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► To cite this version:

Vincent Delobelle, Denis Favier, Hervé Louche, Nathanael Connesson. Determination of Local Thermophysical Properties and Heat of Transition from Thermal Fields Measurement During Drop Calorimetric Experiment. Experimental Mechanics, 2015, 55 (4), pp.711-723. 10.1007/s11340-014-9877-z . hal-01300519

HAL Id: hal-01300519 https://hal.science/hal-01300519

Submitted on 11 Apr 2016 $\,$

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Determination of local thermophysical properties and heat of transition from thermal fields measurement during drop calorimetric experiment

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Abstract

This paper proposes a non-contact original method to estimate local thermophysical properties (heat capacity and thermal conductivity) and heats of transition from plane thin specimens. This method is based on measurement of temperature fields with an infrared camera during a drop calorimetric experiment. A studied specimen and a reference specimen, with similar geometries, are simultaneously tested. Firstly, the method is validated by estimating heat capacity and thermal conductivity of Vanadium specimens and by comparing the determined values with those obtained by Differential Scanning Calorimetry and by a laser flash method, respectively. Secondly, the method is used to determine latent heats of martensitic transformations. These heats of transition are determined during homogeneous and heterogeneous drop calorimetric experiments of NiTi shape memory alloys specimens. Measured transformation temperatures and latent heats are in good accor-

Preprint submitted to Experimental Mechanics

January 27, 2014

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dance with results obtained by Differential Scanning Calorimetry. *Key words:* Infrared thermography, heat capacity, thermal conductivity, heat sources estimation, experimental benchmark, NiTi Shape Memory Alloy

1 1. Introduction

Knowledge of heat capacity and thermal conductivity of materials is of 2 crucial importance to model heat exchanges in materials. Heat capacity of 3 materials can be measured with adiabatic calorimeter, Differential Scanning 4 Calorimetry (DSC) [1], modulated DSC [2, 3], dynamic DSC [4], etc. Ma-5 terials thermal conductivity is generally measured using a hot wire method 6 [5], the derived hot strip method, a laser flash method [6] or a 3ω method 7 [7]. Heat of transition is generally measured by DSC [8] or Differential Ther-8 mal Analysis. All these classical techniques allow a global measurement of g the desired property at the specimen scale. However, in some cases, a local 10 measurement of these properties would be useful, for example in the case 11 of heterogeneous specimens [9] or graded material [10, 11]. The method 12 proposed in this paper allows to locally estimate heat capacity, thermal con-13 ductivity and heat of transition from a thin plate specimen. The method has 14 been validated in this paper for homogeneous specimen although it could 15 also be applied to heterogeneous specimen. In such a case, a field of heat 16 capacity, thermal conductivity and heat of transition could be determined. 17

Infrared measurements are increasingly used. In order to estimate tem perature fields on the specimens surface, emissivity property is classically
 controlled using high emissivity paint on the specimen surface. Heat sources

estimations based on these temperature fields were proposed in [12, 13].
These estimations were used to study several mechanical coupled problems
such as Lüders bands and necking in steels [12, 14], fatigue of materials
[15, 16, 17, 18], plasticity in Al olygocrystal [19], thermomechanical behaviour
of NiTi Shape Memory Alloys (SMA) [13, 20, 21, 22, 23], etc.

In this paper, an original method, called Thermal Field Measurement (TFM) method, is proposed. This non-contact method allows to estimate thermophysical properties of material and heat of transition locally while classical methods are global. This method is based on the observation with an infrared camera of the natural cooling of two specimens (one being the 'reference' while the other is 'studied').

In the first section, the TFM models and method are presented. Heat diffusion models, method principle, and the methods to estimate heat capacity, thermal conductivity and heat of transition are successively proposed.

The second section is an experimental validation. First, experimental 35 setup, materials and data processing are presented. Then, heat capacity 36 of Vanadium C_{Va} was estimated using TFM method and DSC. Results are 37 successfully compared. Then, Vanadium thermal conductivity k_{Va} was esti-38 mated using TFM method. Results are compared to those obtained with the 39 laser flash method. Finally, heat of transition of NiTi SMAs was estimated 40 in a homogeneous and heterogeneous case with the TFM method. Results 41 are successfully compared to those obtained by DSC, which is the standard 42 method to determine characteristic temperatures and heat of the martensitic 43 transformations occurring in this material [24].

⁴⁵ 2. Thermal Field Measurement Models and Methods

46 2.1. Heat diffusion models

In this part, the heat diffusion models used in the following are presented. The general 3D heat diffusion equation linking temperature T(x, y, z, t) and heat sources s(x, y, z, t) at a spatial point located in (x, y, z) at current time t, is expressed in the following form:

$$\rho C \frac{\partial T}{\partial t} - k \, lap(T) = s = \rho \dot{q},\tag{1}$$

⁵¹ where ρ is the mass density, C the heat capacity, k the thermal conduc-⁵² tivity of the material and *lap* stands for the laplacian operator. In the right ⁵³ hand side of this equation, s is the volumic heat sources ($W m^{-3}$) and $\dot{q} = \frac{s}{\rho}$ ⁵⁴ represents the massic heat sources in the material ($W kg^{-1}$).

Figure 1 shows the specimens used in the present paper. The two sides of the sample (1) of thickness e_1 are coated with a high emissivity paint (p) of thickness e_P . The properties of the material or paint are the mass density ρ_i , thermal conductivity k_i , heat capacity C_i and volumic heat sources s_i where *i* indexes the material (*i* = 1) or the paint (*i* = *p*). The two paint layers are supposed identical (properties and thickness).

In order to take into account the two paint layers presented in Fig. 1, the plate is considered as a sandwich material. Mathematical developments to write models presented below are fully explained in [12, 13] in the case of an homogeneous plate in the thickness. Identical development can be done in the case of a sandwich material, as explained in [33, 35].

