conductivity of the ceramic layer under investigation
- $k_{\rm M}$ thermal conductivity of the substance in the calorimeter
- $k_{\rm P}$ thermal conductivity of the calorimeter wall
- k_z equivalent thermal conductivity of the calorimeter with the internal substance

m mass of the substance in the calorimeter

- q specific heat flux defined by equation (18)
- $\dot{Q}, \dot{Q}_1, \dot{Q}_2, \dot{Q}_3$ heat flux defined by equations (7), (9), (10), and (11), respectively
- r, r_i (i = 1, 2, ..., 5) radii according to Fig. 3, where r_1 is the outside radius of the ceramic layer, r_2 and r_3 are the outside and inside radius of the calorimeter wall, respectively, r_4 is the radius of the moving boundary occurring during melting of the substance in the calorimeter
- S, S_1, S_2 thicknesses of the ceramic layer covering the heads of the calorimeter according to Fig. 4
- T temperature, $T(r, \tau)$ (where r, radius; τ , time)
- T_A, T_B thermocouples recording the temperature in the middle of the calorimeter, and of the hot bath, respectively
- \overline{T}_{c} average temperature of the ceramic layer during the measurement (i.e. during melting of the substance in the calorimeter)

- $T_{\mathbf{k}}$ temperature of the hot bath
- $T_{\rm M}$ melting temperature of the substance in the calorimeter
- T₀ initial temperature of the calorimeter

Greek symbols

- δ thickness of the layer under investigation, $r_1 - r_2$
- $\Delta F o_{\rm M}$ dimensionless time duration of melting $\Delta H_{\rm M}$ total enthalpy of melting
- $\Delta T_{\rm M}, \Delta T_{\rm P}, \Delta T_{\rm c}$ temperature drops related with the response time lag of the calorimeter, in the wall of the calorimeter, and in the ceramic layer, respectively
- $\Delta \tau_{\rm M}, \Delta \tau_{\rm e}$ time duration of melting, and of the temperature equalization observed on the record of the temperature history $T = T(0, \tau)$ in Fig. 2
- $\theta(r, \tau)$ dimensionless temperature, [$T(r, \tau) - T_0$]/($T_k - T_0$)
- μ_n root of the characteristic equation (3)
- ρ mass density
- Σ dimensionless quantity defined by equation (17)
- τ, τ^* time, and the moment of time when melting begins in the calorimeter, respectively

1. INTRODUCTION

A NEW simple method for measuring the heat conduction coefficient was developed which is particularly suitable for the investigation layers of low thermal conductivity in high temperature ranges, i.e. cases in which classical calorimetric methods usually fail and when pulse heating methods are complicated. The essence of the method consists of using a calorimeter enclosed by a layer of the material under investigation. During the measurement a certain amount of a substance filling the calorimeter melts because of sudden immersion of the calorimeter in a hot bath, e.g. in a liquid metal. The idea of the heat conduction measurement is to measure the temperature history $T = T(\tau)$ in the middle of the calorimeter using the thermocouple T_A (Fig. 1). The spherical or cylindrical calorimeter is made of a material of good thermal conductivity and covered with the ceramic layer for investigation. In this study, layers of SrZrO₃ and Al₂O₃ with 13% wt. TiO₂ were applied using the plasma spray technique.

The calorimeter of the initial temperature $T_0 < T_M$, where $T_{\rm M}$ is the temperature of melting of the substance in the calorimeter, is suddenly immersed into the hot bath of a high temperature equal to T_k (Fig. 2). The heating of the calorimeter depends on its thermophysical properties, and mainly on the heat conduction of the outer ceramic layer. In this way, by measuring the time duration $\Delta \tau_{\rm M}$ of the change of phase of the internal substance which may be simply realized with a thermocouple, the heat conduction coefficient can be determined. Thus, it is a direct method which does not require knowledge of the heat capacity of the layer because the measurement is reduced to a simple measurement of the time duration $\Delta \tau_{M}$. Also, a stabilization of the temperature field takes place during the measurement and the influence of the initial temperature field distortion is therefore eliminated.

