T3 Thermal characterization of materials through the hot plate/hot wire techniques

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Abstract: This tutorial aims to describe two very well known methods that allow the identification of thermal properties from transient heat transfer experiments. The metrology is based on sensors that play both the roles of the heater and the thermometer. Analytical models for both experimental configurations are based on the formalism of integral transforms (Laplace, Hankel, Fourier) in the framework of quadrupoles method. Identification of the parameters is reaches using two approaches.

Nomenclature

r, x - spatial coordinates (radius, distance) [m]
e_0 - thickness of the probe [m]
e - thickness of the sample [m]
l - length of the cylindrical probe/sample [m]
S - surface of the heated area [m^2]
r_1 - radius of the probe [m]
R_1 - interior radius of the sample [m]
R_2 - outer radius of the sample [m]
t - time [s]
a - thermal diffusivity [$m^2 s^{-1}$]
λ - thermal conductivity [$W m^{-1} K^{-1}$]
c - specific heat [$J kg^{-1} K^{-1}$]
ρ - mass density [$kg m^{-3}$]
R_{tc} - thermal contact resistance [$W^{-1} m^2 K$]
T - temperature [K]
$\Theta(p)$ - Laplace temperature [K]
P - power [W]
p - Laplace variable [W]

3.1 Hot plane and hot wire: methods for thermal characterization

These methods are based on quasi-stationary heat transfer in the "infinite" sample. They are common in the industrial practice due to their simplicity and low cost. The different geometry of the sample and that of the probe leads to different thermal parameters.
This kind of metrology uses the contact probe composed of a heating resistance and a thermocouple for the measurement of the probe temperature variation.

3.2 The hot plane method

The hot plate method is based on simple principle and demands basic equipment. For these characteristics it is widely used in the industrial practice. This technique was proposed by P. Vermote in 1937 [1]. Schematic view of the experiment is represented in Figure 1.

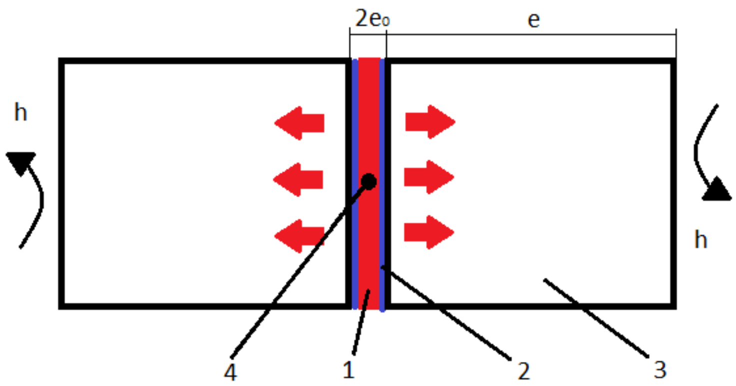

Figure 1: The hot plate measurement principle; 1 - the probe, 2 - thermal contact resistance between probe and sample, 3 - the sample, 4 - thermocouple, h - convective transfer coefficient.

The experimental device is composed of a plane probe of small thickness introduced between two pieces of solid sample leading to the symmetrical system. The probe is heated by the Joule effect using a constant power from an electric power supply. The temperature variation of the probe is measured at its centre by a thermocouple.
Knowing the temperature variation and the supplied power, it is thus possible to estimate the thermal properties of the material by comparing the data with a thermal model.
The thermal model is established assuming 1D heat transfer in the sample i. e. no heat loses on the lateral surfaces of the samples. According to the complexity of the model different cases can be considered. Three cases are addressed here.

