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Numerical and experimental determination of the minimum and maximum measuring times for the hot wire parallel technique
(Determinação numérica e experimental dos tempos máximo e mínimo na técnica de fio quente paralelo)

W. N. dos Santos, R. Gregório
Departamento de Engenharia de Materiais - DEMa
Universidade Federal de S. Carlos - UFSCar
Rod. Washington Luiz, km 235, C.P. 676, S. Carlos, SP, 13565-905
dwns@power.ufscar.br

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 diffusivity, 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 aim of determining the minimum and maximum measuring time for the hot wire parallel technique. The temperature profile generated by this model is in excellent agreement with that one experimentally obtained by this technique, where thermal conductivity, thermal diffusivity and specific heat are simultaneously determined from the same experimental temperature transient. Eighteen different specimens of refractory materials and polymers, with thermal diffusivities ranging from $1x10^{-7}$ to $70x10^{-7}$ m^2/s, in shape of rectangular parallelepipeds, and with different dimensions were employed in the experimental programme. An empirical equation relating minimum and maximum measuring times and the thermal diffusivity of the sample is also obtained.
Keywords: hot wire technique, numerical simulation model, minimum and maximum measuring time, thermal properties, refractories.

Resumo

O método do fio quente é considerado como uma técnica precisa na determinação da condutividade térmica de materiais cerâmicos. Todavia, especificamente para materiais de alta difusividade térmica, o intervalo de tempo apropriado a ser considerado nos cálculos é um fator decisivo na obtenção de resultados precisos e consistentes. Neste trabalho, um modelo de simulação numérica é proposto com o objetivo de se determinar os tempos mínimo e máximo de medida na técnica de fio quente paralelo. O perfil de temperatura gerado por este modelo está em excelente concordância com aquele determinado experimentalmente por esta técnica, onde a condutividade térmica, a difusividade térmica e o calor específico são simultaneamente determinados a partir do mesmo transiente experimental de temperatura. Dezoito amostras diferentes de refratários e polímeros, com difusividades térmicas variando de $1x10^{-7}$ a $70x10^{-7}$ m^2/s, na forma de paralelepípedos retangulares, e com diferentes dimensões foram utilizadas no programa experimental. Uma equação empírica relacionando os tempos mínimo e máximo de medida com a difusividade térmica da amostra é também obtida.
Palavras-chave: *técnica de fio quente, modelo de simulação numérica, tempos mínimo e máximo de medida, propriedades térmicas, refratários.*

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:

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

where: a = thermal diffusivity (m^2/s), k = thermal conductivity (W/m.K), ρ = bulk density (kg/m^3) and c_p = specific heat (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 conductivities of different materials. However, for refractory materials the hot wire technique is a suitable method and it was specifically developed for this kind of material. 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 basic 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 is employed in the experimental programme.

The hot wire parallel technique is an absolute, non-steady state and direct method, and therefore it makes the use of standards unnecessary. The hot wire method was 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 is considered an effective and accurate means of determining the thermal conductivity of ceramic materials. 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 [5]. 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, the thermal conductivity is calculated according to the following equation [6]:

