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AN EXPERIMENTAL TECHNIQUE FOR THE SIMULTANEOUS ESTIMATION OF THERMAL PROPERTIES OF METALS AS FUNCTIONS OF TEMPERATURE

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Abstract. *In this work, a simple experimental approach for the simultaneous estimation of temperature-dependent specific heat and thermal conductivity of metallic materials is presented. The thermal model developed is based on a one-dimensional, nonlinear and transient metal plate, which receives a constant heat flux at the top surface and is insulated at the bottom surface. The study is carried out in a 316 stainless steel sample. The inverse problem is solved by comparing temperature measurements from the sample with the numerical solution of the heat diffusion equation achieved in COMSOL software. The Levenberg–Marquardt method is applied for each time step of the experiment in order to simultaneously evaluate the thermal properties of the material for every slight change in temperature. By performing a sensitivity analysis, a priori information about the viability of the estimation can be obtained and the experimental aspects can be set. A reducing factor is considered on the experimental heat flux due to the imperfect contact between the heater and the metallic sample. In addition, the statistical study of the confidence intervals for each property and the comparison with the literature show the reliability of the outcomes. Lastly, the developed method is validated by analyzing the errors produced in the numerical and experimental processes.*

Keywords: *heat conduction, inverse problem, temperature-dependent thermal properties, metals, optimization*

1. INTRODUCTION

The correct knowledge of the thermal properties of materials is an important issue when dealing with processes in which thermal analysis is required. This kind of investigation is frequently needed in mechanical engineering. It can be applied in manufacturing processes such as welding, machining, forging and others. It is also essential when designing equipment for high temperature applications, like aeronautical, aerospace or automobile components. Trustful properties enable simulations and studies that correctly characterize the behavior of systems and their elements when operating under temperature variations, ensuring the quality of the outcomes.

Over the years, several methods have been developed aiming to determine thermophysical properties of materials. However, some of these methods disregard the dependence of the properties on temperature, others cannot be applied in metallic materials (Jannot *et al.*, 2009, Mohamed, 2009). Also, in some techniques it is necessary to perform more than one procedure to achieve different properties, since the simultaneous estimation is not allowed (Barani *et al.*, 2019, Jannot *et al.*, 2020). Another concern when dealing with temperature-dependent thermal properties is the nonlinearity of equations. Many authors perform the estimations within small temperatures rises, in which the parameters are assumed to be constant (Pavlov *et al.*, 2018, Carollo *et al.*, 2019). In these techniques several experiments must be performed adjusting the initial condition. Moreover, some studies are carried out with no experimental validation, presenting just analytical and numerical solutions (Ren, *et al*., 2016, Lembcke *et al*., 2016). It is also important to show that just a few of these methods can be applied in metals, considering that the effects of contact resistance are intensified in these materials (Tariq and Asif, 2015, Ramos *et al.*, 2020). Finally, in most cases, the experimental apparatus to achieve the thermal properties is expensive. So, in this work is presented a technique to estimate thermal conductivity and specific heat simultaneously and varying with temperature in a 316 stainless steel sample using transient temperature data from a single and low-cost experiment performed at room temperature. The experimental setup does not require an oven and the temperature rise is about 150 °C. In addition, the contact resistance between heater and sample is also studied along the numerical analysis.

The investigation of metals is due to their high applicability in engineering problems. Not only in the situations mentioned above, since their discovery, metallic materials have been playing an important role in human activities, especially in industry. Their usefulness stimulated the development of alloys with consistent improvements in their properties. Example of this is the stainless steel, required when resistance to corrosion is a key issue. The 316 stainless steel alloy, in particular, has been widely employed throughout the industry, bringing together resistance to corrosion, strength, hardness, chemical and thermal stability. For these materials, obtaining thermal properties is a laborious issue because of the lack of sensitivity to perform the estimation and also because of the strong effect of the contact resistance, which restrain the direct measurement of the properties. In these cases, the inverse analysis represents a powerful tool to achieve not only the thermophysical properties of a material, but also initial and boundary conditions (Beck *et al*., 1985, Alifanov, 1994). A gradient-based method, the Levenberg-Marquard (L-M) algorithm, has been commonly applied to solve this kind of problem (Taler *et al.*, 2014, Huang *et al*., 2015, Cui *et al.*, 2017). It shows great performance and a fast convergence rate, being employed for linear and nonlinear models. However, despite being very useful, inverse problems are quite often ill-posed and very sensitive to measurement errors, what may cause instability in the solution.