⁶⁶ A first model is obtained for thin plane sandwich specimen. Integrating ⁶⁷ equation 1 in the specimen thickness (z in Figure 1) provides the following 68 2D model:

$$\frac{2e_p\rho_pC_p + e_1\rho_1C_1}{e_1}\frac{\partial \widetilde{T}}{\partial t} - \frac{2e_pk_p + e_1k_1}{e_1}lap_{2D}(\widetilde{T}) + \frac{f}{e_1} = \widetilde{s_1}, \qquad (2)$$

where $\tilde{T} = \tilde{T}(x, y, t)$ is the averaged temperature in the thickness of the specimen and $lap_{2D}(\tilde{T}) = \frac{\partial^2 \tilde{T}}{\partial x^2} + \frac{\partial^2 \tilde{T}}{\partial y^2}$. In equation 2, $\tilde{s_1}$ represents the volumic heat sources $(W \ m^{-3})$ in the specimen. Heat sources in the paint are supposed null $(s_p=0)$. The function f models heat losses from the lateral surfaces by radiation f_{rad} and convection f_{conv} , leading to the following expression:

$$f(x, y, \widetilde{T}) = \underbrace{2\epsilon\sigma(\widetilde{T^4} - T_0^4)}_{f_{rad}} + \underbrace{2h(\widetilde{T} - T_0)}_{f_{conv}},$$
(3)

⁷⁴ where T_0 is the ambient temperature, ϵ the paint layer emissivity, σ the ⁷⁵ Stephan-Boltzman constant, and h is the convection coefficient. As shown ⁷⁶ in equation 3, this function $f(x, y, \tilde{T})$ is independent on the material. A ⁷⁷ second model can be obtained to study the case of uniaxial heterogeneous ⁷⁸ thermal field, for exemple for slender thin plane specimen. Assuming a quasi-⁷⁹ homogeneous field in the transverse direction, equation 2 can be integrated in ⁸⁰ the transverse direction (y in Figure 1) which allows to write the 1D model:

$$\frac{2e_p\rho_pC_p + e_1\rho_1C_1}{e_1}\frac{\partial\widetilde{T}}{\partial t} - \frac{2e_pk_p + e_1k_1}{e_1}lap_{1D}(\widetilde{\widetilde{T}}) + \frac{f}{e_1} = \widetilde{\widetilde{s}_1},\tag{4}$$

where $\tilde{\widetilde{T}} = \tilde{\widetilde{T}}(x,t)$ is the average of \widetilde{T} in the transverse direction of the specimen and $lap_{1D}(\tilde{\widetilde{T}}) = \frac{\partial^2 \tilde{\widetilde{T}}}{\partial x^2}$. In that case the heat losses function is written:

$$f(x,\widetilde{\widetilde{T}}) = \underbrace{2\epsilon\sigma(\widetilde{\widetilde{T}^4} - T_0^4)}_{f_{rad}} + \underbrace{2h(\widetilde{\widetilde{T}} - T_0)}_{f_{conv}},\tag{5}$$

⁸³ Finally, a third model can be obtained in the case of uniform temperature.

⁸⁴ Integrating equation 4 in the axial direction provides the 0D model:

$$\frac{2e_p\rho_pC_p + e_1\rho_1C_1}{e_1}\frac{d\widetilde{\widetilde{T}}}{dt} + \frac{f}{e_1} = \widetilde{\widetilde{\widetilde{s}_1}},\tag{6}$$

where $\tilde{\widetilde{T}} = \tilde{\widetilde{T}}(t)$ is the temperature average of T(x, y, z, t). The heat losses function is written:

$$f(\widetilde{\widetilde{T}}) = \underbrace{2\epsilon\sigma(\widetilde{\widetilde{T}^{4}} - T_{0}^{4})}_{f_{rad}} + \underbrace{2h(\widetilde{\widetilde{T}} - T_{0})}_{f_{conv}}.$$
(7)

These 2D, 1D and 0D model will be used to analyze the temperature fields variation versus time during different drop calorimetric experiments.

⁸⁹ 2.2. TFM Method principle

All the experiments are based on the observation of the simultaneous natural cooling in the same environment of two specimens, referred to as 'studied' (s) and 'reference' (r) specimen (Figure 2). The thermal capacity and conductivity of the 'reference' specimen are known. Furthermore, no transformation within the 'reference' specimen occurs in the temperature range $(\tilde{s}_1 = \tilde{\tilde{s}}_1 = \tilde{\tilde{s}}_1 = 0)$.

During the experiments, the 'studied' and 'reference' specimens are located in the same thermal environment. The two specimens are painted with an identical high emissivity paint ($\epsilon_r = \epsilon_s = \epsilon$). Thus:

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The radiation part f_{rad} of the heat losses function f is identical for both specimens and is only function of the temperature (equations (3), (5) and (7)).

• The second part of the heat losses function f_{conv} is due to convection. The heat transfer coefficient h is dependent on the heat transfer mode, the flow regime, etc: the heat transfer coefficient h may depend on the local convection mode:

- First, in the homogeneous case (equation (7)), h is only function of $\tilde{\widetilde{T}}$, i.e. $h(\tilde{\widetilde{T}})$.
 - Second, in the 1D case (equation (5)), h is only function of $\widetilde{\widetilde{T}}$ and x, i.e. $h(\widetilde{\widetilde{T}}, x)$
 - i.e. $h(\widetilde{\widetilde{T}}, x)$.
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• Last in the 2D case (equation (3)), h is function of \widetilde{T} , x, and y, i.e. $h(\widetilde{T}, x, y)$.

The 'studied' and 'reference' specimens being in the same environment, the heat losses functions (3), (5) and (7) are assumed identical for the two specimens.

For the 'reference' specimen, heat sources are zero during all the experiments. Thus this specimen is used to estimate the local heat losses function ffrom equations (2), (4) and (6) applied to the 'reference' thermal fields measured during the drop calorimetric experiment. Then, the local heat losses function f is used in different configurations to estimate the local heat capacity (section 2.3), thermal conductivity (section 2.4) and heat of transition (section 2.5) of the 'studied' specimen.

