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Advances on the hot wire technique

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Abstract

The hot wire technique is considered to be an effective and accurate means of determining the thermal conductivity of ceramic materials. However, specifically for materials of high thermal conductivity, the appropriate time interval to be considered in calculations is a decisive factor for getting accurate and consistent results. In this work, a numerical simulation model is proposed with the purpose of determining the minimum and maximum measuring time for the hot wire parallel technique. Nevertheless, the factor that limits the application of the hot wire technique for high thermal conductivity materials is the maximum measuring time for the experimental thermal transient registration. An alternative experimental arrangement is then proposed for materials with thermal conductivity higher than 15 W/mK. In this technique, the time interval considered for the calculations may be several times bigger than that one corresponding to the parallel technique, what means in practice that it may be applicable for materials with higher thermal conductivities. Fourteen different specimens of refractory materials, and four polymers, with thermal conductivities ranging from 0.04 to 14 W/mK, in shape of rectangular parallelepipeds were employed in the experimental programme. An empirical equation determining minimum and maximum measuring times is also obtained for the hot wire parallel technique. In this work is also proposed a technique of insulation of the hot wire, what makes possible its application to electrical conducting materials.

Keywords: Thermal properties; Thermal conductivity; Electrical conductivity; Refractories; Hot wire technique

1. Introduction

Thermal conductivity, thermal diffusivity and specific heat are the three most important physical properties of a material that are needed for heat transfer calculations. The equation relating these properties is given by

$$a = \frac{k}{\rho c_p} \tag{1}$$

where a = thermal diffusivity (m^2/s), k = thermal conductivity (W/mK), ρ = bulk density (kg/m^3) and c_p = specific heat at constant pressure (J/kg K).

Thermal conductivity is the property that determines the working temperature levels of a material, and it is an important parameter in problems involving steady state heat transfer. However, it is one of the physical quantities whose measurement is very difficult and it requires high precision in the determination of the parameters involved in its calculations.

Nowadays, several techniques are available for the determination of thermal conductivity of different materials. However, for refractory materials the hot wire technique is considered to be an effective and accurate means of determining this thermal property. Four variations of the hot wire method are known[1]: hot wire standard technique, hot wire resistance technique, two thermocouple technique and the hot wire parallel technique. The theoretical model is the same, and the basic difference among these variations lies in the temperature measurement procedure. Two pieces are required whatever is the variation to be used. In this work, the hot wire parallel technique and a modification of the standard technique called surface technique are employed in the experimental programme.

The hot wire technique is an absolute, non-steady state and direct method, and therefore, it makes the use of standards unnecessary. This technique was first described by Schieirmacher in 1888,[2] and its first practical application was reported in 1949 by Van der Held and Van Drunen,[3] in the determination of the thermal conductivity of liquids. However, it was Haupin[4] who in 1960 first used this method to measure the thermal conductivity of ceramic materials. Nowadays, the hot wire method

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is considered an effective and accurate means of determining the thermal conductivity of a wide range of materials, including ceramics,[5–7] fluids[8–11] and polymers.[12–14] In addition, with the hot wire technique the concept of "mean temperature" between hot and cold face of a sample in thermal conductivity calculations is eliminated, since the measurement is carried out at a fixed temperature.[15] The temperature gradient across the sample is very low, and this is another virtue of this technique, since an ideal method for measuring the thermal conductivity would have to be capable of measuring this property across a zero temperature gradient throughout the sample.

In the mathematical formulation of the method, the hot wire is assumed to be an ideal, infinitely thin and long heat source which is in an infinite surrounding material, whose thermal conductivity is to be determined. This assumption implies that the temperature transient that is picked up by the thermocouple joint, at the measuring point, during the experiment cannot be altered by the fact that the actual sample has finite dimensions. These considerations imply some restrictions in the applicability of the hot wire technique in terms of possible sample sizes and thermal conductivity allowable ranges.