In this work, the Levenberg–Marquardt method is applied in order to simultaneously evaluate the temperature-dependent thermal conductivity, k, and specific heat, c_p, in a 316 stainless steel sample. The thermal model developed is based on a one-dimensional, nonlinear and transient metal plate, which receives a constant heat flux at the top surface and is insulated at the bottom surface. A reducing factor is considered on the experimental heat flux due to the imperfect contact between the heater and the metallic sample. The inverse problem is solved by comparing temperature measurements from the sample with the numerical solution of the heat diffusion equation achieved in COMSOL software for each time step of the experiment. Thermal properties of the material are achieved for every slight change in temperature. In addition, the statistical study of the confidence intervals for each property and the comparison with the literature show the reliability of the outcomes. A simple and low-cost experimental approach is proposed. The experimental aspects and a priori information about the viability of the estimation can be obtained by performing sensitivity analysis. Lastly, the developed method is validated by analyzing the errors produced in the numerical and experimental processes.

2. METHODS

2.1 Thermal model

This work proposes the study of a nonlinear, transient and one-dimensional problem, disregarding radiation, convection, phase change and heat generation phenomena. The material is homogenous and isotropic, so the thermal properties are considered to be only functions of temperature. A squared section sample is placed between a resistive heater and the thermal insulation, as illustrated in Figure 1.

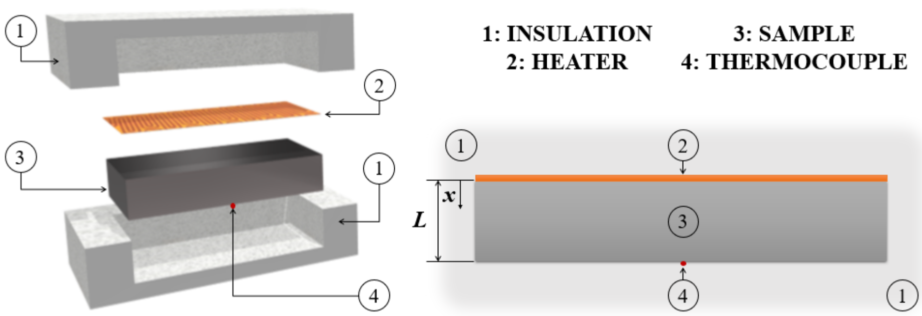

Figure 1. Schematic representation of the one-dimensional thermal model.

The heater delivers a constant heat flux to top surface of the sample and a thermocouple in the interface sample-insulation measure the temperature distribution. The heat diffusion equation for this study is given by:

$$\frac{\partial}{\partial x}\left[k(T)\frac{\partial T(x,t)}{\partial x}\right] = \rho c_p(T)\frac{\partial T(x,t)}{\partial t} \tag{1}$$

With the initial condition:

$$T(x,0) = T_0 \tag{2}$$

Subject to the boundary conditions:

$$-k(T)\frac{\partial T(x,t)}{\partial x}\bigg|_{x=0} = h[T_h - T_p](t) \tag{3}$$

$$\frac{\partial T(x,t)}{\partial x}\bigg|_{x=L} = 0 \tag{4}$$

where x is the heat diffusion direction, T is temperature, t is time, k is the thermal conductivity, c_p is the heat capacity, ρ is density, T_0 is initial temperature, T_h and T_p are the heater and plate temperatures respectively, h is contact conductance - the contact resistance reciprocal, and L is the length of the sample.