122 2.3. Estimation of heat capacity C for homogeneous samples

For homogeneous sample, estimation of heat capacity C is performed while observing simultaneous uniform temperature cooling of the studied and reference specimens (Figure 2.a). During the experiment, the specimen surface temperature fields T_s and T_r of 'studied' and 'reference' specimens, respectively, are measured and the temperature field homogeneities are checked experimentally. The materials and experimental temperature range are chosen so that no phase change occurs in the materials during the experiment; heat sources *s* are zero. Thus, the 0D thermal model (equation (6)) applied to each of the two specimens provides the equations:

$$(\rho_s C_s e_s + 2\rho_p C_p e_p) \frac{d\widetilde{\widetilde{T}}_s}{dt} = -f(\widetilde{\widetilde{T}}_s),$$
(8)

$$(\rho_r C_r e_r + 2\rho_p C_p e_p) \frac{d\widetilde{\widetilde{T}}_r}{dt} = -f(\widetilde{\widetilde{T}}_r).$$
(9)

The reference thermophysical properties being known and $\tilde{\tilde{T}}_{r}$ being measured, $f(\tilde{\tilde{T}})$ can be computed using equation (9) and the estimation of $\frac{\partial \tilde{\tilde{T}}_{r}}{\partial t}$ from experimental cooling curve of the reference specimen. Furthermore, when $\tilde{\tilde{T}}_{s} = \tilde{\tilde{T}}_{r} = \tilde{\tilde{T}}$, the heat losses function $f(\tilde{\tilde{T}})$ is identical

for both 'reference' and 'studied' specimens and the studied specimen heat rate capacity C_s can be computed by:

$$C_s(\widetilde{\widetilde{T}}_s) = \frac{f(\widetilde{\widetilde{T}}_s) - 2\rho_p C_p e_p \frac{d\widetilde{\widetilde{T}}_s}{dt}}{\rho_s e_s \frac{d\widetilde{\widetilde{T}}_s}{dt}}.$$
(10)

All the thermo-physical properties in the right hand side of equation 10 are known. The term $\frac{d\widetilde{\widetilde{T}_s}}{dt}$ is estimated at temperature $\widetilde{\widetilde{T}_s}$ from the experimental cooling curve of the studied specimen.

141 2.4. Estimation of thermal conductivity k

Using the experimental setup proposed in Figure 2.b, axially heterogeneous (1D) temperature fields are obtained during cooling and are used to estimate the thermal conductivity k. Once again for that experiment, the material and temperature range are chosen so that no transition occurs during the experiment; so heat sources are zero. Applying equation (4) to each of the two specimens, and considering hypotheses previously described, it can be written:

$$(\rho_s C_s e_s + 2\rho_p C_p e_p) \frac{\partial \widetilde{\widetilde{T}}_s}{\partial t} - (k_s e_s + 2k_p e_p) \frac{\partial^2 \widetilde{\widetilde{T}}_s}{\partial x^2} = -f(x_s, \widetilde{\widetilde{T}}_s), \qquad (11)$$

$$(\rho_r C_r e_r + 2\rho_p C_p e_p) \frac{\partial \widetilde{\widetilde{T}_r}}{\partial t} - (k_r e_r + 2k_p e_p) \frac{\partial^2 \widetilde{\widetilde{T}_r}}{\partial x^2} = -f(x_r, \widetilde{\widetilde{T}_r}).$$
(12)

In these equations, only f and k_s are unknown. $f(x, \tilde{T})$ can be estimated thanks to equation 12 and the experimental data for the reference specimen. From these data, the terms $\frac{\partial \tilde{T}_r}{\partial t}$ and $\frac{\partial^2 \tilde{T}_r}{\partial x^2}$ are estimated for every position xand temperature T. At the position $x_s = x_r = x$ from the mass, and at the temperature $\tilde{T}_s = \tilde{T}_r = \tilde{T}$, the studied specimen thermal conductivity k_s can thus be determined:

$$k_s(x_s, \widetilde{\widetilde{T}}_s) = \frac{f(x_s, \widetilde{\widetilde{T}}_s) + (\rho_s C_s e_s + 2\rho_p C_p e_p) \frac{\partial \widetilde{\widetilde{T}}_s}{\partial t} - 2k_p e_p \frac{\partial^2 \widetilde{\widetilde{T}}_s}{\partial x^2}}{e_s \frac{\partial^2 \widetilde{\widetilde{T}}_s}{\partial x^2}}.$$
 (13)

The term $\frac{\partial \widetilde{T_s}}{\partial t}$ is estimated for every position from the 'studied' specimen cooling curves and the term $\frac{\partial^2 \widetilde{T_s}}{\partial x^2}$ is estimated for every temperature from the 'studied' specimen thermal profiles. All the thermo-physical properties
in the right hand side of equation 13 are known.

159 2.5. Estimation of local heat of transition

The reference and studied materials and temperature range are chosen in
 this experiment so that:

• no phase change occurs in the 'reference' specimen,

• phase change occurs in the 'studied' specimen.

Heat of transition is estimated using the experimental set-up proposed in
 Figure 2.a (homogeneous cooling) or Figure 2.b (heterogeneous 1D cooling).