3.2.1 Complete representation

This model takes into account the probe as a heating element of rectangular geometry. Its average temperature is considered. The thermal contact resistance between the probe and the sample is taken into account. The sample is considered as a wall with heat losses at the rear face. The representation using the thermal impedances in the Laplace domain leads to a model as shown in Figure 2 [2].
In this model, the probe is represented by the impedances Z_2 and Z_3, the sample is represented with impedances Z_4 , Z_5 and Z_6. The two resistances R_{tc} and 1/(hS) represent the thermal contact resistance and the heat losses at rear surface respectively.
The expressions of the impedances are given below [2]:

$$Z_2 = \frac{1}{\dfrac{\lambda S}{e_0} s \tanh(s_0)} - \frac{1}{\rho c S e_0 p},\; Z_3 = \frac{1}{\rho c S e_0 p},\; Z_4 = Z_5 = \frac{\cosh(s) - 1}{\lambda m S \sinh(s)},\; Z_6 = \frac{1}{\lambda m S \sinh(s)}$$

With: $s = e\sqrt{p/a}; s_0 = e_0\sqrt{p/a}; m = \sqrt{p/a}$

This complete model accounts for several unknown parameters. It is thus delicate to use it in practice. When the thickness of the sample is sufficient to consider it infinite for the duration of the experiment, this model can be simplified.

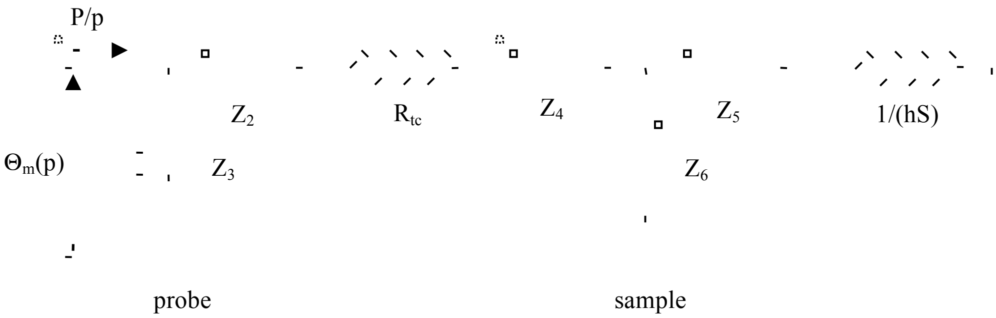

Figure 2: The complete model of hot plate experiment.

3.2.2 Simplified model

In this simplified model, the sample is considered as a half space. The model can be represented with thermal impedances as shown in Figure 3:

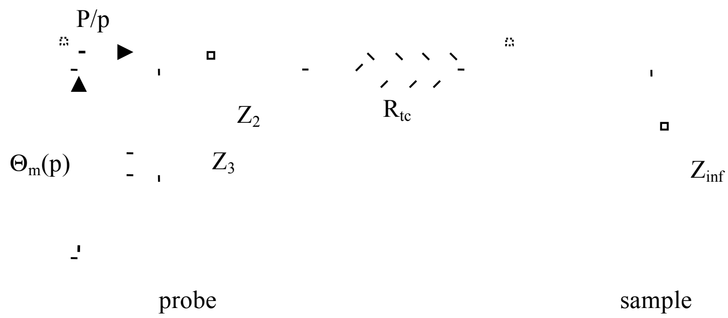

Figure 3: The simplified model of hot plate experiment.

If only the long times are considered the impedance Z_2 becomes:

$$Z_2 = \frac{e_0}{3\lambda S} \text{ and } Z_{inf} = \frac{1}{S\sqrt{p}\sqrt{\lambda \rho c}}$$

The impedance Z_2 corresponds thus to the thermal resistance of the probe.

3.2.3 Model of a half space media

This model takes into account only the heat transfer in the sample. The half space sample (for the duration of the experiment) is considered and the presence of the probe is neglected, one can say that the probe has neither thermal capacity nor thermal contact resistance with the sample. The model can be represented with thermal impedances as shown in Figure 4:

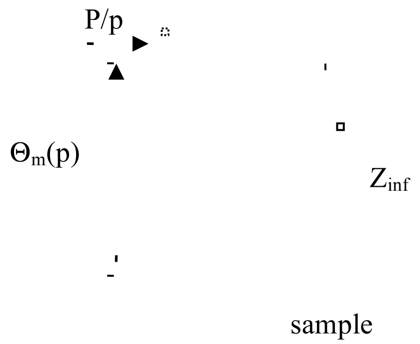

Figure 4: The impedance of plane half space.