2.2 Numerical solution

In this work, the numerical solution of the direct heat conduction problem was achieved by COMSOL Multiphysics, since it is necessary to evaluate the temperature field of a transient non-linear problem. Besides that, at the interface of the heater and the metallic sample, the contact conductance acts as a reducing factor in the heat flux delivered to the sample. The contact conductance is due to two factors: the presence of air, causing interstitial conductance; and contact points, responsible for the constriction conductance. These two factors are computed by the correlation of parallel-plate gap gas and the correlation of Cooper-Mikic-Yovanovich (CMY), respectively. To perform the study, variables such as the roughness of the sample and heater must be set in COMSOL. The complete methodology used to assess the contact conductance is detailed in Ramos *et al.* (2020). The software efficiently calculates the sensitivity coefficients for experimental improvement, in addition to having the contact resistance already taking into account in its solver. In order to evaluate the numerical temperature distribution COMSOL Multiphysics uses the Backward Differentiation Formula (BDF), an implicit time approximation method, and the PARDISO package, an efficient and straightforward solver for linear and nonlinear equations.

2.3 Sensitivity analysis

Quality of the outcomes is strongly affected by how sensitive the temperature is in relation to changes in the parameters being estimated. In other words, sensitivity coefficients aim to quantify the effect of the parameter in the temperature distribution. These coefficients are defined by Beck *et al.* (1985) as the first partial derivative of temperature in relation to the thermal properties k and c_p. When dealing with simultaneous estimation it is appropriate to multiply these coefficients by the current parameter, so that the normalized coefficients (X) have the same basis, the unit of temperature. The normalized coefficients can be obtained by the following expression:

$$X_{P_j} = P_j \frac{\partial T}{\partial P_j} \cong P_j \left[\frac{T\big(P_1, \dots, P_j + \delta P_j, \dots, P_N\big) \; - \; T\big(P_1, \dots, P_j, \dots, P_N\big)}{\delta P_j}\right] \tag{5}$$

where T is the numerically calculated temperature, N is the number of parameters, P_j the parameter to be analyzed and δ is a small positive scalar, equal to 0.001.

In order to achieve optimal conditions to perform the inverse analysis, sensitivity coefficients may be maximized. Higher coefficients show that the temperature is sensitive enough to provide reliable information about the estimates. So, through sensitivity analysis, several experimental aspects can be defined, such as intensity of heat flux and interval of application, data acquisition frequency, number of sensors and their location. These variables are defined to ensure maximum sensitivity for both parameters.

In simultaneous estimations it is also important to analyze the behavior of these coefficients when compared to each other. In order to guarantee the correct estimation for k and c_p, the magnitude of their sensitivity cannot be substantially different, as well as the variation of the coefficients through time cannot present dependence on each other. These two conditions avoid problems in the estimation process.

2.4 Inverse estimation

In order to perform the estimation of the thermal parameters (P), the minimization of an objective function (S) is proposed. For this purpose, k and c_p remain constant within a number of future time steps, r, and then are simultaneously estimated for each time step, M. The properties are determined using the L-M method to minimize the function S, defined as the squared difference of experimental temperatures, Y, and numerical temperatures, T, as follows:

$$S(\boldsymbol{P}) = \sum_{j=1}^{r} \big[Y_{i+j-1} - T_{i+j-1}(\boldsymbol{P})\big]^2 \tag{6}$$