166 2.5.1. Homogeneous (0D) cooling

Homogeneous (0D) temperature fields are obtained during cooling. Equation 6 applied to the 'studied' and 'reference' specimens provides two equations:

$$\frac{(\rho_s C_s e_s + 2\rho_P C_P e_P)}{e_s} \frac{d\widetilde{\widetilde{T}}_s}{dt} = \rho_s \widetilde{\widetilde{q}}_s - f(\widetilde{\widetilde{T}}_s), \tag{14}$$

$$(\rho_r C_r e_r + 2\rho_P C_P e_P) \frac{d\widetilde{\widetilde{T}}_r}{dt} = -f(\widetilde{\widetilde{T}}_r).$$
(15)

Again, the heat losses function $f(\tilde{\widetilde{T}})$ is identical for 'reference' and 'studied' specimens. $f(\tilde{\widetilde{T}})$ is estimated from equation 15. Thus, at the temperature $\tilde{\widetilde{T}}_{s}$, the studied specimen heat source can be computed by:

$$\widetilde{\widetilde{\widetilde{q}}}_{s}(\widetilde{\widetilde{\widetilde{T}}}_{s}) = \frac{(\rho_{s}C_{s}e_{s} + 2\rho_{P}C_{P}e_{P})}{e_{s}\rho_{s}}\frac{\partial\widetilde{\widetilde{\widetilde{T}}}_{s}}{\partial t} + \frac{f(\widetilde{\widetilde{\widetilde{T}}}_{s})}{e_{s}\rho_{s}}.$$
(16)

174 2.5.2. Axially heterogeneous (1D) cooling

Using the experimental setup proposed in Figure 2.b, heterogeneous (1D) temperature fields are obtained. In that case equation 4 applied to each of the two specimens provides two equations:

$$\frac{(\rho_s C_s e_s + 2\rho_P C_P e_P)}{e_s} \frac{\partial \widetilde{\widetilde{T}}_s}{\partial t} - \frac{(k_s e_s + 2k_P e_P)}{e_s} \frac{\partial^2 \widetilde{\widetilde{T}}_s}{\partial x^2} = \rho_s \widetilde{\widetilde{q}}_s - \frac{f(x_s, \widetilde{\widetilde{T}}_s)}{e_s}, \quad (17)$$

$$(\rho_r C_r e_r + 2\rho_P C_P e_P) \frac{\partial \widetilde{\widetilde{T}}_r}{\partial t} - (k_r e_r + 2k_P e_P) \frac{\partial^2 \widetilde{\widetilde{T}}_r}{\partial x^2} = -f(x_r, \widetilde{\widetilde{T}}_r).$$
(18)

Equation 18 and experimental data for the reference specimen is used to estimate $f(x, \tilde{T})$. At the temperature $T = T_s = T_r$ and at the position $x = x_s = x_r$, heat sources released by the 'studied' material are:

$$\widetilde{\widetilde{q}_{s}}(x_{s},\widetilde{\widetilde{T}_{s}}) = \underbrace{\frac{\left(\rho_{s}C_{s}e_{s}+2\rho_{P}C_{P}e_{P}\right)}{e_{s}\rho_{s}}\frac{\partial\widetilde{\widetilde{T}_{s}}}{\partial t}}_{Heat \ storage \ term}} \underbrace{-\frac{\left(k_{s}e_{s}+2k_{P}e_{P}\right)}{e_{s}\rho_{s}}\frac{\partial^{2}\widetilde{\widetilde{T}_{s}}}{\partial x^{2}}}_{Conduction \ term}} \underbrace{+\frac{f\left(x_{s},\widetilde{\widetilde{T}_{s}}\right)}{e_{s}\rho_{s}}}_{Heat \ losses \ term}.$$
(19)

This experiment would be useful for heterogeneous specimen and wouldallow to determine local heat of transition.

183 3. Experimental validation

184 3.1. Experimental setup

In this study a SC7600 (Flir) camera was used. The camera works in the IR wavelength $\lambda = 3 - 5 \ \mu m$ with an InSb detector matrix (15 x 15 μm^2) and a resolution of 640 x 480 pixels. The accuracy of the camera is $\pm 2^{\circ}C$ in the concerned temperature range and its thermal resolution is about 0,02°C. The frame rate was 25 Hz. The industrial camera calibration has been performed with a black body in the range -10 to 300°C.

All specimens were painted with high emissivity paint measured to be $\epsilon = 0,95$. Paint thickness was measured with scanning electron microscope to be $25 \pm 5 \ \mu m$. IR transmission through the specimen and reflection of the environment, that can affect the measure, have been neglected.

Temperature measurements of the specimen located in the chamber were 195 performed through a quasi-transparent IR windows in CaF2 (Figure 3). This 196 window offers a transmission coefficient of $\tau = 0,92$ in the used wavelength. 197 The two specimens were heated up to a known temperature T_{ini} using a first 198 hot climatic chamber before being carried into a second cold climatic chamber 199 controlled at a known temperature T_0 . To reduce cooling while carrying the 200 specimens between the two chambers, a specific device was used. The natural 20 cooling of the specimens in this second climatic chamber, from T_{ini} to T_0 , was 202 observed with the IR camera. 203

In order to obtain homogeneous (0D) or axially heterogeneous (1D) temperature fields during cooling, two types of specimens and fixing were chosen (Figure 3). In the first case, small specimens were suspended with thin thermal insulator wires in order to obtain uniform temperature in each specimen (Figure 3.a). In the second case, slender specimens were used and gripped in a steel block, acting as a thermal mass. This thermal mass initially at temperature T_0 cooled very slowly and created an axial temperature gradient between the free and gripped extremities in each specimen (Figure 3.b).

Reproducibility and heat losses distributions in time and space were tested. Experiments were realized using for the reference and studied specimens two identical specimens with the same material. In such a case, responses of the two specimens were measured to be identical in the whole range of temperature.

