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Analytical evaluation of non-Fourier heat pulse experiments on room temperature

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Abstract: The classical constitutive equation for heat conduction, Fourier's law, plays an important role in the engineering practise and can be applied only for homogeneous materials. However, most of the materials consist of heterogeneity, such as porosity, cracks, or different materials are in contact.

We used heat pulse measurement method ("flash method"), a standard method in the engineering practise, to measure the thermal diffusivity of each rock sample. During the heat pulse experiment the pulse reaches the front of the sample and the temperature of the rear is measured with a thermocouple. This is a common method to determine the thermal diffusivity of a material on room temperature. We found that the rear side temperature history can deviate from the one predicted by Fourier's law. Therefore, in the evaluation of the experimental data, we used an analytical solution of a non-Fourier model, called Guyer-Krumhansl equation. We present the measurement methodology and data recorded together with the evaluation procedure.

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1. INTRODUCTION

Engineering practice requires reliable procedure to determine the necessary parameters that are enough to characterise the behaviour of a material. In the following, we focus on the thermal characterisation of materials, with particular emphasis on heterogeneous materials such as rocks. In previous publications (see Both et al. (2016) and Ván et al. (2017)), it has been reported that the presence of different heterogeneities can lead to non-Fourier thermal conduction effects due to the simultaneous presence of thermal conduction channels with different characteristic times. Such effects can be observed in the thermal pulse (so-called "flash") experiment, in which the front face of the sample is excited by a short thermal pulse and the temperature is measured on the rear side. This temperature history is used to determine the thermal diffusivity to characterise the transient behaviour of the material (see Parker et al. (1961), Fehér et al. (2020) and James (1980)).

This non-Fourier effect occurs over a specific time interval, as Figure 1 shows for a typical outcome of the flash experiments; this called over-diffusion. After that interval, the Fourier equation seems to be a suitable choice for modelling, the effect of heterogeneities disappears, furthermore there is no difference between the steady-states described by the Fourier and the non-Fourier heat equation, only the transient evolution of temperature differs in these cases. Our experimental experience suggests that the existence

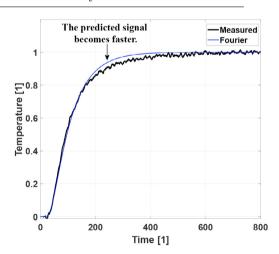


Fig. 1. Measured rear side temperature history for the rock sample and the prediction provided by Fourier's theory.

of over-diffusion depends on a number of factors, such as the sample thickness, the typical parallel time scales and the excitation (i.e. boundary conditions) (see Fülöp et al. (2018)).

The evaluation procedure for flash experiments with non-Fourier heat equations is not yet standard. Even nu-

merically, finding solutions to non-Fourier models is not straightforward, some methods may lead to incorrect solutions, and commercial built-in algorithms are not efficient for these models (see Rieth, Kovács and Fülöp (2018)).

Although there are several generalisations of Fourier's law in the literature (Ván (2016), Müller and Ruggeri (2009), Cimmeli (2009), Joseph and Preziosi (1989), Sobolev (2014) and Sobolev (1997)), there is only one of them that has been shown to be reasonable beyond Fourier theory, called the Guyer-Krumhansl (GK) equation (Parker et al. (1961), Guyer and Krumhansl (1966)). The constitutive equation reads in one spatial dimension

$$\tau_a \partial_t q + q + \lambda \partial_x T - \kappa^2 \partial_{xx} q = 0. \tag{1}$$

In the equation, τ_q is the relaxation time of the heat flux q and κ^2 is a kind of 'dissipation parameter', usually related to the free path in kinetic theory. The $\partial_{xx}q$ in the constitutive equation allows to properly characterize the so-called over-diffusive propagation, presented in Figure 1

We call it Fourier resonance condition as that setting recovers the solutions of Fourier equation, when the equality $\kappa^2/\tau_q=\alpha$ holds (Ván et al. (2017) and Fülöp, Kovács and Ván (2015)). Equation 1 is the time evolution equation for the heat flux, and to have a mathematically and physically complete system we need the balance of the internal energy e.

$$\rho c \partial_t T + \partial_x q = 0. (2)$$

in which the equation e=cT is used, where c is the specific heat and ρ is the mass density. All these coefficients are constant, we assume only rigid bodies without a volumetric heat source.