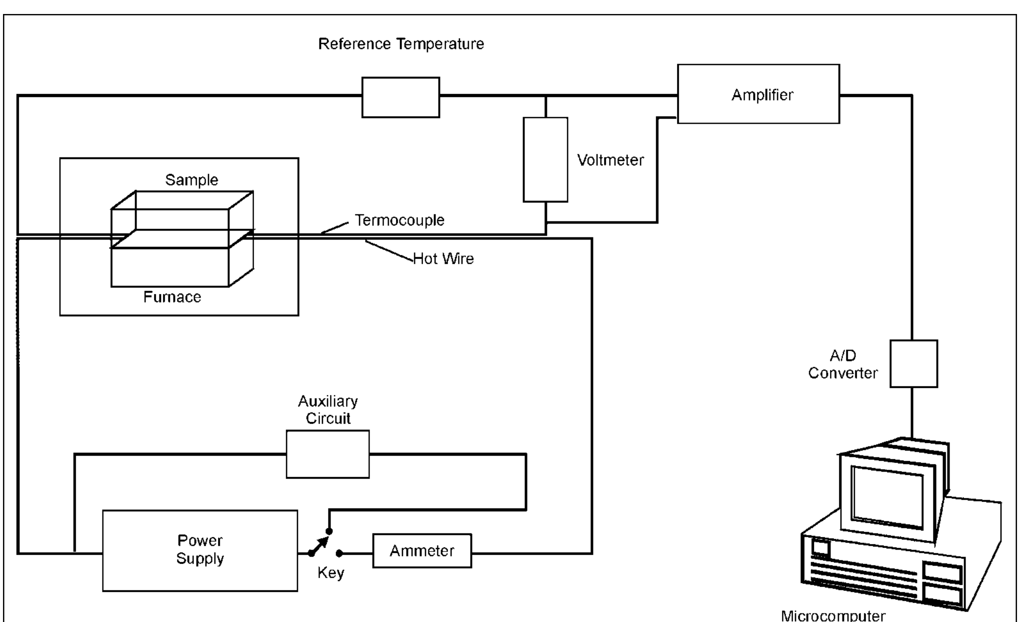

Figure 1: Schematic of hot wire parallel equipment.
[Figura 1: Diagrama esquemático da técnica de fio quente paralelo.]

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

where: k = thermal conductivity of the material (W/m.K), q' = linear power density (W/m), ρ = material bulk density (kg/m³), c_p = specific heat of the material (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.

The calculations, starting from the recorded temperature transient in the sample are carried out by using a non-linear least squares fitting method [7]. Both thermal conductivity and specific heat in equation B 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 calculated using equation A. So, using the same apparatus it is possible to determine these three thermal properties in the same experiment.

The schematic diagram of the apparatus used in this work, as shown in Fig. 1, 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.

r_i^*

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 [8, 9]. It is assumed that the solid is composed of **N**

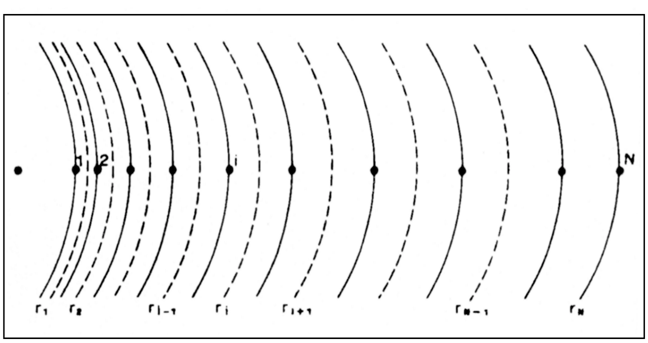

Figure 2: Annular cylindrical regions for the numerical analysis.
[Figura 2: Regiões cilíndricas anulares para análise numérica.]

concentric individual layers with radii r_i measured from the center of the specimen where the hot wire is embedded. Fig. 2 illustrates this procedure.

An energy balance of the form of equation C may be defined for each region:

$$[\text{Heat Flux in}]_i - [\text{Heat Flux out}]_i = [\text{Rate of Internal Energy Change}]_i \tag{C}$$

Then by applying balance equation C for each region, we derive equations D, E and F.

For i = 1:

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

For i = 2, 3,…, N-1:

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

For i = N:

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

where: <Ti> = average temperature of the region i between times t and (t+Δt), 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 equations G, H and I is introduced.