217 3.2. Materials

As explained in the first section, a 'reference' specimen is used to evaluate 218 heat losses functions in equations (9),(12),(15),(18). Pure Titanium, in 219 the form of 0.51 mm thickness sheet, was selected in our experiments as 220 'reference' specimen. The thermophysical properties of this material are 22 given in Table 1 as reported in the literature. They were also measured using 222 classical methods. Its heat capacity was measured with a DSC experiment 223 and was estimated to $C_{Ti}^{DSC} = 530 \pm 30 \ Jg^{-1}K^{-1}$. This result is in good 224 agreement with the literature values (Table 1). In the following, a value of 225 $C_{Ti} = 530 \ Jg^{-1}K^{-1}$ has been used. The thermal diffusivity $(\lambda = \frac{k}{\rho C})$ of 226 this Titanium specimen was estimated using a laser flash method [33, 36, 22 37]. Knowing the heat capacity of the material, the thermal conductivity of 228 Titanium was estimated to $k_{Ti}^{lf} = 20 \pm 2 W m^{-1} K^{-1}$. This result is in good 229 agreement with the values of the literature (Table 1). In the following, a 230 value of $k_{Ti} = 20 \ Wm^{-1}K^{-1}$ has been used. 23



The 'studied' specimens were cut either in pure Vanadium rolled plate

of thickness 0.50 mm (Sections 3.3 and 3.4) or in a Ti - 50.2 at.% Ni SMA bright rolled plate of thickness 0.39 mm (Section 3.5). The thermophysical properties of these materials and of the high emissivity paint are given in Table 1 as reported in the literature.

Material	Volumic mass	Specific heat	Thermal conductivity
	$ ho$ $(kg~m^{-3})$	$C \ (J \ kg^{-1} \ K^{-1})$	$k \ (W \ m^{-1} \ K^{-1})$
Ti [25, 26, 27, 28, 29]	4510	520 - 555	17 - 22
Va [25, 26, 27, 30]	6000	480 - 505	28 - 36
NiTi [31, 32, 33]	6400	480 - 520	9 - 15
Paint $[34]$	1500	1500	0.2

Table 1: Thermophysical properties of used materials (literature values).

Figure 4 shows the DSC of the NiTi SMA selected for the study (section 3.5). This DSC was performed using a DSC TA Q200, with a 10°C min⁻¹ heating/cooling rate, with a specimen of 22.9 mg cut with a diamond blade. During cooling, two successive phase transformations were observed:

• from Austenite to R-phase, with $R_s = 62^{\circ}C$, $R_f = 53^{\circ}C$ the starting and finishing temperatures, respectively,

243 244 • from R-phase to Martensite, with $M_s = 39^{\circ}C$ and $M_f = 12^{\circ}C$ the starting and finishing temperatures, respectively.

The latent heat released during complete phase transformation, evaluated from the baseline plotted in Figure 4, is equal to $\Delta H_{A-M} = 21 J g^{-1}$. During heating, a single phase transformation from Martensite to Austenite is observed, with starting and finishing transformation temperatures equal to A_s = 69°C and $A_f = 84$ °C, respectively.

250 3.3. Data processing

Temperatures $T_s(x, y, t)$ and $T_r(x, y, t)$ measured with the infrared camera on the surface of the samples need to be processed to estimate the desired properties $(C, k \text{ or } \dot{q})$. The complete data processing flowchart performed with Matlab is presented in Figure 5 for the 1D heterogeneous case.

In the 1D heterogeneous case, the temperature is supposed homogeneous 255 in the y direction. This assumption was experimentally checked. Thus, tem-256 poral averaging in the y direction was estimated and data $\tilde{\widetilde{T}}_s$ and $\tilde{\widetilde{T}}_r$ were 25 obtained. Low pass temporal filtering was then applied to data as reported 258 in [12, 13]. Then, first derivative terms were estimated by finite difference 259 method and were spatially filtered. To estimate laplacian terms, data $\widetilde{\widetilde{T}_s}$ and 260 \widetilde{T}_r were spatially filtered with a polynomial of degree 4. Knowing the polyno-261 mial coefficients, laplacian terms were estimated. This laplacian estimation 262 was studied and checked in [33]. Once the spatial and temporal derivative 263 terms estimated, the heat losses function f and desired property can be es-264 timated using equations presented in section 2. A quasi similar flowchart 26 could be presented in the homogeneous case. Temperatures $T_s(x, y, t)$ and 266 $T_r(x, y, t)$ were averaged in x and y directions and only the derivative term 26 versus time was estimated. 268

$_{269}$ 3.4. Validation of the TFM heat capacity C measurement

In this section, the experimental setup proposed in Fig. 3.a and temperatures $T_{ini} = 60^{\circ}C$ and $T_0 = 0^{\circ}C$ were chosen (Table 2). The reference

Ref. Mat.	Studied Mat.	Exp. Setup	T_{ini}	T_0
Ti	Va	Fig. 3.a	$60^{\circ}C$	$0^{\circ}C$
e = 0.51 mm	e = 0.50 mm			

²⁷² material was Titanium. The studied material was Vanadium.

Table 2: Information and parameters for TFM heat capacity measurement.

Figure 6.a shows the measured natural cooling curves of the Ti and Va specimens. Due to heat losses during the specimens transportation between the heat and cold chambers, the starting temperatures of the two specimens were slightly lower than temperature of the first chamber $T_{ini} = 60^{\circ}C$. Figure 6.b shows the cooling rate of the two specimens in function of the specimen temperatures (estimated from the cooling curves). During that experiment, cooling rate decreases approximately linearly with the temperature.

Figure 7 shows the Vanadium heat capacity estimated with the TFM method for three experiments in the range 10 to 30°C using equation 10. This range was chosen because filtering process presented in section 3.3 involves edge effects on the estimation of the derivative terms. Moreover, below 10° C, the denominator of equation 10 is low and induces important noise. Results were obtained using the cooling rates $(\frac{dT}{dt})$ presented in Figure 6.b and a constant heat capacity $C_{Ti} = 530 \ Jkg^{-1}K^{-1}$.

A constant mean value $C_{Va}^{TFM} = 495 \pm 10 J k g^{-1} K^{-1}$ was determined from these curves. As the C_{Ti} is known with an accuracy of $\pm 30 J g^{-1} K^{-1}$ from the DSC measurement, the Va heat capacity is finally estimated to be $C_{Va}^{TFM} = 495 \pm 40 J k g^{-1} K^{-1}$ with the TFM method. This result is in good agreement with those obtained from DSC on the same material: $C_{Va}^{DSC} = 480 \pm 40 \ Jkg^{-1}K^{-1}$ and from literature $C_{Va}^{lit} = 480 - 505 \ Jkg^{-1}K^{-1}$, obtained on different Va materials. The method proposed is thus considered experimentally validated. Heat capacity can be estimated with the same accuracy than the DSC method.