Applying a constant electric current through the wire, a constant amount of heat per unit time and unit length is released by the wire and propagates throughout the material. In practice, the theoretical infinite linear source is approached by a thin electric resistance and the infinite solid is replaced by a finite sample. In the plot of the recorded temperature at the measuring point as a function of time, the early part of the curve must be neglected in calculations because of the non-vanishing contact resistance between wire and sample, and the heat capacity of the wire. Also, a limit to the maximum measuring time has to be considered in the calculations because of the finite sample size. When heat reaches the outer surface of the sample, heat exchanges between the sample and the environment make the theoretical model no longer valid. The intermediate region of the curve where the theoretical model is valid defines the time limits to be considered in any measurement. So, the correct determination of the minimum and maximum times to be considered in the calculations is of fundamental importance for getting accurate and consistent results.

Applying a constant electric current throughout the wire, and recording the temperature increase at the measuring point, located at a distance r from the hot wire (parallel technique) thermal conductivity, is calculated according to the following equation[16]:

$$k = \frac{-q'}{4\pi T(t)} E_i\left(\frac{-\rho c_p r^2}{4kt}\right) \tag{2}$$

where k = thermal conductivity of the material (W/mK), q' = linear power density (W/m), ρ = material bulk density (kg/m^3), c_p = specific heat at constant pressure (J/kg K), r = distance between hot wire and thermocouple (m), t = elapsed time after beginning of heat release (s), $T(t)$ = temperature rise registered by the thermocouple related to the initial reference temperature (K), and $-E_i(-x)$ is the exponential integral function given by

$$-E_i(-x) = \int_x^{\infty} \frac{e^{-t}}{t}\, dt \tag{3}$$

Eq. (3) may be approximated by the following power series:

$$-E_i(-x) = E1(x) = -\gamma - \ln x - \sum_{n=1}^{\infty} \frac{(-1)^n x^n}{nn!} \tag{4}$$

since $|\arg x| < \pi$, and $\arg x = \arctan(x_2/x_1)$, where $x = x_1 + ix_2$, and $\gamma = 0.5772156649\ldots$ is the Euler constant.

When the temperature transient is recorded at a point very close to the surface wire (surface technique), the equation for the thermal conductivity calculation may be derived from Eqs. (2) and (4) according to the following approximation.[17]

If $r^2/4at$ is very small, the function $-E_i(-x)$, given by Eq. (4) may be approximated only by the first two terms: $\gamma - \ln(x)$. So, the temperature very close to the hot wire may be given by

$$T = \frac{q'}{4\pi k}\left(\ln \frac{4at}{r^2} - \gamma\right) \tag{5}$$

Eq. (5) shows that the thermal conductivity may be evaluated from the slope of the curve temperature against the log of the time. Since the hot wire is not an ideal line source, and the sample has a finite size, similar restrictions to minimum and maximum measuring times are also applicable like in the hot wire parallel technique.

When using the hot wire parallel technique, the calculations, starting from the recorded temperature transient in the sample are carried out by using a non-linear least squares fitting method.[5] Both thermal conductivity and specific heat in Eq. (2) are fitted in order to obtain the best possible approximation between the thermal transient experimentally registered and that one predicted by the theoretical model. In this case, these two thermal properties, thermal conductivity and specific heat are simultaneously determined from the same experimental transient, and with the knowledge of the density, the thermal diffusivity is then derived using Eq. (1). When using the surface technique, the thermal conductivity is determined by Eq. (5) from the slope of the curve temperature against the log of the time.

The apparatus used in this work, is fully automatic, and the transient of temperature detected by the thermocouple is recorded and processed by a computer via an analog-to-digital converter using a software specially written for this purpose.

2. The simulation model

Since the method assumes an infinitely long and thin heat source, the heat conduction inside the specimen is of a radial nature.[5,18] It is assumed that the solid is composed of N concentric individual layers with radii r_i measured from the centre of the specimen where the hot wire is embedded. Fig. 1 illustrates this procedure.

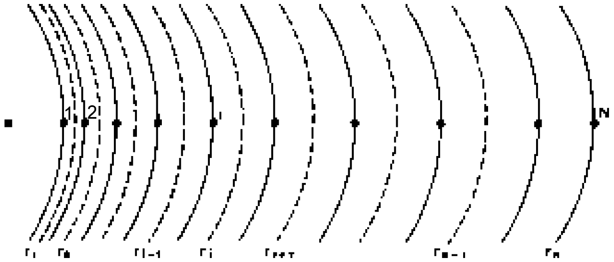

Fig. 1. Annular cylindrical regions for the numerical analysis.