The method is not inherently tied to the necessity of immersing the calorimeter in the molten metal. For example, a gas volume of high temperature may be used as the hot zone. In such a case, the difficulty arises with the determination of the boundary conditions at the ceramic layer. The bare calorimeter can be used in such a case to determine experimentally these conditions. Additionally, the present method of the internal calorimeter can be used to investigate the heat exchange coefficient in various technical situations, e.g. during boiling.

2. RESPONSE TIME LAG OF THE CALORIMETER

The thermocouple which records the temperature T_A and which indicates the occurrence of melting is located in the middle of the calorimeter. Thus the beginning of this change of phase at the surface of the melting substance is registered by this thermocouple at a temperature lower by ΔT_M than the change of phase temperature T_M (Fig. 2). This effect is caused by a temperature distribution which exists in the substance at the moment when melting begins [Fig. 3(a)]. Thus in the first period of melting the total heat flux \dot{Q} divides



FIG. 1. Schematic diagram of apparatus. (1—Investigated ceramic layer, 2—Determined mass of a substance for melting, 3—Thermocouple T_{A} , 4—Hot bath, 5—Thermocouple T_{B}).



FIG. 2. Schematic diagram illustrating the idea of the heat conduction measurements (I—bare homogeneous calorimeter, i.e. without the external ceramic layer and internal substance for melting, II—homogeneous calorimeter with the external ceramic layer for investigation, III—nonhomogeneous calorimeter, i.e. with the internal substance for melting and with the ceramic layer).

into two parts: $\dot{Q}_{\rm M}$ which changes into the enthalpy of melting and $\dot{Q}_{\rm D}$ which equalizes the temperature of the substance up to the temperature $T_{\rm M}$ [Figs. 3(a) and (b)]. From that moment of time the total heat flux \dot{Q} causes purely melting of the internal substance [Fig. 3(c)] and a flat plateau of temperature is observed on the temperature record (Fig. 2). The time taken for temperature equalization $\Delta \tau_e$ (Fig. 2) will decrease as the value of the thermal diffusivity of the melting substance increases. The influence of this effect on the total time of melting $\Delta \tau_M$ decreases as the enthalpy of melting ΔH_M of the substance increases.

Thus it is essential to determine the temperature drop $\Delta T_{\rm M}$ in relation to the change of phase temperature $T_{\rm M}$ for the exact reading of the time duration $\Delta \tau_{\rm M}$. It should be also said that the temperature calculations up to the beginning of melting can be conducted as for a multilayer solid state material with transient conditions.



FIG. 3. Sequence of the temperature fields occurring during melting in the calorimeter for the following moments of time: (a) $\tau = \tau^*$; (b) $\tau^* < \tau < \tau^* + \Delta \tau_e$, and (c) $\tau^* + \Delta \tau_e < \tau < \tau^* + \Delta \tau_M$ (see Fig. 2).

It is a rather complicated task: to reach the first approximation some additional assumptions will be made.

2.1. An approximate calculation of the transient response time lag of the calorimeter

The transient state calculations for the calorimeter can be conducted as the first step using an analytical solution for an infinite cylindrical rod which is suddenly plunged into a convective environment. An infinite homogeneous cylindrical rod of radius r_2 of the equivalent heat conduction coefficient k_z is considered to represent the heat conductivity of the calorimeter without the ceramic layer because of its high thermal conductivity.

The thermal action of the ceramic layer is described by introduction of the equivalent Biot number

$$B = k_{\rm c} r_2 / \delta k_{\rm Z} = {\rm const.} \tag{1}$$

where k_e is the heat conduction coefficient of the ceramic layer (its numerical value can be assumed at the first step and improved in further calculations), and $\delta = r_1 - r_2$ is the ceramic layer thickness. This approach is connected with the omission of the transient effects in this layer and is equivalent to the omission of both the heat capacity of the ceramic layer and of the temperature variations of T_2 during the melting period. The heat capacity is really small, as are the variations of T_2 in comparison to the large temperature gradient ΔT_e occurring in the ceramic layer.