The temperature on the half space sample at its surface is in time domain is [4] :

$$T(0,t) = \frac{2P}{S\sqrt{\pi}\sqrt{\lambda\rho c}}\sqrt{t} + T_{inf}$$

Measuring the power *P* dissipated by the probe and the temperature on the sample surface a simple linear regression permits obtaining directly the thermal effusivity of the studied sample. Of course a minimization algorithm (e. g. Levenberg-Marquardt) can also be used to identify the effusivity on temporal thermogramme.

3.3 Hot wire method

The hot wire method as the hot plane is based on simple principle and demands basic equipment. This method was introduced at the beginning of the 30'ies of the precedent century by B. Stahlane et S. Pyk [3] for measurements of granular media. Nowadays it is used for solids and liquids also.
The experimental device is composed of a metallic wire immersed in liquid or introduced between two pieces of solid sample, depending of the studied media (Figure 5). Using an electric power supply, the wire is supplied with a constant electric potential. Due to the Joule effect, the wire is heating and the temperature evolution is measured at its centre by a thermocouple.

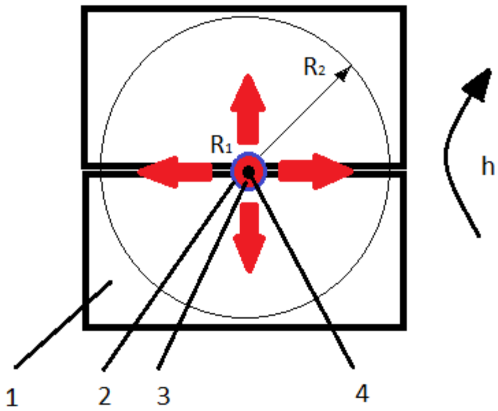

Figure 5: The hot wire method principle. 1 - the sample, 2 - thermal contact resistance between probe and sample, 3 - the probe, 4 - thermocouple, h - convective transfer coefficient.

As for the hot plane, three cases of modeling for this experiment can be proposed.

3.3.1 Complete representation

This model takes into account the probe as a heating element of cylindrical geometry. Its average temperature is considered. The thermal contact resistance between the probe and the sample is considered. The sample is considered as a cylinder with heat losses at the out face. The representation using the thermal impedances in the Laplace domain leads to a model as shown in Figure 2. Only the expressions of the impedances change. The expressions of the impedances for the hot wire experiment are [2]:

$$Z_2 = \frac{1}{2\pi\lambda l}\frac{I_0(s)}{sI_0(s)} - \frac{1}{\rho c\pi S r_1{}^2 lp};\ Z_3 = \frac{1}{\rho c\pi S r_1{}^2 lp};\ Z_4 = \frac{\alpha_2[I_0(\alpha_1)K_1(\alpha_2)+I_1(\alpha_2)K_0(\alpha_1)]-1}{2\pi\lambda l\,\alpha_1\alpha_2[I_1(\alpha_2)K_1(\alpha_1)+I_1(\alpha_2)K_1(\alpha_2)]};$$

$$Z_5 = \frac{\alpha_1[I_0(\alpha_2)K_1(\alpha_1)+I_1(\alpha_1)K_0(\alpha_2)]-1}{2\pi\lambda l\,\alpha_1\alpha_2[I_1(\alpha_2)K_1(\alpha_1)+I_1(\alpha_2)K_1(\alpha_2)]};\ Z_6 = \frac{1}{2\pi\lambda l\,\alpha_1\alpha_2[I_1(\alpha_2)K_1(\alpha_1)+I_1(\alpha_2)K_1(\alpha_2)]}$$

with:

$$s = r_1\sqrt{p/a};\ m = \sqrt{p/a};\ \alpha_1 = R_1\sqrt{p/a};\ \alpha_2 = R_2\sqrt{p/a};$$

This complete model accounts for several unknown parameters. It is thus delicate to use it in practice. When the radius of the sample is sufficient to consider it infinite for the duration of the experiment, this model can be simplified.

3.3.2 Simplified model

In this simplified model, the sample is considered as an infinite media. The model can be represented with thermal impedances as shown in Figure 3 for the hot plane. If only the long times are considered the impedance Z_2 becomes:

$$Z_2 = \frac{1}{8\pi\lambda l} \text{ and } Z_{inf} = \frac{1}{2\pi kl} - \frac{K_0\left(r_1\sqrt{p/a}\right)}{r_1\sqrt{p/a}\,K_0\left(r_1\sqrt{p/a}\right)}$$

The impedance Z_2 corresponds thus to the thermal resistance of the probe.