Figure 2 shows the typical rear side solutions for the Fourier, MCV and GK models. It can be seen that while the MCV equation gives sharp waveforms for the solution, the GK equation gives a significantly better match with the measured data. Therefore, the GK equation appears to be a necessary extension of the Fourier equation that covers practical needs to determine the thermal properties of heterogeneous materials as accurately as possible.

2. HEAT PULSE EXPERIMENT

The heat pulse experiments are used to measure the thermal diffusivity of a material on room temperature by registering the rear side temperature history. For the specified settings, see Figure 3. The front surface of a sample is painted black for better absorption, and the rear side is coated with silver in order to achieve good contact with the thermocouple. A flash lamp generates the heat pulse; its length is 0.01 seconds. For further details, we refer to the earlier papers published by our group Both et al. (2016), Ván et al. (2017), Fülöp et al. (2018) and Fülöp, Kovács and Ván (2015).

3. EVALUATION METHOD

The dimensionless set of parameters which was used in the evaluation methods can be found in Both et al. (2016).

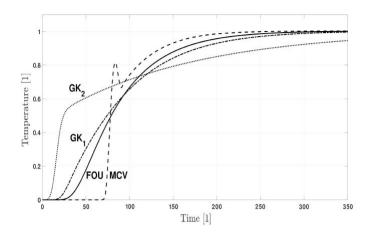


Fig. 2. Typical rear side temperature histories for the Fourier ("FOU"), Maxwell-Cattaneo-Vernotte ("MCV") and Guyer-Krumhansl ("GK1" - slightly over-diffusive and "GK2" - strongly over-diffusive) equations.

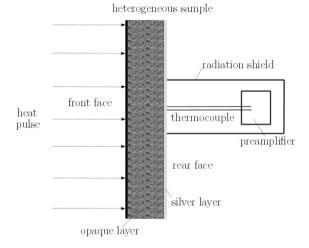


Fig. 3. Shematic figure of the heat experiments.

3.1 Evaluation with the Fourier-theory

The analytical solution of the Fourier equation $(q = -\lambda \nabla T)$ for the rear side is

$$T(x = 1, t) = Y_0 exp(-ht) - Y_1 exp(x_F t),$$

$$x_F = -2h - \alpha_F \pi^2, t > 30$$
(3)

which is analogous with the classical known 'one-term solution' see in Fehér et al. (2021). Equation 3 is only the first term of an infinite series, after t > 30, it provides solution with enough precision (as further terms converges to zero much faster) (Fehér and Kovács (2021)). First, the heat transfer coefficient h is estimated by selecting two temperature values (T_1 and T_2 , see Figure 4) at the decreasing part of temperature history and reading the corresponding time instants (t_1 and t_2). In this region $\exp(x_F t) \approx 0$,

$$h = -\frac{\ln(T_2/T_1)}{t_2 - t_1}. (4)$$

While h can be determined from the rear side temperature history, it should be noted that the sample is small and as long as $h \cdot A_h$ is constant (A_h) is the surface area where

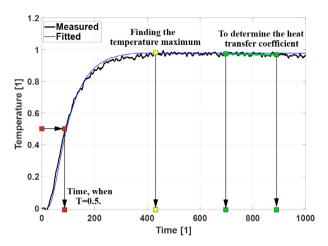


Fig. 4. Schematic representation of the Fourier evaluation method. The green section shows the determination of the heat transfer coefficient, the red squares show the meaning of $t_{(1/2)}$ and the yellow squares show the location of the maximum temperature.

heat transfer occurs), it is not necessary to determine a heat transfer coefficient for all surface. In Fourier theory, the thermal diffusivity can be expressed explicitly, i.e.

$$\alpha_F = 1.38 \cdot \frac{L^2}{\pi^2 t_{1/2}}.\tag{5}$$

and can be determined immediately after reading $t_{(1/2)}$ (Fehér and Kovács (2021), Fehér et al. (2021)). The thermal diffusivity is the ratio of the thermal conductivity λ and the specific heat capacity ρc . Then comes the maximum of the temperature history (T_{max}) , where the moment of time t_{max} can be read when T_{max} occurs. This gives the heat transfer coefficients, the thermal diffusivity and T_{max} , which are used in the Guyer-Krumhansl theory.