²⁹⁶ 3.5. Validation of the TFM thermal conductivity k measurement

In this section, the used experimental setup is shown in the Figure 3.b and temperatures $T_{ini} = 60^{\circ}C$ and $T_0 = 0^{\circ}C$ were chosen (Table 3). Vanadium was used as the 'studied' material. Titanium was the 'reference' material.

Ref. Mat.	Studied Mat.	Exp. Setup	T_{ini}	T_0
Ti	Va	Fig. 3.b	$60^{\circ}C$	$0^{\circ}C$
e = 0.51 mm	$e=0.50~\mathrm{mm}$			

Table 3: Information and parameters for TFM thermal conductivity measurement.

Figure 8 shows the thermal responses of Ti and Va specimens during the 300 1D experiment. Figure 8.b is the spatio-temporal thermal response of the 301 Ti specimen along the dashed line sketched in Figure 8.a. Figure 8.c shows 302 the temperature temporal evolution of three pixels of Ti and Va specimens. 303 Figure 8.d shows the axial thermal profiles sketched in Figure 8.a at different 304 times for the Ti specimen. The specimens cooled during the experiment 305 (Figures 8.b and c), from a quasi-homogeneous temperature to a thermally 306 heterogeneous state with a gradient (Figures 8.b and d) in the specimen main 30 direction. The Ti specimen cooled faster than the Va specimen, as during the 308 0D experiment (Figure 8.c). Initial temperatures of the two specimens were 309 almost equal (Figure 8.c). At the end of the experiment, the edge close to 310 thermal mass was hotter than the free edge (Figure 8.d). As noted in Table 311

³¹² 1, thermal conductivity of Titanium and Vanadium are different, leading to ³¹³ different heat flux through the specimens. This explains why, at the end of ³¹⁴ the experiment, the two specimens were not at room temperature T_0 and ³¹⁵ that the temperatures were different at a given distance of the thermal mass.

Figure 9 shows the estimated thermal conductivity of the Va specimen, 316 using the equation (13). In this equation, the heat loss function $f(x, \tilde{\widetilde{T}})$ was 31 estimated from equation 12 and experimental data for the 'reference' spec-318 imen. Conductivity values are presented along the axial profile for instants 319 higher than t=30s in Figure 8.c. For those instants, first derivative terms 320 $\left(\frac{\partial \tilde{T}}{\partial t}\right)$ in equations 12 and 13 were negligible compared to the spatial second 32 derivative $\frac{\partial^2 T}{\partial x^2}$. For the two samples, as observed on profiles presented in 322 Fig. 8.d, laplacian term is higher close to steady state. To avoid important 323 edges effect due to spatial filtering, only the central pixels were considered. 324 Using $k_{Ti} = 20 \ Wm^{-1}K^{-1}$, a mean thermal conductivity of Vanadium is 325 estimated to be $k_{Va}^{TFM} = 37 W m^{-1} K^{-1}$. Note that this result is in good 326 agreement with literature results (Table 1). Taking into account dispersion 32 of Ti thermal conductivity, heat capacity, and Va heat capacity, Va thermal 328 conductivity is estimated to be $k_{Va}^{TFM} = 36 \pm 4 \ Wm^{-1}K^{-1}$ while it was estimated 329 timated to be $k_{Va}^{lf} = 34 \pm 3 \ Wm^{-1}K^{-1}$ with a classical laser flash method 330 and to be $k_{Va}^{lit} = 28 - 36 \ Wm^{-1}K^{-1}$ in the literature. Thus, while esti-33 mating thermal conductivities, the accuracy depends on the knowledge of 332 thermophysical properties of the reference material with the TFM method. 333

334 3.6. Validation of the TFM heat of transition measurement

In this last section, Ti is the 'reference' material and NiTi is the 'studied' specimen. To observe the complete exothermic transformation from Austenite to Martensite occurring in the NiTi specimen, temperature T_{ini} had to be above A_f and temperature T_0 below M_f . In this experiment, the following values were thus chosen: $T_{ini} = 100^{\circ}C$ and $T_0 = 0^{\circ}C$.

Experimental results obtained from homogeneous (using device presented in Fig. 3.a) and then from axially heterogeneous (using device presented in Fig. 3.b) cooling are presented and discussed.

343 3.6.1. Homogeneous cooling

Information and parameters chosen for this experiment are given in Table4. The experimental setup is described in Fig. 3.a.

Ref. Mat.	Studied Mat.	Exp. Setup	T_{ini}	T_0
Ti	NiTi	Fig. 1.a	$100^{\circ}C$	$0^{\circ}C$
e = 0.51 mm	$e=0.39~\mathrm{mm}$			

Table 4: Information and parameters for TFM heat heat of transition measurement in the homogeneous case.

Figure 10.a shows the temporal evolution of temperature measured in the 346 Ti and NiTi specimens during homogeneous cooling experiment (Figure 3.a). 347 The Ti temperature decreasing curve obtained for the Ti specimen is 348 exponential like. However, for the NiTi specimen, two bumps due to the two 349 exothermic phase transformations are noted, from Austenite to R-phase and 350 from R-phase to Martensite, successively. The two specimens coolings started 35 and finished to identical temperatures. Figure 10.b shows the cooling rate T352 of Ti and NiTi specimens. The cooling rate range is between -1 and $-8Ks^{-1}$. 353 From these curves and from equations 18 and 19, heat source occurring in 354 the NiTi specimen can be estimated. 355

Figure 11.a presents the ratio $\frac{\dot{q}}{|\dot{T}|}$ results for the TFM method using a heat capacity for NiTi specimen equal to 500 $Jkg^{-1}K^{-1}$ [31, 32, 33]. DSC curve is also presented in black.