The experimental temperature is measured from the sample and the numerical temperature is solved in COMSOL. The L-M algorithm here described to minimize the objective function $S(P)$ was modified. Thermal properties are taken as constants over a number of future times. So, each time step considers information from M until the observation (M - r). This introduces regularization to the inverse estimation, allowing the outcomes to be less affected by the measurement errors. So, in this case, as a result of the minimization of Eq. (6) the parameters P for each iteration n can be written as:

$$\boldsymbol{P}^{n+1} = \boldsymbol{P}^n + [(\boldsymbol{J}^n)^T \boldsymbol{J}^n + \mu^n \boldsymbol{\Omega}^n]^{-1} (\boldsymbol{J}^n)^T [\boldsymbol{Y} - \boldsymbol{T}(\boldsymbol{P}^n)] \tag{7}$$

where μ is the damping factor, a positive scalar updated at each iteration and $\boldsymbol{J}$ and $\boldsymbol{\Omega}$ are both matrices given by the expressions:

$$\boldsymbol{J}(\boldsymbol{P}) = \left[\frac{\partial \boldsymbol{T}^T(\boldsymbol{P})}{\partial \boldsymbol{P}}\right] \tag{8}$$

$$\boldsymbol{\Omega} = diag[(\boldsymbol{J})^T \boldsymbol{J}] \tag{9}$$

The procedure to achieve the sensitivity matrix is the same applied before in the calculation of the sensitivity coefficients of k and c_p:

$$J_l = \frac{\partial T}{\partial P_l} \cong \frac{T(P_1, \dots, P_l + \delta P_l, \dots, P_N) - T(P_1, \dots, P_l, \dots, P_N)}{\delta P_l} \tag{10}$$

Possible instabilities in the first iterations are also regularized by the term $\mu^n \boldsymbol{\Omega}^n$. Subsequently, depending on the resultant objective function, the dumping factor is updated.

The iterative procedure is performed until any of the following stopping criteria are satisfied.

$$S(\boldsymbol{P}^{n+1}) \leq \varepsilon \tag{11}$$

$$|\boldsymbol{P}^{n+1} - \boldsymbol{P}^n| \leq \varepsilon \tag{12}$$

In Equations (11) and (12) ε is the tolerance level, specified to be 0.001.

For each time step, both properties are iteratively estimated. After obtaining the values for thermal conductivity and specific heat for all experimental points measured, a linear regression is performed to fit the estimated points with the temperature data. The resultant function expresses how the thermal properties vary with temperature.

2.5 Confidence analysis

The accuracy of an estimation can be achieved performing the confidence interval analysis. When dealing with estimation procedures, it is appropriate to evaluate the bounds in which is likely that the true value of a parameter is placed. This kind of analysis brings reliability to the estimated points.

Through the Bonferroni method (Seber and Wild, 2003), the standard deviation can be approximated for each point of the estimation. The confidence bounds are achieved considering elements from a covariance matrix, derived from the sensitivity matrix, described as follows:

$$cov(\boldsymbol{P}) = (\boldsymbol{J}^T \boldsymbol{J})^{-1} \sigma^2 \tag{13}$$

The confidence interval for an estimated parameter is given by:

$$\bar{P}_l \pm t_{1-\alpha/2l}^{m-l} (C_{ll})^{1/2} \sigma \tag{14}$$

where $\bar{P}$ is the parameter estimated, σ is the standard deviation of measured data, $t_{1-\alpha/2l}^{m-l}$ is the t-distribution value for a 95% probability, assuming α of 0.05. The number of observations is denoted by m, while l is the number of parameters. The term $C_{ll}^{1/2}\sigma$ represents the element of the covariance matrix related to the parameter being estimated.

Consequently, the true value for the parameter estimated is within the range $\left[\bar{P}_l - t_{1-\alpha/2l}^{m-l} (C_{ll})^{1/2} \sigma \leq \bar{P}_l \leq \bar{P}_l + t_{1-\alpha/2l}^{m-l} (C_{ll})^{1/2} \sigma\right]$, with a confidence level of 95%.

For each point of the experiment, besides the value of the properties, are also computed the upper and lower bounds of the confidence interval. The points are then fitted through a linear regression giving the approximated values for the deviations of the thermal properties.