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

$$\tau = \frac{at}{L_{ref}} \tag{H}$$

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

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), = 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+Δt, one can write equations J, K and L.

$$<\theta_{i-1}> = \frac{\theta_{i-1}^{t+\Delta t} + \theta_{i-1}^{t}}{2} \quad \text{(J)},$$

$$<\theta_{i}> = \frac{\theta_{i}^{t+\Delta t} + \theta_{i}^{t}}{2} \quad \text{(K)},$$

$$<\theta_{i+1}> = \frac{\theta_{i+1}^{t+\Delta t} + \theta_{i+1}^{t}}{2} \quad \text{(L)}$$

Combining equations D to L, and after some algebraic simplifications, the final result may be expressed in a matrix form given by equation M.

$$B^{K}\theta^{K+1} = C^{K}\theta^{K} + D \quad \text{(M)}$$

Table I – Sample details.
[Tabela I - Detalhes sobre as amostras.]

Sample	**Material**	**Density (kg/m^3)**	**t_{min} (s)**	**t_{max} (s) for r_s=5cm***
A1	Silicon carbide	2614	12	81
A2	Fired magnesia	2931	11	88
A3	Alumina zircon	3428	17	140
A4	Alumina	2901	22	177
A5	Alumina	2904	31	225
A6	Alumina	2676	28	243
A7	Mulite	2363	30	246
A8	Mulite	2654	31	261
A9	Alumina	2466	43	400
A10	Vermiculite	734	42	400
A11	Calcium silicate	224	58	540
A12	Calcium silicate	224	84	765
A13	Insulating Portland concrete	332	88	792
A14	Insulating refractory concrete	1710	108	957
A15	Rigid PVC	1368	205	1650
A16	Acrylic	1186	225	1775
A17	Polypropylene	906	225	2286
A18	Nylon	1151	260	2499

* *r_s = distance between hot wire and edge of specimen.*

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+Δt.

The temperature profile generated by equation M 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.

EXPERIMENTAL PROGRAMME

Eighteen different specimens of refractory materials and polymers in the shape of rectangular parallelepipeds with different dimensions were employed in the experimental programme. Details of the samples are given in Table I.

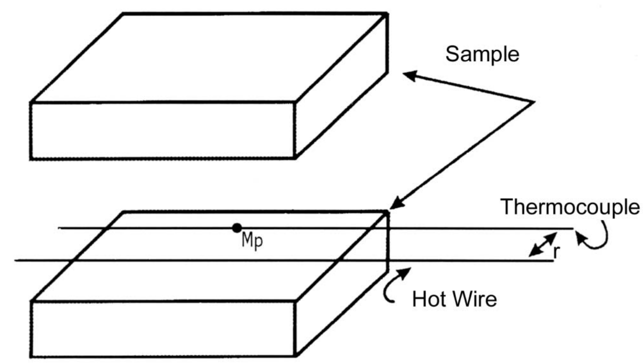

Figure 3: Parallel hot wire technique. Experimental set-up.
[Figura 3: Técnica de fio quente paralelo: arranjo dos corpos de prova.]

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. When calculations are carried out in this time interval, deviations among them are around 5% for all samples.

Specific procedures for sample arrangements were described in several occasions [1, 10, 11]. In this work, the measuring point Mp was kept at 16 mm from the hot wire. Fig. 3 shows the samples arrangement.

Table II – Experimental results for the appropriate measuring time interval.
[Tabela II – Resultados experimentais para os adequados intervalos de tempo de medida.]

Sample	K (W/m.K)	Cp (J/kg.K)	α ($x10^{-7}m^2/s$)	R*
A1	13.7431	777.2289	67.745	0.99955
A2	11.0601	605.8201	61.725	0.99897
A3	4.2704	455.1872	27.355	0.99958
A4	4.1034	669.3070	21.145	0.99960
A5	4.0369	693.7408	20.070	0.99937
A6	2.4580	667.9017	13.730	0.99835
A7	1.9688	674.5910	12.370	0.99994
A8	2.9872	766.5044	14.710	0.99964
A9	1.5463	731.4535	8.558	0.99983
A10	0.2524	613.3826	5.607	0.99997
A11	0.0656	963.9372	3.037	0.99995
A12	0.0416	991.5800	1.874	0.99995
A13	0.1317	1253.1149	3.165	0.99993
A14	0.4650	1226.0105	2.218	0.99975
A15	0.1962	1004.6938	1.426	0.99849
A16	0.2012	1411.9189	1.197	0.99932
A17	0.2331	1814.9901	1.417	0.99951
A18	0.2960	1786.6821	1.441	0.99798