Global shapes of the curves obtained by the two techniques are similar; 359 the amplitude of the peaks by the two methods are in good accordance. Ta-360 ble 5 summarizes the two peak temperatures, respectively noted T_{A-R} and 361 T_{R-M} and the transformation temperature R_s , R_f , M_s and M_f for the two 362 techniques (DSC (Figure 4) and TFM 0D): peak and transformation temper-363 atures are almost equal for the two techniques. From these values, starting 364 temperature R_s and M_s for the two transformations appear to be very close 36! for the two methods. Finishing temperature R_f and M_f are however lightly 366 higher with the TFM method. Such a difference can partially be explained 36 by thermal inertia effects in the DSC experiment: this inertia leads to un-368 derestimate finishing temperature of the transformation with this technique 369 [33, 38]. With the TFM method, the specimen transforms naturally and 370 independently of the environment; the TFM method exhibits no thermal in-37 ertia. Differences in the temperatures may also be due to the accuracy of the 372 IR camera $(\pm 2^{\circ}C)$ and of the DSC one. 373

Eventually, integrating the ratio $\frac{\dot{q}}{|\dot{T}|}$ over temperature allows to estimate the energy released by the material versus temperature:

$$E = \int_{T_1}^{T} \frac{\dot{q}}{|\dot{T}|} dT.$$
 (20)

Its evolution is plotted versus temperature, for TFM and DSC techniques in Figure 11.b. For the two methods, transformed energy E is estimated between the initial starting temperature $T_1 = R_{si} = 74^{\circ}C$ and the cur-

$^{\circ}C$	DSC	TFM 0D	TFM 1D
T_{A-R}	58	60	59
T_{R-M}	28	32	30
R_s	62	63	62
R_f	53	57	54
M_s	39	39	39
M_f	12	17	19

Table 5: Peaks temperatures and transformation temperatures estimated from DSC, TFM 0D and TFM 1D.

rent temperature. The energy, for the transformation finishing temperature $M_{fe} = 12^{\circ}C$ (see Figure 11.a), corresponding to the latent heat of transition of the material, is estimated to be 19 Jg^{-1} for the TFM method. From the DSC curve, the latent heat of transition is estimated to be 19.5 Jg^{-1} , using the baseline plotted in Figure 11.a. The results using the TFM method and DSC are in good agreement.

For the TFM method, the main issue is the knowledge of the thermophysical properties of the material. A similar drawback occurs for the DSC, where the choice of the baseline is the key point to realize quantitative latent heat of transition measurements [23]. The two techniques are however quantitatively in good agreements.

390 3.6.2. One dimensional heterogeneous cooling

Information and parameters chosen for this experiment are given in Table 6.

³⁹³ Spatio-temporal evolution along an axial profile is plotted for Ti specimen

Ref. Mat.	Studied Mat.	Exp. Setup	T_{ini}	T_0
Ti	NiTi	Fig. 1.b	$100^{\circ}C$	$0^{\circ}C$
$e=0.51~\mathrm{mm}$	$e=0.39~\mathrm{mm}$			

Table 6: Information and parameters for TFM heat of transition measurement in the heterogeneous case.

in Figure 12.a and for NiTi specimen in Figure 12.b during heterogeneous 394 cooling (Figure 3.b). In Figures 12.c.d, temporal evolution of Ti and NiTi 395 specimens temperatures are respectively plotted for three pixels (pixel 65, 396 100 and 150) located at different distances of the thermal mass. At the be-39 ginning of the experiment, all points had identical temperature and difference 398 of temperature appeared during cooling. At the end of the experiment, the 399 thermal mass was still hot and conductivity occurred in the specimens: pixels 400 close to the thermal mass were hotter than those close to the free edge (Fig-401 ures 12.e.f). In the Ti specimen, natural cooling of every pixel was observed, 402 as during the 0D experiment. Additionally for NiTi specimen (Figure 12.d), 403 the two phase transformations from Austenite to R-phase and from R-phase 404 to Martensite can be observed. Figures 12.e.f eventually show the axial ther-405 mal profiles of the two specimens at different times. However, for the NiTi 406 specimen, bumps appeared around times t=10s and t=20s in the thermal 40 profiles due to exothermic phase transformations occurring in the specimen. 408 The ratio $\frac{\dot{q}}{|\dot{T}|}$ was estimated from 1D thermal profiles (Figure 13.a), using 409 a heat capacity for NiTi specimen equal to 500 $Jkg^{-1}K^{-1}$ [31, 32, 33] and a 410 thermal conductivity of 11 $Wm^{-1}K^{-1}$. Current study [33] shows that ther-41 mal conductivity can be considered identical for Austenite and Martensite 412

⁴¹³ phases. The spatio-temporal evolution of this ratio is plotted in Figure 13.b. ⁴¹⁴ The spatio temporal propagation of the two phase transformations, from ⁴¹⁵ Austenite to R-phase and from R-phase to Martensite are clearly visible: the ⁴¹⁶ transformation started from the bottom (fast cooling) and finished to the top ⁴¹⁷ (slow cooling due to thermal mass inertia) of the specimen.

The ratio $\frac{\dot{q}}{|\dot{T}|}$ calculated with the TFM method is plotted in Figure 13.c, 418 versus specimen temperature, for pixels 65, 100 and 150 in red, blue and green 419 respectively. The ratio $\frac{\dot{q}}{|\dot{T}|}$ obtained with DSC is also plotted. Qualitatively, 420 the ratios estimated for the three pixels are well superimposed. The peak 42 temperatures and the transformation temperatures for the two techniques 422 (DSC and TFM 1D) are given in Table 2. The peaks obtained with the TFM 423 method and with the DSC are in good agreement. As in the homogeneous 424 case, transformation temperatures estimated with the TFM and DSC (Figure 42 4) methods are in good agreement. As in the homogeneous case, thermal 426 inertia effects are once again observable in the DSC method. 427

Evolution of the energy released as function of the temperature during 428 the transformation is also plotted in Figure 13.d. It is estimated from TFM 429 technique for considered pixels and from DSC for the baseline presented in 430 Figure 13.c. Evolution of the energy is similar for the two techniques. The 431 latent heat of transition obtained from TFM method is $17.5 \pm 1 Jg^{-1}$ for all 432 considered pixels and is slightly lower than the one estimated by DSC. This 433 error is partially due to the underestimation of the heat sources with the 434 process used, but also due to some experimental constraints: as explained, 435 the room temperature was $T_0 = 0^{\circ}C$ (to avoid freezing problems on the 436 window and on the specimen) which is close to M_f . In the heterogeneous case, 43

the final temperature of every pixel is not T_0 but lightly above, as observed in Figures 12.c.d. Thus, in a part of the specimen, the phase transformation did not occurred totally and released less energy.