2.6 Experimental procedure

Following the thermal model shown in Figure 1, the experimental setup developed in this work is composed by a metallic sample of 316 stainless steel placed between a resistive heater (SRFGA-202 10 OMEGA, 332.1 Ω) and a ceramic insulator. The heater applies a constant heat flux of 18000 W/m² in the top surface of the sample ($x = 0$) while the insulator keeps the bottom surface ($x = L$) under thermal insulation boundary condition. Furthermore, ensuring a one-dimensional heat transfer, all other surfaces have also been insulated and the sample thickness is much smaller than other dimensions.

In Figure 2 is illustrated the experimental assembly used to perform each experiment. The dimensions of the sample, 50.10 mm x 50.10 mm x 9.90 mm, were measured by a vernier caliper (Mitutoyo 530-104BR, resolution: ± 0.05 mm). A precision balance (Bel S2202H, resolution: ± 0.1 g) was used to achieve the sample weight, which was used to evaluate the density, calculated to be 7986 kg/m³. The data from the experiment was obtained by a T-type thermocouple (30 AWG, resolution: ± 0.1 ºC, diameter of 0.25 mm) placed in the insulated surface by a capacitive discharge. The heat flux imposed in the upper surface of the sample is regulated by a DC power supply (IT6953A, least count: 0.001 A, and 0.001 V) connected to the resistive heater. In order to obtain the total heat flux provided to the sample, the electrical current was measured with a digital multimeter (Minipa ET-2042-C, resolution: ± 0.01 A, ± 0.1 Ω).

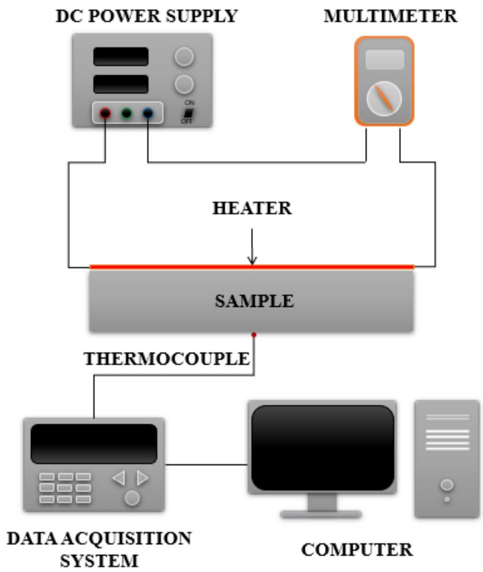

Figure 2. Schematic diagram of the experimental setup.

The surface roughness of heater and the sample were specified by a digital roughness meter (Mitutoyo SJ-210, resolution: ± 0.01 µm). Also with the precision balance, the experimental setup weight was measured in order to achieve the contact pressure between heater and sample. A hardness tester (Otto Wolpert-Werke Testor HT1, resolution ± 0.5 HB) was applied to obtain the hardness of the material. These quantities are specified in order to perform the contact conductance study in COMSOL. Besides, since the finishing of the surface has strong influence in the study, the stainless steel sample was rectified in order to decrease the effects of contact resistance.

A data acquisition system (DAQ-Keysight 34980A) was employed to collect the experimental data from the thermocouple in a frequency of 10 Hz. The temperatures were, then, registered in a computer used to perform the inverse problem. To ensure the reliability of the outcomes, 20 experiments have been performed following the procedure described.

3. RESULTS AND DISCUSSION

Information about the feasibility of the estimation is achieved by sensitivity analysis. So, in first place, this study was performed considering constant thermophysical properties to solve the direct problem and obtain the numerical temperature profile. In Figure 3 the sensitivity coefficients for thermal conductivity, X_k, and for specific heat, X_{cp}, are shown. Efforts to measure temperature in the heated surface ($x = 0$) have been made, however, the procedure increases the air gaps between heater and sample, affecting the heat conduction. Thus, sensitivity coefficients have been both calculated in $x = L$, where the thermocouple is located. One can observe that X_k has a fast growth rate and stabilizes after a few seconds, being stable all along the experiment. This behavior is similar to the heat flux applied and proportional to its intensity. On the other hand, X_{cp} is clearly proportional to the temperature variations, T, also shown in Figure 3. It is also clearly observed that the coefficients are not dependent on each other, their variation is uncorrelated and they both present reliable values during the experimental procedure, so, the quality of the estimation can be guaranteed.