The analytical solution in this case has the following form [1]:

$$\theta(r,\tau) = \frac{T(r,\tau) - T_0}{T_k - T_0}$$

= $2B \sum_{n=1}^{\infty} \frac{J_0(\mu_n r)}{[(\mu_n r_2)^2 + B^2] J_0(\mu_n r_2)} e^{-a\mu_n^2 \tau}$ (2)

where the characteristic roots μ_n are the solution of the following characteristic equation:

$$(\mu_n r_2) J_1(\mu_n r_2) + B J_0(\mu_n r_2) = 0.$$
(3)

Additionally $a = k_z/\rho c_p$ is the thermal diffusivity of the rod where ρ , and c_p are an equivalent density of the rod, and its specific heat, respectively.

By means of equation (2), the following temperature difference can be calculated :

$$\Delta T_{\rm M} = T(r_3, \tau^*) - T(0, \tau^*)$$
 (4)

where τ^* is the time of the beginning of melting at radius r_3 , and which can be calculated from the following condition (Fig. 3):

$$T(r_3, \tau^*) = T_{\mathrm{M}}.$$
 (5)

2.2. Quasi-stationary calculations of the state before melting

obtained from the following formula:

$$T_{i} - T_{i+1} = \frac{T_{1} - T_{4}}{k_{i} \sum_{j=1}^{3} \frac{1}{k_{j}} \ln \frac{r_{j}}{r_{j+1}}} \ln \frac{r_{i}}{r_{i+1}}$$
(6)

where $i = 1, 2, 3, 4, k_i$ and $k_j = k_c, k_P, k_M$, respectively. By use of this formula the temperature drop ΔT_M for the beginning of melting can be calculated.

In equation (6) the existence of a hole in the middle of the substance before melting is taken into account. This is necessary to obtain rational results from the formula for the quasi-stationary state. It is also physically justified by the existence of the hole in the middle of the calorimeter for the thermocouple T_A . Additionally in the numerical calculations it is assumed that the temperature drop $\Delta T_M \ll \Delta T_c$.

3. CALCULATION OF HEAT CONDUCTION FROM THE TIME DURATION OF MELTING

Though the present idea of calculation of the heat conduction coefficient from the time duration $\Delta \tau_{\rm M}$ of melting of a given mass of substance looks very simple, an exact determination of this relation is still not easy because of the nonlinear character of problems with change of phase (known as the Stefan problems). The characteristic feature of these problems is the existence of a moving isothermal surface on which a liberation (or absorption) of the change of phase enthalpy takes place and which separates the new and old phases. The exact analytic solutions are known only for some plane 1dim. change of phase problems. Usually, approximate or numerical methods are used. Despite many works on this subject, only a few effective methods are available in practice. Most of them are based on the assumption that the heat capacity of the medium is negligible in relation to the change of phase enthalpy. (It is a quasistationary approximation which is exact for Jakob number $Ja \rightarrow 0$.) This approximation is equivalent to the assumption that the steady state heat conduction takes place in the region with the moving boundary $r_{4}(\tau)$ (Fig. 3). In practice (in the absence of a better solution) the quasi-stationary approximation is used, particularly for cylindrical and spherical geometries.

3.1. A simplified method of the heat conduction calculation

The first approximation of the formula for the heat conduction coefficient of the ceramic layer can be obtained, if the following assumptions are made:

(a) the properties of the material from which the calorimeter is made are assumed as for the ideal material from the heat transfer point of view, i.e. $k_{\rm P} \rightarrow \infty$ and $k_{\rm M} \rightarrow \infty$, and

(b) a negligibly small ratio of the subcooling enthalpy of the old phase[†] (i.e. the substance which

A different method of estimation of the temperature drop $\Delta T_{\rm M}$ can be based on the assumption of the quasistationary state in a multi-layered cylinder just before melting. By use of symbols as on Fig. 3(b), the temperature drop for any layer of the cylinder can be

[†] The specific enthalpy of the subcooling can be expressed in the form $\varphi c_{pM} \Delta T_M$ where the coefficient φ depends on the temperature distribution in the old phase and $0 \le \varphi \le 1$ (Fig. 3).

melts in the calorimeter) during the melting phase to the enthalpy $\Delta H_{\rm M}$ of melting.