However, knowledge of materials thermophysical properties can affect 441 results. Faulkner et al. [39] measured conductivity coefficients equal to 442 14 $Wm^{-1}K^{-1}$ and 28 $Wm^{-1}K^{-1}$ for martensite and austenite, respectively. 443 In this study, thermal conductivity of k_{NiTi} was assumed constant $(11 Wm^{-1}K^{-1})$ 444 and independent of the considered phase, as measured in [33]. Figure 14 yet 445 shows the evolution with the temperature of the terms involved in the equa-446 tion 19 to estimate massic heat sources \dot{q} in NiTi sample for pixels 60 to 70. 44 Red curve shows the heat storage term, the blue one is for conduction in the 448 sample and the green one represents heat losses. Adding these terms leads 449 to the red curve plotted in Figure 13.c. Main terms are linked to the storage 450 one (red) and to heat losses (green). The conduction term (blue) is almost 45 negligible for temperature above 20° C. Thus, the accuracy of the k_{NiTi} is 452 not important in that study. 453

The TFM method in an heterogeneous case exhibits yet quantitative results in good agreement with those obtained by DSC. Note that in this study, the chosen material had homogeneous transformation behavior. As the determination of the properties is local, this technique would allow to determine local latent heat of transition even if the material exhibited heterogeneous transformation behavior.

460 4. Conclusion

Thermal Field Measurement (TFM) method has been developed and used 461 to estimate heat capacity, thermal conductivity and latent heat of transition 462 of materials. Advantages of this original method are (i) the non-contact as-463 pect, measuring temperature with an infrared camera, while others methods 464 are with contacting methods and (ii) the local aspect of the measurement in 465 the heterogeneous case while other method are global. This last point is very 466 interesting in the case of heterogeneous material. (iii) The absence of inertia 46 effect as in DSC measurement. 468

The experimental setup to perform the measurement was based on the simultaneous observation, with an infrared camera, of the natural cooling of two specimens: a 'reference' and a 'studied' material. Titanium was used as 'reference' specimen to estimate convection and radiation heat losses.

Firstly, Vanadium has been used to validate the method abilities to es-473 timate thermophysical properties. The TFM method has been proved to be 474 able to provide an accurate measurement of heat capacity C and local ther-475 mal conductivity k of Vanadium. Results obtained from TFM techniques, 476 classical DSC, laser flash method and literature were in good agreement. Sec-47 ondly, NiTi specimen was studied and chosen for its exothermic thermally 478 induced phase transformation. Latent heat of transition of this material was 479 estimated during homogeneous and heterogeneous thermal cases. Results 480 obtained with this technique and with DSC are in good agreement. 48

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Figure 1: Specimen of thickness e_1 coated with the two paint layers of thickness e_P .



Figure 2: Reference(r) and Studied (s) specimens configurations in (a) homogeneous (0D) and (b) heterogeneous (1D) cooling.



Figure 3: Experimental setup proposed to obtain an (a) homogeneous (0D) and (b) an heterogeneous (1D) cooling.



Figure 4: Differential Scanning Calorimetry thermogram of a NiTi DSC specimen.



Figure 5: Data processing flow chart.



Figure 6: Homogeneous natural cooling of Titanium and Vanadium specimens. a) Temperature versus time and b) Temperature rates versus temperature.



Figure 7: Heat capacity estimation of Vanadium for three distinct experiments.



Figure 8: Thermal responses for Titanium and Vanadium specimens in 1D experiment. (a) Thermal image at a given time showing the two specimens. (b) Spatio-temporal representation of temperature along the axial profile plotted in (a) for Titanium specimen. (c) Temporal evolution of the temperature in three different localizations. (d) Axial thermal profiles at different times.



Figure 9: Local thermal conductivity estimation of Vanadium using $k_{Ti} = 20 W m^{-1} K^{-1}$.



Figure 10: Homogeneous natural cooling of Titanium and NiTi specimens. a) Temperature versus time and b) Temperature rate versus temperature.



Figure 11: Estimation of transition heat - homogeneous natural cooling of the NiTi specimen. (a) Ratio $\frac{\dot{q}}{|T|}$ and (b) energy evolution, measured by DSC and by TFM techniques.



Figure 12: Thermal responses for Titanium and NiTi specimens in 1D experiment. Spatiotemporal representation of temperature along the axial profile for (a) Titanium and (b) NiTi specimen. Temporal evolution of the temperature in three different localizations for (c) Titanium and (d) NiTi specimen. Axial thermal profiles at different times for (e) Titanium and (f) NiTi specimen.



Figure 13: Estimation of transition heat - heterogeneous natural cooling of the NiTi specimen. (a) Spatio-temporal representation of temperature along the axial profile for NiTi specimen. (b) Spatio-temporal representation of ratio $\frac{\dot{q}}{|\vec{T}|}$ evaluated with TFM technique. (c) Comparison of the ratio calculated from DSC and TFM techniques and (d) Energy evolution during cooling for pixels 60 to 70, 95 to 105 and 145 to 155.



Figure 14: Evolution, with the temperature, of the terms involved in the massic heat sources estimation for NiTi sample (equation 19) for pixels 60 to 70.