3.3.3 Model of an infinite media

This model takes into account only the heat transfer in the sample. The infinite sample (for the duration of the experiment) is considered and the presence of the probe is neglected, one can say that the probe has no thermal capacity nor thermal contact resistance between probe and sample. The model can be represented with thermal impedance of infinite media as shown in Figure 4.
The temperature of the infinite sample at the radius r_0=0 is [4]:

$$T(0,t) = \frac{P}{4\pi kl}\ln(t)$$

Measuring the power P dissipated by the probe and its temperature a simple linear regression permits obtaining directly the thermal conductivity of the studied sample. Of course a minimization algorithm (e. g. Levenberg-Marquardt) can also be used to identify the thermal conductivity on temporal thermogramme.

3.4 Example of application: hot plane method

The model of a half space is used to simulate a thermogramme of a hot plane measurement. Figure 6 represents the temperature variation in time and square root of time. The simulated temperature was slightly noised to simulate the measurement error.

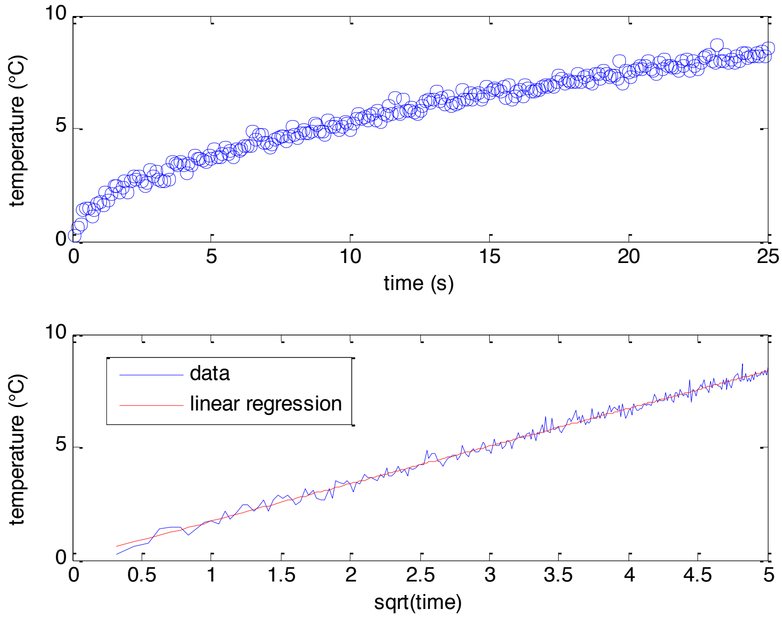

Figure 6: Simulated thermogramme of hot plane measurement. The properties used for simulation are given in Matlab code, see Appendix.

The slope of the straight line from the linear regression leads to the thermal effusivity of the material 676.2 ± 6.2 [J $s^{-1/2}$ m^2 K]. The coefficient of determination of the linear regression is 0.99.
The Levenberg-Marquardt optimization (Figure 7) on the same data gives effusivity equal 671.3 ± 2.0 [J $s^{-1/2}$ m^2 K]. The difference in precision of estimation of the two methods comes from the variable time step in the case of square root of time plot.