By use of the foregoing assumptions, the rate of heat transfer to the melting substance depends directly on the enthalpy of melting $\Delta H_{\rm M}$ and on the time duration $\Delta \tau_{\rm M}$ of the phenomenon

$$\dot{Q} = \Delta H_{\rm M} / \Delta \tau_{\rm M} \tag{7}$$

where the total enthalpy of melting is

$$\Delta H_{\rm M} = mh_{\rm M} \tag{8}$$

where *m* is the total mass of the melting metal. On the other hand the heat transfer to the melting metal depends mainly on the thermophysical properties of the ceramic layer under investigaton. Having defined geometry and thickness of the layer δ , the heat transfer rate may be determined in relation to the heat conduction coefficient k_c of this layer as follows:

$$\dot{Q}_1 = 2\pi L k_c (T_k - T_{kl}) / \ln \frac{r_1}{r_2}.$$
 (9)

When additionally the heat transfer rate \dot{Q}_2 through the heads (bottom and top) of the calorimeter and \dot{Q}_3 through a fin, which is formed by the small pipe for thermocouple T_A and which keeps up the calorimeter in the hot bath (Fig. 1), are taken into account, then the thermal energy balance of the calorimeter takes the form

$$\dot{Q} = \dot{Q}_1 + \dot{Q}_2 + \dot{Q}_3.$$
 (10)

In practice it can be taken that $\dot{Q}_3 \simeq 0$, and that the heat transfer through the heads is

$$\dot{Q}_2 = \pi r_1^2 (T_k - T_M) \left(\frac{1}{S_1} + \frac{1}{S_2} \right) k_c$$
 (11)

where S_1 and S_2 are thicknesses of the ceramic layer on both sides of the heads [Fig. 4(b)]. From equation (10) and by use of equations (7)-(9), and (11) one can obtain a simple formula for the heat conduction coefficient of the ceramic layer in the form

$$k_{\rm c} = mh_{\rm M} \left/ \left[2\frac{L}{\ln\frac{r_1}{r_2}} + r_1^2 \left(\frac{1}{S_1} + \frac{1}{S_2}\right) \right] (T_{\rm k} - T_{\rm M}) \Delta \tau_{\rm M}.$$
(12)

Thus, from measurements of the time duration $\Delta \tau_{\rm M}$, the value of the heat conduction coefficient $k_{\rm c}$ can be determined. Of course, the effect of the time response delay of the calorimeter should be taken into account.

The use of equation (12) is connected with the assumption that the heat transfer through both heads of the calorimeter is comparable. In fact, at the top of the calorimeter a gas gap exists which disturbs the heat transfer. To avoid this effect an internal fin [Figs. 4(a) and (b)] was used, which facilitates the heat transfer from this side of the calorimeter surface to the melting substance.

A different approach to the problem is shown on Fig. 4(c), where the top part of the calorimeter erects above the hot bath level. In this case, equation (12) reduces to the following expression:

$$k_{\rm c} = mh_{\rm M} \left| \left(\frac{2L}{\ln \frac{r_1}{r_2}} + \frac{r_1^2}{S} \right) (T_{\rm k} - T_{\rm M}) \Delta \tau_{\rm M}. \right.$$
(13)

Some correction of the calculation results can also be obtained when instead of the temperature $(T_k - T_{hl})$ the temperature drop ΔT_c is introduced in equations (12) and (13) according to the following equation [Fig. 3(a)]:

$$\Delta T_{\rm c} = (T_{\rm k} - T_{\rm M}) - \Delta T_{\rm P} \tag{14}$$

where $\Delta T_{\rm P}$ can be taken according to equation (6).



FIG. 4. Simplified design of the calorimeters : (a) closed one-point calorimeter, (b) closed two-point calorimeter, (c) 'open' one-point calorimeter.

The value of the heat transfer coefficient from equations (12) or (13) should be related with the mean temperature \overline{T}_c of the ceramic layer

$$\overline{T}_{c} = T_{k} - \Delta T_{c}/2. \tag{15}$$

An additional source of an error is the omission of the temperature drop of T_k at the calorimeter surface. This effect can also be simply estimated and incorporated into equation (14) by use of the analytic solution for the infinite medium with a cylindrical cavity which is cooled by a constant heat flux.