An energy balance of the form of Eq. (6) may be defined for each region:

$$[\text{Heat flux in}]_i - [\text{Heat flux out}]_i = [\text{Rate of internal energy change}]_i \tag{6}$$

Then by applying balance Eq. (6) for each region, we derive Eqs. (7)–(9).

For $i = 1$,

$$q' + k2\pi\left(r_1 + \frac{r_2 - r_1}{2}\right)\frac{\langle T_2\rangle - \langle T_1\rangle}{r_2 - r_1} = \rho c_p \pi\left[\left(r_1 + \frac{r_2 - r_1}{2}\right)^2 - \left(r_1 - \frac{r_2 - r_1}{2}\right)^2\right]\frac{T_1^{t+\Delta t} - T_1^t}{\Delta t} \tag{7}$$

For $i = 2, 3, \ldots, N-1$,

$$k2\pi\left(r_i - \frac{r_i - r_{i-1}}{2}\right)\frac{\langle T_{i-1}\rangle - \langle T_i\rangle}{r_i - r_{i-1}} + k2\pi\left(r_i + \frac{r_{i+1} - r_i}{2}\right)\frac{\langle T_{i+1}\rangle - \langle T_i\rangle}{r_{i+1} - r_i} = \rho c_p \pi\left[\left(r_i + \frac{r_{i+1} - r_i}{2}\right)^2 - \left(r_i + \frac{r_i - r_{i-1}}{2}\right)^2\right] \times \frac{T_i^{t+\Delta t} - T_i^t}{\Delta t} \tag{8}$$

For $i = \mathrm{N}$,

$$k2\pi\left(r_N - \frac{r_N - r_{N-1}}{2}\right)\frac{\langle T_{N-1}\rangle - \langle T_N\rangle}{r_N - r_{N-1}} = \rho c_p \pi\left[r_N^2 - \left(r_N - \frac{r_N - r_{N-1}}{2}\right)^2\right]\frac{T_N^{t+\Delta t} - T_N^t}{\Delta t} \tag{9}$$

where $\langle \mathrm{Ti}\rangle$ = average temperature of the region i between times t and $(t + \Delta t)$, t = time and r_{i} = radius of the region i.

In order to facilitate the numerical calculations, a dimensionless transformation for position, time and temperature, as shown in Eqs. (10)–(12) are introduced.

$$r_i^* = \frac{r_i}{L_{\mathrm{ref}}} \tag{10}$$

$$\tau = \frac{at}{L_{\mathrm{ref}}^2} \tag{11}$$

$$\theta_i = \frac{T_i - T_{\mathrm{rt}}}{T_{\mathrm{ref}}} \tag{12}$$

where L_{ref} = any reference linear dimension (distance hot wire-thermocouple, for example), T_{rt} = room temperature, T_{ref} = any reference temperature (water vaporization temperature, for example), r_i^* = dimensionless radius of the region i, τ = dimensionless time and θ_i = dimensionless temperature of the region i.

Taking as the dimensionless average temperature for each region, the arithmetic mean of the temperatures between instants t and $t + \Delta t$, one can write Eqs. (13)–(15).

$$\langle\theta_{i-1}\rangle = \frac{\theta_{i-1}^{t+\Delta t} + \theta_{i-1}^t}{2} \tag{13}$$

$$\langle\theta_i\rangle = \frac{\theta_i^{t+\Delta t} + \theta_i^t}{2} \tag{14}$$

$$\langle\theta_{i+1}\rangle = \frac{\theta_{i+1}^{t+\Delta t} + \theta_{i+1}^t}{2} \tag{15}$$

Combining Eqs. (7)–(15), and after some algebraic simplifications, the final result may be expressed in a matrix form given by Eq. (16).

$$B^K\theta^{K+1} = C^K\theta^K + D \tag{16}$$

where θ = column matrix of dimensionless temperatures, B and C = square matrices of dimensionless temperature coefficients terms, D = column matrix of dimensionless independent coefficients terms, and the superscripts K and $K + 1$ refer to the times t and $t + \Delta t$.

The temperature profile generated by Eq. (16) at the measuring point Mp and at the edge of the sample can be used to determine the minimum and maximum measuring times to be considered in the calculations.

3. Experimental programme

For the parallel technique, 14 different specimens of refractory materials, and four polymers, in the shape of rectangular parallelepipeds were employed in the experimental programme.