3.2 Evaluation with the Guyer-Krumhansl theory

The situation becomes more difficult in the Guyer-Krumhansl case (Equation 1), because in contrast to the Fourier theory, it consists of two 'time constants' (x_1 and x_2) instead of one (x_F in Fourier theory). Consequently, it is not easy to find these constants. To check the effect of the simplifications made in the following, a parameter analysis was carried out for all possible τ_q and κ^2 values that may be possible in practice and beyond. However, beyond these, we always had to restrict ourselves to a region $3 > \kappa^2/(\alpha \tau_q) \ge 1$. The lower limit expresses the Fourier case, and all other combinations fall in the overdiffusive region. The highest ratio observed in the experiments so far has been around 2.5, so a value of 3 should be appropriate. For κ^2 , the region is $0.02 < \kappa^2 < 1$. We would like to emphasize that the GK theory allows the underdiffusive ('wave-like') case also. In the GK theory, the rear side temperature history can be expressed as follows

$$T(x = 1, t > 40) = Y_0 exp(-ht) - Z_1 exp(x_1 t) - Z_2 exp(x_2 t), x_1, x_2 < 0,$$
(6)

which is also a 'one-term solution'. For the detailed calculation, parameter, boundary condition definitions, convergence properties we refer to the Fehér and Kovács (2021).

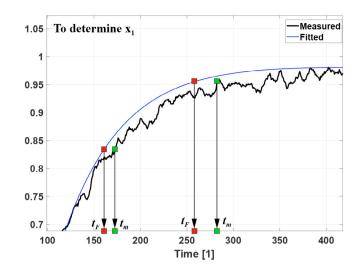


Fig. 5. The schematic representation of the evaluation method using the Guyer-Krumhansl theory, how to determine x_2 . Here, the 'fitted curve' belongs to the Fourier equation.

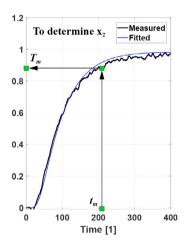


Fig. 6. The schematic representation of the evaluation method using the Guyer-Krumhansl theory, how to determine x_2 . Here, the 'fitted curve' belongs to the Fourier equation.

In the following, we present a step-by-step approach (Fehér and Kovács (2021)) to the determination of the GK parameters, which is illustrated in Figure 5 and 6.

• Step 1/A. It can be observed (Figure 1) that the temperature predicted by Fourier's theory is close to the measured temperature at the beginning of the process, then rises more rapidly as it approaches the maximum. In other words, the same temperature is reached more quickly in this region with Fourier's theory (usually around 0.7-0.95). Mathematically, this can be expressed by formally writing the equations of Fourier and GK theory as follows where the fraction on the right is close to 1. It would be possible to introduce a correction factor for x in the iteration procedure if we knew more about τ_q and κ^2 . After rearrangement, we obtain the following closed formula for x_1 :



Fig. 7. Szarsomlyo Limestone Formation sample (left) and Szaszvar Limestone Formation sample (right).

$$x_1 = x_F \frac{t_{F1} - t_{F2}}{t_{m1} - t_{m2}}. (7)$$

• Step 1/B. In parallel with part A, we can determine the coefficient Z_1 for each t_m and each corresponding $x_{1,m}$,

$$Z_{1,m} = -exp(-x_{1,m}t_m)(T_m - Y_0exp(-ht_m))$$
 (8)

where the subscript m represents the value for a measurement point. After 20 subsequent measurement points, the average value of the set $\{Z_{1,m}\}$ is taken

• Step 2. In this step, to obtain x_2 we rearranged the following equation

$$T = Y_0 exp(-ht) - Z_1 exp(x_1t) - Z_2 exp(x_2t), \quad (9)$$

to x_2 and calculate the average of the values for each t_m giving the average value of the set $\{x_{2,m}\}$. Unfortunately, for noisy data, this approach can result in positive x_2 values. These values must be excluded, otherwise they may lead to instability and meaningless results. Careful filtering of the data can help to solve this problem and in fact we use this to facilitate the calculation.

- Step 3. Now that we have both exponents and coefficients, the analytical expressions can be explicitly rearrange to the GK parameters and the α_{GK} , τ_q and κ^2 values can be calculated.
- Step 4. Each parameter of the temperature history can be characterized with R^2 , the coefficient of determination, which represents the value for the fitted curve with fine-tuned α_F .