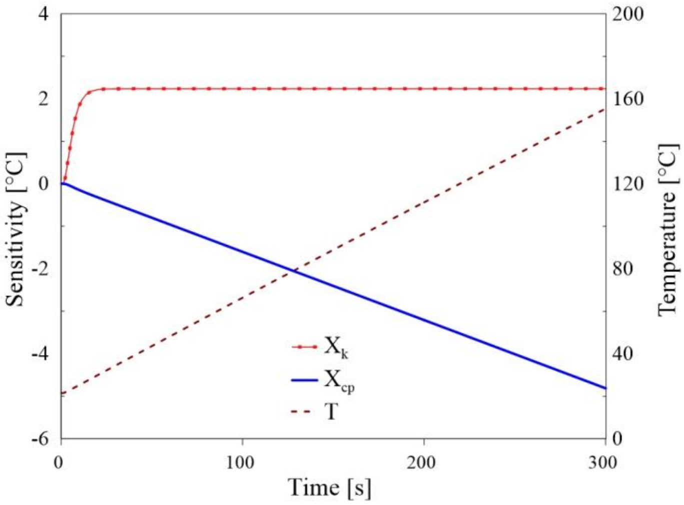

Figure 3. Sensitivity coefficients for k and c_p at $x = L$.

Also in Figure 4, the temperature profile measured along the experiment (Y) is shown in comparison to the numerical solution achieved in COMSOL (T). To evaluate the numerical temperatures, the thermophysical properties were taken from the estimation procedure. As one can observe, measured and calculated temperatures have a good agreement. The residuals are kept under 0.4 °C, which represents less than 0.3% of the maximum temperature rise. In addition, a mesh convergence study has also been accomplished to solve the numerical problem in COMSOL. Four meshes were considered with different number of elements (6, 8, 11 and 14). The first two presented some instability meanwhile the last ones provided difference no larger than 0.0006 °C. Thus, in order to achieve a lower computational cost, the 11-element mesh is employed.

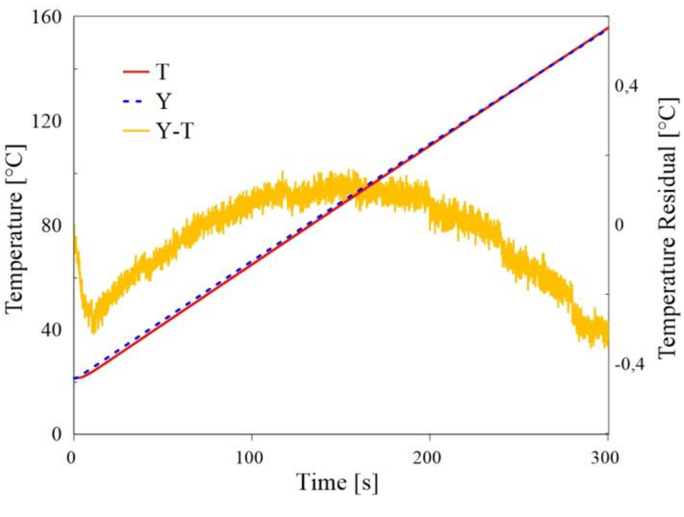

Figure 4. Compared experimental and numerical temperature profiles.

It is important to highlight that the initial guess is a key issue when it comes to the convergence of the method. After analyzing possible guesses to be applied in the algorithm, it has been defined that feasible results are provided in a range of 0.5 to 1.5 times the actual value of the parameter. So, for each time step of the estimation, the initial guesses were taken as the value of each property at room temperature.