When it is possible to estimate the thermal conductivity and specific heat of the specimen to be tested, those values can be used as input data for the simulation model programme, in order to determine the adequate minimum and maximum measuring times for the hot wire parallel technique. However, if this estimate is not possible for any reason, an initial run of the hot wire technique will provide these values.

When the appropriate region of the temperature profile at the measuring point Mp is considered in the calculations, the resulting thermal property values are approximately constant, irrespective of the time interval considered inside this region. In this work, the criteria to determine the minimum and maximum measuring times were the increase by half a degree centigrade in the temperature at the measuring point and at the edge of the sample compared with the initial reference temperature. The measuring point Mp was kept at 16 mm from the hot wire.

Table 1
Sample details

Sample	Material	$t_{\min}$ (s)	$t_{\max}$ (s)	k (W/mK)	c_p (J/kg K)	a ($\times 10^{-7}$ m^2/s)	R^*
A1	Silicon carbide	12	81	13.7431	777.2289	67.745	0.99955
A2	Fired magnesia	11	88	11.0601	605.8201	61.725	0.99897
A3	Alumina zircon	17	140	4.2704	455.1872	27.355	0.99958
A4	Alumina	22	177	4.1034	669.3070	21.145	0.99960
A5	Alumina	31	225	4.0369	693.7408	20.070	0.99937
A6	Alumina	28	243	2.4580	667.9017	13.730	0.99835
A7	Mulite	30	246	1.9688	674.5910	12.370	0.99994
A8	Mulite	31	261	2.9872	766.5044	14.710	0.99964
A9	Alumina	43	400	1.5463	731.4535	8.558	0.99983
A10	Vermiculite	42	400	0.2524	613.3826	5.607	0.99997
A11	Calcium silicate	58	540	0.0656	963.9372	3.037	0.99995
A12	Calcium silicate	84	765	0.0416	991.5800	1.874	0.99995
A13	Portland concrete	88	792	0.1317	1253.1149	3.165	0.99993
A14	Refractory concrete	108	957	0.4650	1226.0105	2.218	0.99975
A15	Rigid PVC	205	1650	0.1962	1004.6938	1.426	0.99849
A16	Acrylic	225	1775	0.2012	1411.9189	1.197	0.99932
A17	Polypropylene	225	2286	0.2331	1814.9901	1.417	0.99951
A18	Nylon	260	2499	0.2960	1786.6821	1.441	0.99798

R^* = correlation coefficient.

For the surface technique, six different samples with thermal conductivities ranging from 0.25 to 13 W/mK were selected from Table 1. In this case, the measuring point Mp was kept less than 1 mm from the hot wire.

Specific procedures for sample arrangements were described in several occasions.[1,6,19]

4. Results and discussions

Table 1 shows the experimental results obtained with the parallel technique for all samples.

Fig. 2 displays the temperature profile at the measuring point Mp during the measuring interval, evaluated with the simulation model, and experimentally determined with the hot wire parallel technique, for two selected samples: the sample with the highest thermal diffusivity, and that one with the lowest thermal diffusivity. Both plots show an excellent agreement between the results predicted by the simulation model and those ones experimentally determined by the hot wire parallel method. This agreement assures the correct determination of the temperature profile at the measuring point and at the edge of the sample, and consequently the exact instant of the beginning of heat exchange between surface sample and environment, which defines the appropriate region of the thermal transient to be considered in the calculations.

Since the heat conduction inside the specimen is of a radial nature and the samples tested have the parallelepiped shape, the heat exchange starts at points of the sample surface at a distance r_s, the radius of the maximum cylinder inscribed in the experimental arrangement and having its central axis in the hot wire. After a relatively long elapsed time t_e, all the points on the surface will be exchanging heat with the environment, and then a stable temperature at the measuring point Mp is registered, as shown in Fig. 3. The value of the time t_e depends on the samples thermal diffusivity, relation between the parallelepiped dimensions, and the environmental conditions.