3.2. Second approximation of the heat conduction calculations

Among the approximate methods (the next step of the quasi-stationary solutions) good results of the time duration calculations can be obtained by the method in which the temperature field is related to a form of the function which describes the moving boundary position of the change of phase [2]. In this method the initial nonlinear boundary problem is reduced to a determination of the boundary position $r_{4}(\tau)$ from the nonlinear ordinary differential equation. At the same time a partial solution is used which satisfies the heat conduction equation and the boundary conditions on the change of phase boundary. The partial solution of the problem has a form of the infinite functional set, which relates the temperature distribution to the $r_4(\tau)$. It is a solution of the so-called inversed Stefan problem. Approximating the temperature field by first terms of this solution, one comes to an approximate equation for $r_4(\tau)$, which in many cases can be solved analytically for various boundary conditions and for the three basic geometries: plane, cylindrical and spherical. In this way the following formula was obtained for the total time duration of the change of phase of the infinite cylindrical rod which was subjected to the heat flux q[2]:

$$\Delta Fo_{\rm M} = \frac{1}{\Sigma} + \frac{1}{2} \begin{cases} \frac{1}{(1-\Sigma)^{1/2}} \ln \frac{\Sigma^{1/2}}{1-(1-\Sigma)^{1/2}} & \text{for } \Sigma < 1\\ \frac{1}{(\Sigma-1)^{1/2}} & \text{arcsin} (1-1/\Sigma)^{1/2} & \text{for } \Sigma \ge 1 \end{cases}$$

where $\Delta F o_{\rm M} = a \Delta \tau_{\rm M} / r_3^2$ is the dimensionless time duration of melting and the quantity

$$\Sigma = 2c_{pM}r_3q/h_Mk_M \tag{17}$$

characterizes the thermophysical properties of the melting substance through their specific heat c_{pM} , heat conduction coefficient k_M and h_M and also takes into account the heat transfer rate to the melting substance through the heat flux q. In the present method of the heat conduction calculations the heat flux is approximated by the formula

$$q = k_c \Delta T_c / r_2 \ln \frac{r_1}{r_2}.$$
 (18)

The use of equation (18) is connected with an assumption of $\Delta T_{\rm c} = \text{const.}$, what is justified for the

case of large temperature gradients ΔT_c and low heat conduction coefficient k_c . For $\Delta T_c \neq \text{const.}$ in time, a mean value of ΔT_c can be introduced into this formula.

Equation (16) for the time duration $\Delta Fo_{\rm M}$ was derived for the simplified case, when the old phase is in the equalized temperature $T_{\rm M}$. In fact, some temperature distribution exists, a consequence of the heat transfer with the ambient before the change of phase period. A modified change of phase specific enthalpy

$$h_{\rm M}^* = h_{\rm M} + \varphi c_{p\rm M} \Delta T_{\rm M} \tag{19}$$

can be introduced instead of $h_{\rm M}$ into equation (17) to reduce the error which follows from the omission of the temperature distribution in the old phase.

It can be seen that the second term in equation (16) is a correction to the quasi-stationary solution of the problem. On Fig. 5, the function $\Delta Fo_{\rm M} = f(\Sigma)$ is shown for the quasi-stationary solution, next calculated according to equation (16) and finally calculated by the use of some transformation rule of the plane solution for the cylindrical geometry [3]. It should be underlined that equation (16) as well as the curves on Fig. 5 concern the melting of the infinite cylindrical rod.

To use the foregoing theory for the interpretation of the measurement results with the calorimeter of finite dimensions the following steps are suggested:

(1) Calculation of k_c by use of equation (12) [or equation (13)] as the first approximation.

(2) Next using the above value of k_c the time duration of melting should be calculated by use of equation (16) and compared with the quasi-stationary solution $Fo_M = 1/\Sigma$. From this comparison a conclusion can be drawn about how to modify the measured time duration $\Delta \tau_M$ and a modified value $\Delta \tau_M^*$ of this time should be obtained.

(3) By use of the modified time duration $\Delta \tau_{\mathbf{k}}^*$, the value of the heat conduction coefficient k_c is calculated by use of equation (12) [or equation (13)], and in this



FIG. 5. Dimensionless total time of melting ΔFo_M as a function of the characteristic number Σ [(a) quasi-stationary approximation, (b) curve according to equation (16), (c) transformation of the plane solution [3].

final calculation all corrections according to equations (14) and (19) should be introduced.