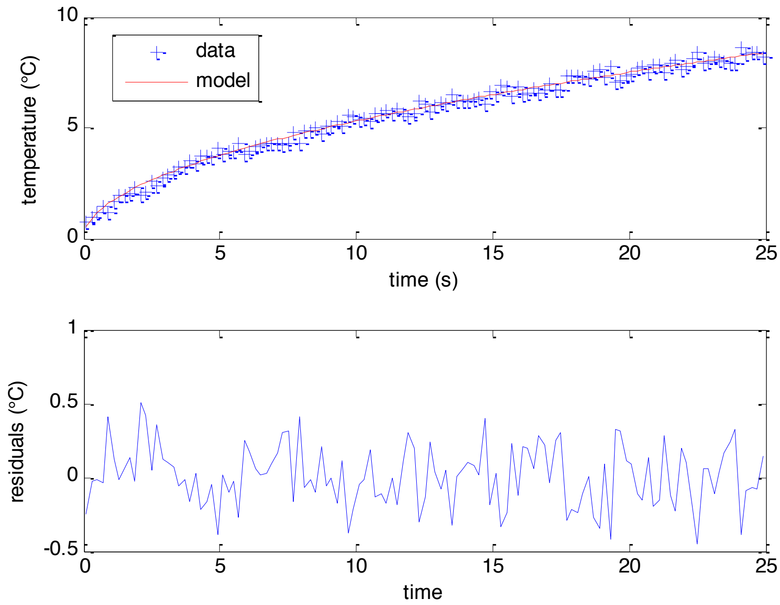

Figure 7: The Levenberg-Marquardt optimization on simulated hot plate measurement.

The same properties of the sample are now used in the simplified model. The properties of the probe are given in the Matlab code. Figure 8 represents the simulated temperature from the simplified model with that computed from the half space model. It can be clearly seen the influence of the probe. Its capacity makes that the linear evolution of temperature occurs not directly after the switching of power. The resistance due to the conduction in the probe leads to slightly lower values of temperature.

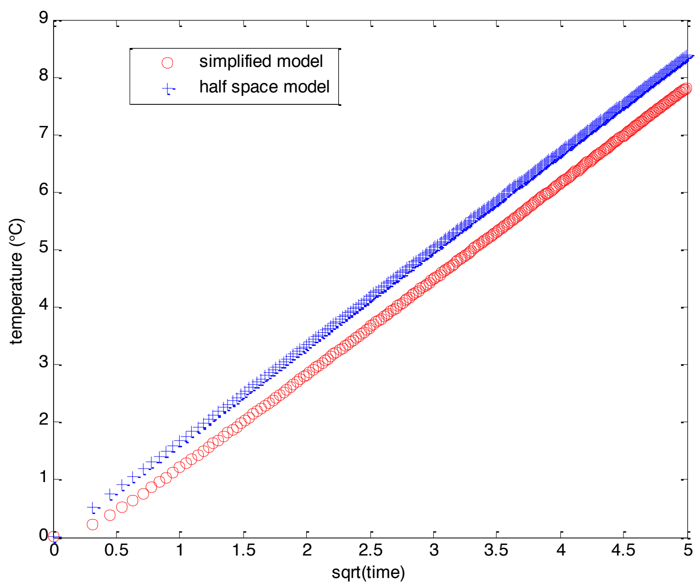

Figure 8: Simulated temperatures from half space model and simplified model.

Finally the Levenberg-Marquardt optimization is carried out on the noised temperature from simplified model (Figure 9). The estimated effusivity is 673.8 ± 1.7 [J s$^{-1/2}$ m^2 K].

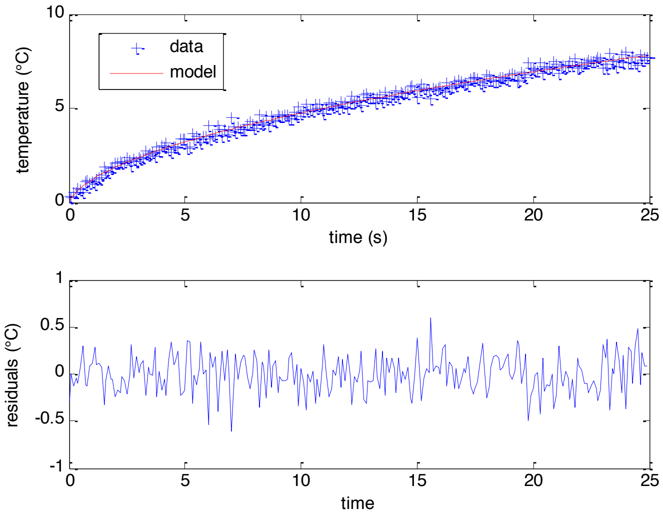

Figure 9: The Levenberg-Marquardt optimization on simulated hot plate measurement: simplified model.

The exact value of effusivity is 671.9 [J s$^{-1/2}$ m^2 K]. The confidence intervals given for the estimations are 67%. Even with this narrow confidence interval, the estimations meet the theoretical value of effusivity. Naturally, the data for estimations were simulated from models and only noised with low random noise.