** R = correlation coefficient.*

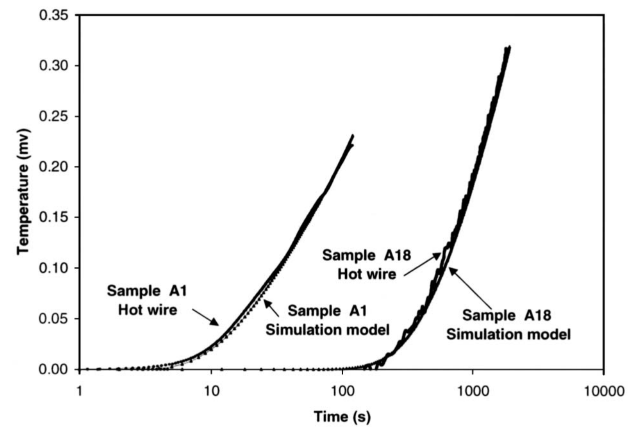

Figure 4: Temperature profile at measuring point.
[Figura 4: Perfil de temperatura no ponto de medida.]

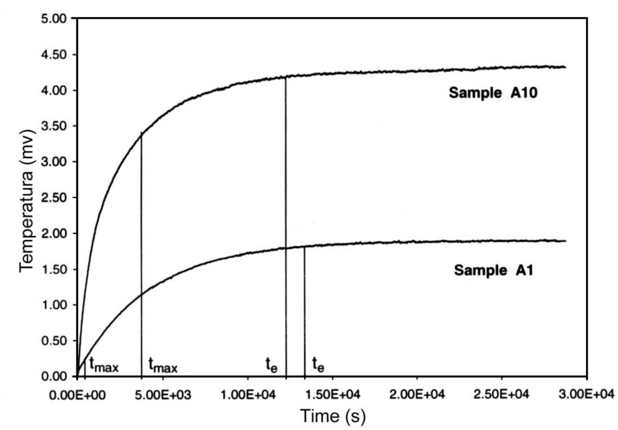

Figure 5: Temperature profile at measuring point: stable temperature after a long time.
[Figura 5: Perfil de temperatura no ponto de medida: temperatura estável após longo tempo.]

RESULTS AND DISCUSSION

Measurements were carried out using the apparatus described in Fig. 1, and Table II shows the experimental results obtained for all samples.

Fig. 4 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 the perfect agreement between the results predicted by the simulation model and those ones experimentally determined by the hot wire 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 (Fig. 3) 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. 5. The value of the time t_e depends on the samples thermal diffusivity, relation between the parallelepiped dimensions, and the environmental conditions.

In this work the criteria to determine minimum and maximum measuring times were the increase by half a degree centigrade at the measuring point and at the outer surface temperatures of the sample, in relation to the initial reference temperature. When calculations are carried out inside this time interval, the deviation in the thermal properties measurements is of approximately 5% for all samples. The correct determination of the minimum and maximum measuring times is a crucial and decisive task in the hot wire parallel

Table III – Experimental results for displaced time intervals.
[Tabela III – Resultados experimentais para os intervalos de tempo deslocados.]