4. GEOMETRY AND DESIGN OF THE CALORIMETER

The present investigation was conducted using finite cylindrical calorimeters. One- and two-point calorimeters were developed. The one-point calorimeter is shown in Fig. 4(a), and this represents a simple technical realization of the described method. In the two-point calorimeter [Fig. 4(b)], two substances of different melting points T_{M1} and T_{M2} are used. The temperatures fulfil the condition that $T_0 < T_{M1} < T_M < T_k$. In this way, two values of the heat conduction coefficient for two different mean temperatures \bar{T}_{c1} and \bar{T}_{c2} of the ceramic layer can be obtained in one measurement. In the calorimeter of this type the finite values of the heat capacity and heat conductivity of the construction materials play a more important role. Thus the temperature drops ΔT_{M1} , ΔT_{M2} , ΔT_{P1} and ΔT_{P2} can be specified, and these should be taken into account in the calculations of k_{c} .

In Fig. 4(c) a modified design of the calorimeter is presented which eliminates the problems connected with the temperature field disturbance at the top head of the calorimeter.

5. MEASUREMENT OF THE HEAT CONDUCTION OF THE PLASMA-SPRAYED CERAMIC LAYERS

Measurements of the heat conduction coefficient of the ceramic layers of $SrZrO_3$ and $Al_2O_3 + TiO_2$ (13%) wt.) were conducted. The layers were made by the plasma-spray technique and they had thicknesses from 0.4 to 1.15 mm. Under each ceramic layer, a 0.1 mm thin-ground layer of AlNi (404) was applied. The grain diameter of the ceramic powders for spraying was ~ 50 μm for SrZrO₃ and ~20 μm for Al₂O₃+TiO₂. The calorimeter was immersed in a bath of molten aluminium in the range of temperatures from about 680 to about 1230°C. Zinc, aluminium and copper were used in the one-point calorimeter. (Dimensions ϕ 18 dia. ×40 mm.) In the two-point calorimeter, a Zn/Al pair was taken, and some tests were made with a Al/Cu pair. Both measurements were done with calorimeters of ϕ 23 dia. × 45 mm.

Induction heating of the melting crucible was used (switched off during measurements) and two chromelalumel thermocouples of $\phi 0.5$ mm dia. Three measuring channels were available with tape feed of up to 500 mm min⁻¹. By use of the thermocouple T_A , the time history of the inside temperature could be recorded, as well its time derivative $dT_A/d\tau$, and by means of the thermocouple T_B , the temperature of the molten metal bath (Fig. 1) could be measured. The selected temperature records of T_A and T_B are shown in Figs. 6 and 7 from which it follows that $k_c = 1.080$ W m⁻¹ K⁻¹ for $T_c = 1140^{\circ}$ C and $k_c = 0.925$ W m⁻¹ K⁻¹ for $\overline{T}_c = 1155^{\circ}$ C. A comparison of the T_A and $dT_A/d\tau$



FIG. 6. Temperature history for SrZrO₃ layer measured by means of a closed one-point calorimeter with copper as a melting substance (m = 62.58 g, L = 41.1 mm, $r_1/r_2 = 9.5/$ 9.1 mm, $S_1 = 0.5$ mm, $S_2 = 0.6$ mm) Case No. 1.



FIG. 7. Temperature history for SrZrO₃ layer measured by means of a closed one-point calorimeter with copper as a melting substance (m = 62.58 g, L = 41.1 mm, $r_1/r_2 = 9.5/$ 9.1 mm, $S_1 = 0.5$ mm, $S_2 = 0.6$ mm) Case No. 2. (T.F., failure of the thermocouple $T_{A.}$)

recording indicates first of all that the final point of the melting is unmistakably determined, but the starting point of the change of phase requires some additional considerations. In the case of the SrZrO₃ layer this range of uncertainty is, however, narrow for temperatures above ~1100°C and copper as the melting substance. The time record of dT_A/dt indicates the existence of characteristic inflections just before melting which correspond to the temperature drop ΔT_M of ~12°C in Fig. 6 and ~8°C in Fig. 7. A calculation of ΔT_M results respectively in values from 10 to 20°C for the approximate transient and quasi-stationary approach.