Figs. 4 and 5 display minimum and maximum measuring times as a function of the thermal diffusivity, since this is the property that determines the minimum and maximum measuring

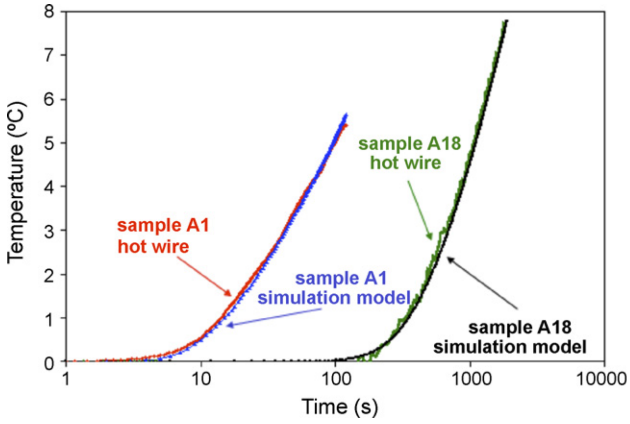

Fig. 2. Temperature profile at measuring point.

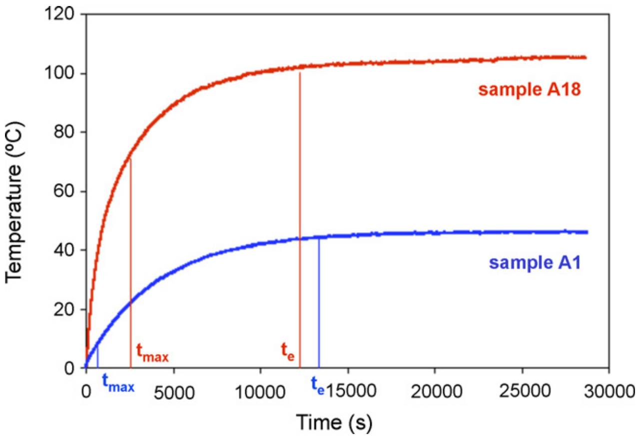

Fig. 3. Temperature profile at measuring point: stable temperature after a long time.

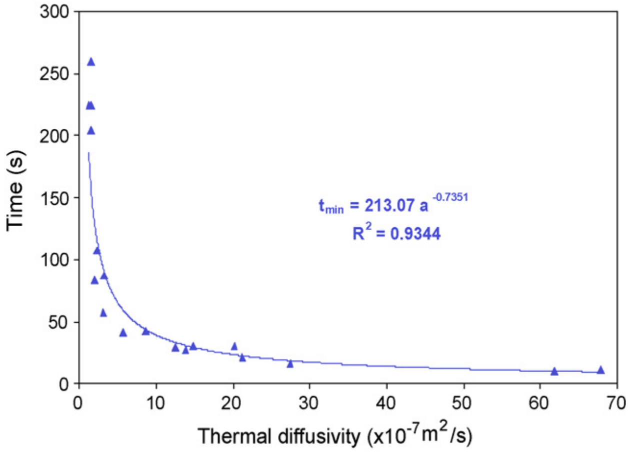

Fig. 4. Minimum measuring time as a function of the thermal diffusivity.

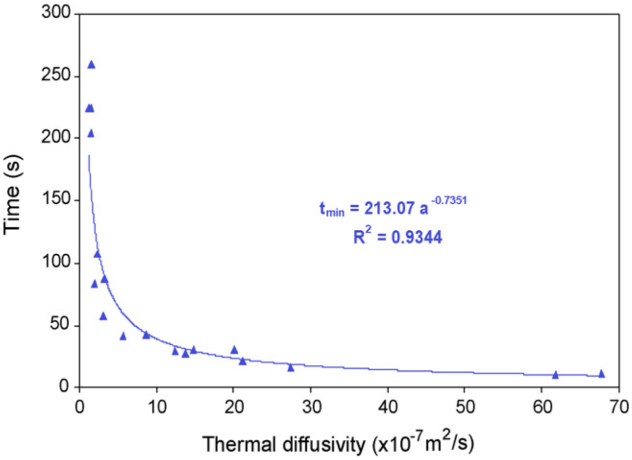

Fig. 5. Maximum measuring time as a function of the thermal diffusivity.

times. Parameters $t_{\min}$ and $t_{\max}$ were fitted in order to provide a function relating these parameters and the thermal diffusivity. Correlation factor R^2 shows the quality of the fitting, indicating the validity of this procedure. Both equations should be used for an initial estimate of $t_{\min}$ and $t_{\max}$.