3.5 Appendix - Matlab codes for hot plane simulations

Temperature of half space and linear regression

```
clear all; close all;
s = 0.1;
time = [.1:s:25];                % domaine de variation temporel
q = 1;                          % densité de flux déposé
lambda = 0.3;                      % conductivité thermique du matériau
rho = 700;                         % masse volumique thermique du matériau
cp = 2150;                         % chaleur spécifique du matériau
S = 1e-3;
effu = sqrt(lambda*rho*cp);
% milieu semi infini plan flux imposé - température en surface
T = q*2*sqrt(time)/sqrt(pi)/effu/S;
Texp = T+randn(size(time))/5;
figure(1),subplot(2,1,1),plot(time,Texp,'o'),xlabel('time (s)'),
ylabel('temperature (°C)'); hold on;
figure(1),subplot(2,1,2),plot(sqrt(time),Texp),xlabel('sqrt(time)'),
ylabel('temperature (°C)');hold on;

% routine which permits to do linear regression on x,y (sqrt(time) and Texp)
data with estimation
% of parameter of the function and theirs standard deviations
```

```
X=sqrt(time)';
X=X';
Y=Texp';
Y=Y';

% function [b0,b1,r2,svr]=regfit(Y,X)
m = length(X);
Ym = Y';
Xm = ones(m,2);
Xm(:,2) = X;
b = (inv(Xm'*Xm)*Xm')*Ym;
b0 = b(1);
b1 = b(2);
Ye = b1*X+b0;
ec = (Y-Ye).^2;
ssr = sum(ec);
sst = sum((Y-mean(Y)).^2);
sse = sum((mean(Y)-Ye).^2);
vr = ssr/(m-2);
svr = sqrt(vr);
V = vr*inv(Xm'*Xm); % matrix of variance covariance of estimated parameters
sb0 = sqrt(V(1,1)); % standart deviations of estimated parameters
sb1 = sqrt(V(2,2));
corr = V(1,2)/(sb0*sb1);
r2 = sse/sst; % coefficient of determination
figure(1),subplot(2,1,2),plot(sqrt(time),(b1*X+b0),'r-')
```

Temperature of half space and Levenberg Marquardt optimization

```
clear all; close all;
s=0.2;                        % time step
time=[.1:s:25];               % time vector

q=1;                          % heat flux density
lambda=0.3;                     % thermal conductivity of the sample
rho=700;                        % density of the samplemasse volumique
thermique du matériau
cp=2150;                        % specific heat of the sample
effu = sqrt(lambda*rho*cp);     % effusivity of the sample
S=1e-3;                         % surface of the sample
% temperature of hals space with heat flux on its surface
Tth=q*2*sqrt(time)/sqrt(pi)/effu/S;
Texp=Tth+randn(size(time))/5;   % some random noise addition

effuini=sqrt(lambda/12*rho*cp); % initial value for effusivity
inco = [effuini];
options=optimset('Display','iter','LevenbergMarquardt','on');
[inco,resnorm,residual,exitflag,OUTPUT,LAMBDA,Jacobian] =
LSQNONLIN(@minimization_function,...
    inco,[],[],options,S,q,time,Texp);
effuest = inco(1);
[T]=model_plan_chaud(effuest,S,q,time);
%Estimated parameter variance
Jacobian = full(Jacobian);
varp = resnorm*inv(Jacobian'*Jacobian)/length(time);
stdp = sqrt(diag(varp)); %standard deviation is square root of variance
```

```
figure(2),subplot(2,1,1),plot(time,Texp,'b+',time,T,'r-'),xlabel('time (s)'),
ylabel('temperature (°C)'); hold on;
figure(2),subplot(2,1,2),plot(time,residual),xlabel('time'), ylabel('residuals
(°C)');
```

Minimization function

```
function f = minimization_function(inco,S,q,time,Texp);

effu = inco;
[T]=model_plan_chaud(effu,S,q,time);
f = zeros(size(time))';
f = (T-Texp);
```