The outcomes of the estimation procedure can be visualized in Figs. 5 and 6. One can observe that both properties present a linear trend, so the estimates are fitted by means of linear regressions. The temperature-dependence is so expressed in Eqs. (15) and (16). Also, in Figures 5 and 6, results have been compared to the 316 stainless steel properties given by Valencia and Quested (2008). The estimation process has been performed with 100 future time steps employed in the regularization. Even though, outcomes for thermal conductivity remain noisy. This behavior is due to the fact that temperature is less sensitive to k, so results for this parameter are more sensible to measurement errors than results for specific heat. Although, estimates for both properties present good agreement with literature, as observed by the minor contrast between the curves in Figs. 5 and 6.

$$k(T) = 12.76 + 0.0357 \times T \qquad \text{[W/mK]} \tag{15}$$

$$c_p(T) = 468.2 + 0.1282 \times T \qquad \text{[J/kgK]} \tag{16}$$

In Equations (15) and (16) T is temperature, measured in °C.

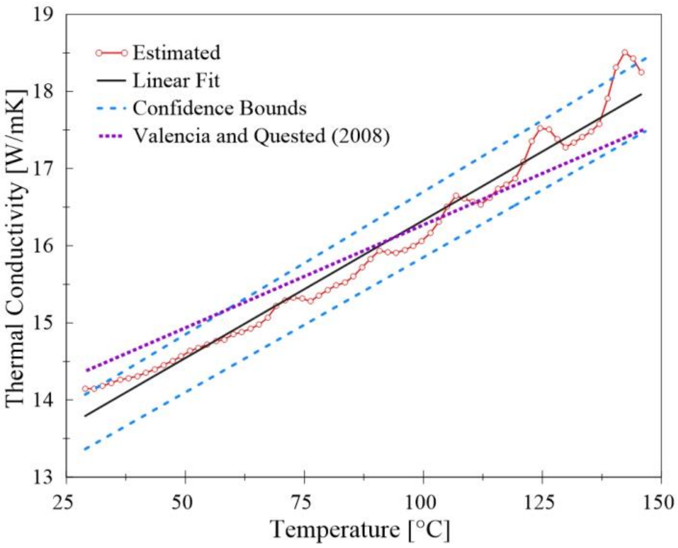

Figure 5. Estimated temperature-dependent thermal conductivity for 316 stainless steel compared to Valencia and Quested (2008).

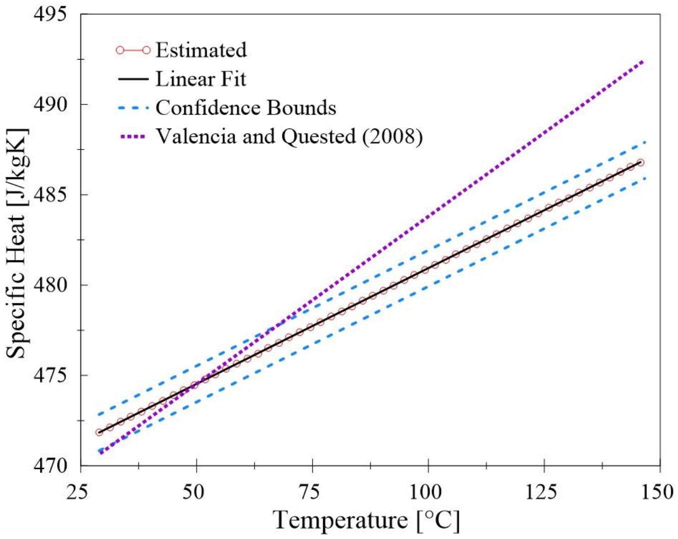

Figure 6. Estimated temperature-dependent specific heat for 316 stainless steel compared to Valencia and Quested (2008).