It was finally found that the heat conduction coefficient of $SrZrO_3$ is of order 1 W m⁻¹ K⁻¹ and does not depend on the mean temperature of the layer from about 550°C up to about 1150°C. The thermal conductivity of $ZrO_2 + TiO_2$ was found to be a little higher, up to 1.5 W m⁻¹ K⁻¹, but the small number of measurements possible did not allow a precise value to be determined. These results should be considered as preliminary, because the main purpose of the work was to check the measurement method.

6. CONCLUSIONS

The present investigation and measurements proved the efficiency and usefulness of the internal calorimeter method for the determination of the heat transfer coefficient in the broad range of temperatures. The proposed method can be realized in various ways, e.g. by use of one-point and two-point calorimeters. The method can also be used for other tasks, as e.g. for investigation of the heat transfer coefficient. The calculation method could still be further refined, but the use of even very simple expressions given in the paper allows satisfactory results to be obtained. Further work on this method will be aimed at developing the construction of the calorimeter, checking the reproducibility of the measurements, and determining the accuracy of the method.

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NOUVELLE MÉTHODE DE MESURE DU COEFFICIENT DE CONDUCTIVITÉ THERMIQUE DES FINES COUCHES CÉRAMIQUES EN HAUTS TEMPERATURES

Résumé—On présente la nouvelle, simple méthode de mesure du coefficient de conductivité thermique des fines couches céramiques. Le principe de cette méthode se contient à l'utilisation du calorimètre entouré à l'extérieure de couche étudiée. Cependant à l'intérieure du calorimètre on trouve une matièce qui se fonds quand le calorimètre est introduit en bain suffisement chand. On a presenté l'interprétation théorique et la méthode de calculs du coefficient de conductivité thermique de couche en se basant sue le temps de durée de transformation de phase qui se déroule à l'intérieure du calorimètre. On a fait des mesures et on a presenté des résultas préliminaires pour des couches SrZrO₃ et Al₂O₃ + TiO₂ (13% wt).

EIN NEUES VERFAHREN ZUR MESSUNG DES WÄRMELEITFÄHIGKEITSKOEFFIZIENTEN VON DÜNNEN KERAMIKSCHICHTEN BEI HOHEN TEMPERATUREN

Zusammenfassung – Ein neues einfaches Verfahren zur Messung des Wärmeleitfähigkeitskoeffizienten von dünnen Keramikschichten ist beschrieben. Das Wesen der Methode besteht in der Benutzung eines Kalorimeters, welches mit der untersuchten Keramikschicht bedeckt ist. Im Kalorimeter befindet sich eine Substanz, welche schmelzen wird, wenn das Kalorimeter in ein angemessen heisses Bad eingetaucht ist. Eine theoretische Interpretation und eine Methode der Leitfähigkeitskoeffizientenberechnung-welche von der Zeit-dauer der Phasenumwandlug im Kalorimeter ausgeht-ist gegeben. In der Arbeit sind die Resultate der Vormessungen für SrZrO₃ und Al₂O₃+TiO₂ (13% wt.)-Schichten angegeben.

НОВЫЙ МЕТОД ИЗМЕРЕНИЯ КОЭФФИЦИЕНТА ТЕПЛОПРОВОДНОСТИ ТОНКИХ КЕРАМИЧЕСКИХ ПОКРЫТИ В ВЫСОКИХ ТЕМПЕРАТУРАХ

Аннотация—В работе описан простой новый метод измерения коэффициента теплопроводности керамических покрытий. Сушность этого метода состоит в употреблении калориметра покрытого снаружи испытываемым покрытием. Внутри калориметра находится исследуемое вещество, которое растопится если калориметр будет помещён в соответствующую горячую баню.

В работе представлены теоретические обоснования и способ расчёта коэффициента теплопроводности покрытия на основании продолжительности фазового перехода происходящего в калориметре.

Приведены результаты измерений для покрытий SrZrO3 и Al2O3 + TiO2 (13%).