Sample	Displaced Δt (minutes)	K (W/m.K)	δk (%)	c_p (J/kg.K)	δ c_p (%)	a (x 10^{-7}m²/s)	δa (%)
A1	1	9.7854	40.44	1107.3913	42.48	38.360	76.60
	2	9.7466	41.00	1151.5255	48.16	32.430	108.90
	5	3.4981	292.87	1918.5002	146.84	6.986	869.72
A2	1	12.0626	9.06	490.6859	23.46	83.900	35.93
	2	9.2291	19.84	712.0692	17.54	44.240	39.52
	5	6.3959	72.92	1089.0456	79.76	20.040	208.01
A4	1	4.5659	7.29	671.9928	2.57	23.430	10.05
	2	4.3351	1.87	690.5222	0.19	21.650	1.69
	5	3.8521	10.47	766.1334	11.16	17.340	22.78
A10	1	0.2520	0.16	613.3381	0.01	5.597	0.18
	2	0.2488	1.45	615.0868	0.28	5.511	1.74
	5	0.2441	3.40	619.8121	1.05	5.365	4.51
A16	1	0.2001	0.55	1408.7107	0.23	1.193	0.34
	2	0.1986	1.31	1405.0487	0.49	1.888	0.76
	5	0.2007	0.25	1409.3090	0.19	1.197	0.00

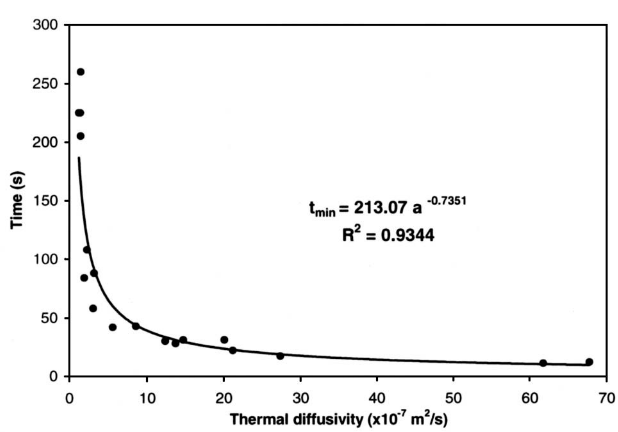

Figure 6: Minimum measuring time as a function of the thermal diffusivity.
[Figura 6: Tempo mínimo de medida em função da difusividade térmica.]

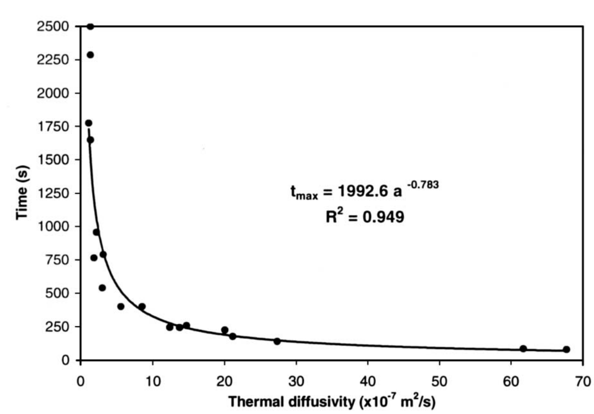

Figure 7: Maximum measuring time as a function of the thermal diffusivity.
[Figura 7: Tempo máximo de medida em função da difusividade térmica.]

calculations, mainly for materials with high thermal diffusivities, and the higher the thermal diffusivity to be measured, the more critical its influence on the thermal properties measurements. Table III shows calculations taking into account the appropriate time interval [t_{min}, t_{max}] as stated in this work, and the results obtained when this same interval is displaced by 1, 2 and 5 minutes ahead for selected samples. The deviation between those values increases as thermal diffusivity increases, showing the importance of these parameters for high thermal diffusivity materials. For sample A1 (silicon carbide brick) the displacement by only one minute in the measuring time interval is enough to introduce an error of 40% in the thermal conductivity, 42% in the specific heat and 77% in the thermal diffusivity.

Figs. 6 and 7 display minimum and maximum measuring times as a function of the thermal diffusivity. 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}.

CONCLUSIONS

A numerical simulation model is proposed to determine minimum and maximum measuring times in the hot wire parallel technique, with perfect agreement between the results predicted by this model and those ones obtained by the experimental method. 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 diffusivity increases. With this simulation model it is also possible to determine minimum sample dimensions for a previous measuring time interval stated.

The minimum and maximum measuring times were fitted to a power function and should be used for an initial estimate of t_{min} and t_{max} in the hot wire parallel technique.

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