The quality of the fit presented in Figs. 5 and 6 may be accessed by the coefficient of determination (R-squared) close to unity for both parameters. Also, for a significance level α = 0.05, P-value is less than 0.0001, ensuring the linear dependency of thermal properties on temperature.

4. UNCERTAINTY ANALYSIS

Inherent to development of experimental and numerical methods, there are always errors associated with the procedures performed. These errors can be defined according to the uncertainty related to each procedure. The uncertainty analysis is a great ally when defining processes with the most error sources, making it possible to work on reducing their effect. Besides that, this kind of study also increases the reliability of the method.

The process presented here to calculate the uncertainty in the experimental and numerical procedure, is explained in Ramos *et al.* (2020). The first step is to identify the parts of each procedure that influence in the estimation accuracy. Then, according to the objective function described in Eq. (6), the uncertainty is achieved through the squared sum of the independent errors occasioned in the calculation of the numerical data (E_T) and in the procedure to obtain the experimental temperatures (E_Y). Moreover, the uncertainty associated with the estimation algorithm (E_{L-M}) is also taken into account, so the total uncertainty (E_{total}) can be written as:

$$E_{total} = (E_T^2 + E_Y^2 + E_{L-M}^2)^{1/2} \tag{15}$$

$$\begin{aligned} E_{total} = \big(&E_{electrical\ resistance}^2 + E_{electrical\ current}^2 + E_{roughness}^2 + E_{weight}^2 \\ &+ E_{dimensions}^2 + E_{hardness}^2 + E_{PARDISO}^2 + E_{BDF}^2 + E_{thermocouple\ \ calibration}^2 \\ &+ E_{temperature\ reading}^2 + E_{data\ acquisition}^2 + E_{thermal\ insulation}^2 + E_{L-M}^2\big)^{1/2} \end{aligned} \tag{17}$$

Each of the uncertainties reported need to be assessed. For measuring tools, the individual uncertainty is taken as their resolution. For numerical methods, it is associated with the regarded tolerances. Then, the individual uncertainties are divided by the mean value of the variable. The process differs when dealing with temperatures. In this case is taken the maximum rise.

Therefore, the total uncertainty of the method shown in the work for 316 stainless steel is:

$$\begin{aligned} E_{total} = (&9.18\text{x}10^{-6} + 1.11\text{x}10^{-1} + 7.32\text{x}10^{-2} + 2.64\text{x}10^{-5} + 1.23\text{x}10^{-4} + 1.01\text{x}10^{-4} \\ &+ 9.10\text{x}10^{-19} + 9.10\text{x}10^{-7} + 9.10\text{x}10^{-7} + 9.10\text{x}10^{-5} \\ &+ 9.10\text{x}10^{-7} + 4.68\text{x}10^{-4} + 9.10\text{x}10^{-9})^{1/2} = 4.30\% \end{aligned} \tag{18}$$

As demonstrated in Eq. (18), the uncertainty for the simultaneous estimation of k and c_p is less than 5%, ensuring the accuracy of the method proposed.

5. CONCLUSIONS

The present work has presented an affordable experimental technique to simultaneously estimate the thermal conductivity and the specific heat of metallic materials as functions of temperature. Temperature dependent thermophysical properties were determined for a sample of 316 stainless steel using information from all the measured points of the temperature field. The Levenberg-Marquardt method was applied to solve the inverse problem, minimizing the objective function defined as the squared difference of measured temperatures and numerical temperatures, evaluated in COMSOL. Both thermal conductivity and specific heat have presented standard deviation under 2% for a confidence level of 95%, besides a good agreement with literature. Moreover, the total uncertainty of the technique for the simultaneous estimation was below 5%. Thus, the inverse technique here presented has been proved to achieve reliable results for the estimation of thermal conductivity and specific heat as functions of temperature from 20°C to 130°C in metallic materials.

6. ACKNOWLEDGEMENTS

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