Half space model

```
function [T]=model_plan_chaud(effu,S,q,time);

% milieu semi infini plan flux imposé - température en surface
T=q*2*sqrt(time)/sqrt(pi)/effu/S;
```

Temperature from simplified model and Levenberg Marquardt optimization

```
clear all; close all;
s = 0.1;
time = [.000001:s:25];                  % time, duration of experiment
q = 1;                                  % power density
lambda = 0.3;                                % thermal conductivity of sample
material
rh = 700;                              % density of sample material
cp = 2150;                              % specific heat of sample
effu = sqrt(lambda*rh*cp);                 % effusivity of sample

Cp = 1000;            % specific heat of probe
e0 = 0.001;           % half thickness of probe
lambdap = 5;                  % thermal conductivity of probe
rho = 300;              % specific heat of the probe
S = 1e-3;                % sample/probe area
Rtc = 1e-4;              % thermal contact resistance between the probe and
the sample
[T] = model_complet_hotplate(lambdap,e0,S,effu,rho,Cp,Rtc,q,time); % computing
theoretical temperature
Texp = T+randn(size(time))/5; % some random noise
[T1]=model_plan_chaud(effu,S,q,time);
figure(1),plot(sqrt(time),T,'ro',sqrt(time),T1,'b+'),xlabel('sqrt(time)'),
ylabel('temperature (°C)');

% intial values for effusivity
effuini = sqrt(lambda/12*rh*cp);
inco = [effuini];
options=optimset('Display','iter','LevenbergMarquardt','on');
[inco,resnorm,residual,exitflag,OUTPUT,LAMBDA,Jacobian] =
LSQNONLIN(@minimization_function_hotplate,...
    inco,[],[],options,lambdap,e0,S,rho,Cp,Rtc,q,time,Texp); %
LevenbergMarquardt optimization

effuest = inco(1);
%Estimated parameter variance
Jacobian = full(Jacobian);
varp = resnorm*inv(Jacobian'*Jacobian)/length(time);
stdp = sqrt(diag(varp));   %standard deviation is square root of variance
```

```
[T]=model_complet_hotplate(lambdap,e0,S,effuest,rho,Cp,Rtc,q,time);
figure(2),subplot(2,1,1),plot(time,Texp,'b+',time,T,'r-'),xlabel('time (s)'),
ylabel('temperature (°C)'); hold on;
figure(2),subplot(2,1,2),plot(time,residual),xlabel('time'), ylabel('residuals
(°C)');
```

Minimization function

```
function f =
minimization_function_hotplate(inco,lambdap,e0,S,rho,Cp,Rtc,q,time,Texp)

effu = inco(1);
 [T]=model_complet_hotplate(lambdap,e0,S,effu,rho,Cp,Rtc,q,time);
f = zeros(size(time))';
f = (T-Texp);
```

Simplifed model

```
function [T]=model_complet_hotplate(lambdap,e0,S,effu,rho,cp,Rtc,q,time)

%
% lambda                              % thermal conductivity
% alpha                               % thermal diffusivity
% e0                                   % probe half thickness

N=10;                                 % Stehfest variable
v=[1/12 -385/12 1279 -46871/3 505465/6 -473915/2 1127735/3 -1020215/3 328125/2
-65625/2];
A=zeros(size(time));
for j=1:N;
    s=j*log(2)./time;
    Z3=1./(rho*cp*S*e0*s);
    Z2=e0/3/lambdap/S;
    Zinf=1./(effu*sqrt(s)*S);
    Z=1./(1./Z3+1./(Z2+Rtc+Zinf));
    Z=Z./s*q;
    A=A+v(j)*Z;
end;
T=A*log(2)./time;       % Laplace inverse
```

3.6 References

[1] P. Vermote, C.R. Acd., Paris, Vol 204, P 563,1937
[2] D. Maillet, S. André, J.-C. Batsale, A. Degiovanni, C. Moyne, Thermal Quadrupoles: Solving the Heat Equation through Integral Transforms ISBN: 978-0-471-98320-0
[3] B. Stalahne and S. Pyk, Teknish Tidskrift, Vol 61, pp 389-393, 1931.
[4] J. Taler, P. Duda, Solving Direct And Inverse Heat Conduction Problems, Springer-Verlag, ISBN-13: 978-0312195762