Table 2 shows the experimental results obtained with the surface technique checked against those ones obtained with the parallel technique, as well as the correlation coefficient. The maximum deviation (7% for sample A4) is slightly higher the experimental error associated to each measurement, showing a good agreement between both techniques.

According to DIN 51046 standard, with the parallel technique is possible to measure thermal conductivities up to 25 W/mK. However, for thermal conductivities above 15 W/mK, the measurements become troublesome. The hot wire and thermocouple embedding technique start to play a fundamental role on the value of the thermal conductivity experimentally determined. Nevertheless, the factor that limits the parallel technique for high thermal conductivity materials is the maximum measuring time $t_{\max}$, since data must be collected before the beginning of heat exchange between the sample and the environment. So, for materials with thermal conductivity above 15 W/mK, the appropriate time interval becomes very small, what may compromise the accuracy of the results. This limitation is partially removed when the surface technique is employed, since in this case the available time interval is bigger when compared to the parallel technique, as shown for sample A1. For this sample, the time interval considered for calculations was [10, 300] for the surface technique instead of [12, 81] for the parallel technique. Since it is not necessary a so long interval like [10, 300] in order to get reliable results, it means that decreasing this time interval it is possible to apply this technique for materials with higher thermal conductivities.

The hot wire technique faces basically two limitations: high thermal conductivity, and electrical conducting materials. Concerning the first limitation,[16,20,21] as higher is the thermal conductivity, as smaller is the time interval to be considered in the calculations, what may compromise the reliability of the results obtained. This limitation may be partially solved with the surface technique, since the time interval available for the calculations becomes bigger. Another solution for high thermal conductivity materials should be the to increase the sample sizes. This procedure however has practical and economical limitations.

Concerning the second limitation, the restriction to the application of the hot wire for electrical conducting materials may be solved if any process of insulation between the hot wire and the samples is introduced. However, this process cannot alter the results that should be obtained without the insulation. In this work, an aqueous solution of alumina is applied on both faces of two electrical conducting materials and the thermal conductivity at room temperature is determined using the surface technique. Results obtained were 14.34 and 8.69 W/mK.

The influence of the alumina solution applied onto the faces of the samples was checked experimentally using an electrical insulating sample, and making measurements in the same conditions with and without coating. Experimental results obtained

Table 2
Experimental results – surface technique x parallel technique

	H.W.P.T., *k* (W/mK)	*R*	$t_{\min}$ (s)	$t_{\max}$ (s)	H.W.S.T., *k* (W/mK)	*R*	$t_{\min}$ (s)	$t_{\max}$ (s)	δ (%)
A10	0.2524	0.9999	42	400	0.2525	0.9997	33	5100	0.04
A9	1.5463	0.9997	38	338	1.4595	0.9926	98	1850	5.95
A4	4.1034	0.9996	22	177	3.8263	0.9511	70	850	7.24
A3	4.2704	0.9996	17	140	4.5445	0.9834	15	800	6.42
A2	11.0601	0.9990	11	88	10.3842	0.9987	10	400	6.51
A1	13.7431	0.9996	12	81	13.4626	0.9382	10	300	2.08

H.W.P.T. = hot wire parallel technique and H.W.S.T. = hot wire surface technique.

were 0.25 W/mK for the non-coated sample, and 0.24 W/mK for the coated sample. This result clearly shows that the influence of the alumina coating is negligible, since the deviation between measurements with and without coating is only 4% and it lies within the accuracy of this technique that is 5%.

5. Conclusions

A numerical simulation model is proposed to determine minimum and maximum measuring times in the hot wire parallel technique, with an excellent agreement between the results predicted by this model and those ones obtained experimentally. The importance of the knowledge of the appropriate time interval to be considered in the calculations lies in the accuracy and reliability of the experimental results obtained. The influence of $t_{\min}$ and $t_{\max}$ in the hot wire parallel calculations is greatly increased as thermal conductivity increases. With this simulation model it is also possible to determine minimum sample dimensions for a previous measuring time interval stated. The surface technique is also proposed as an alternative arrangement for materials with higher thermal conductivities. Finally, it is important to remember that although the insulating process introduced in this work is efficient in the application of the hot wire for electrically conducting materials, normally good electrical conductors are also good thermal conductors, and so, the restriction to high thermal conductivity materials is still valid.

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