In practice, this evaluation method reduces the number of 'fitted' parameters. Moreover, it is limited to a relatively narrow range, i.e. the evaluation procedure takes only a few seconds instead of running computationally expensive algorithms that take hours.

4. NON-FOURIER THERMAL BEHAVIOUR

The evaluation procedure described above is used on several rock samples, the results of which are shown below. The rock samples have the same cylinder geometry, but all geologocally different. It is noted that the samples are manufactured by Kőmérő Ltd. as the necessary infrastructure and technology is available there. The thin 1.9 mm thick rock sample is challenging to produce due to, for example, its fragility, so only one of each thickness of rock is available.

	α_F	α_{GK}	$ au_q$	κ^2
	$10^{-6} \ [\mathrm{m^2/s}]$	$10^{-6} \ [\mathrm{m^2/s}]$	[s]	$10^{-7} [m^2]$
2 mm	0.5113	0.5812	0.4180	0.0835
$2.15~\mathrm{mm}$	1.1186	1.0254	0.4010	0.1127
$2.85~\mathrm{mm}$	1.1413	-	-	-
$3.85~\mathrm{mm}$	1.1197	-	-	-

Table 1. Summary of fitted thermal parameters of the Szársomlyó limestone formation.

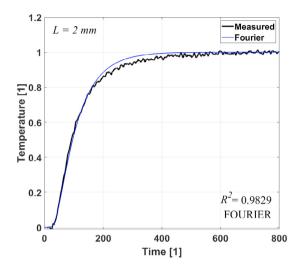


Fig. 8. Fourier evaluation for Szarsomlyo limestone formation L=2 mm sample.

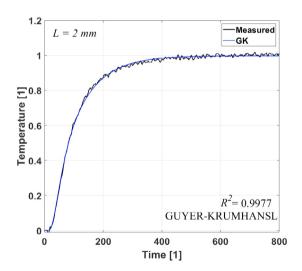


Fig. 9. GK evaluation for Szarsomlyo limestone formation L=2 mm sample.

4.1 Szarsomlyo limestone formation

The results obtained from the evaluation for the Szársomlyó limestone formation are shown in Table 1. The non-Fourier effect is observed in the thinner samples, and as it increases, the Fourier theory fits the tempearture history well. It is clearly seen that the thermal diffusivity of the sample increases with increasing thickness until the non-Fourier effect is eliminated. Then, in essence, the thermal diffusivity becomes independent of the thickness. The evaluations are shown in Figures 10-13.

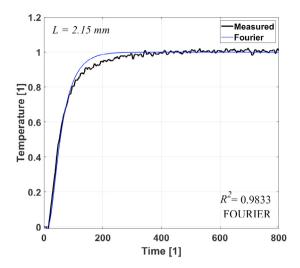


Fig. 10. Fourier evaluation for Szarsomlyo limestone formation L=2.15 mm sample.

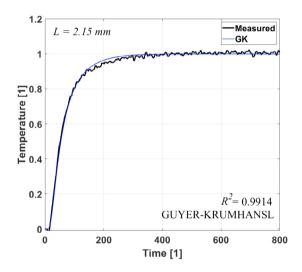


Fig. 11. GK evaluation for Szarsomlyo limestone formation L=2.15 mm sample.

	α_F	α_{GK}	$ au_q$	κ^2
	$10^{-6} \ [\mathrm{m^2/s}]$	$10^{-6} \ [\mathrm{m^2/s}]$	[s]	$10^{-7} [m^2]$
3.05 mm	1.4048	1.4505	0.4273	0.0815
3.8 mm	0.8598	0.8815	0.5229	0.0409
$3.9~\mathrm{mm}$	1.251	-	-	-

Table 2. Summary of fitted thermal parameters of the Szászvár limestone formation.

4.2 Szászvár limestone formation

In the case of the Szászvár Limestone Formation, the results of the evaluation are shown in Table 2.

In the case of the rocks, a similar phenomenon can be observed as in the case of the Szársomlyó sample. On thinner samples, a non-Fourier effect is observed, which disappears with increasing sample thickness and the Fourier equation models our rock sample well. The thermal diffusivity of the samples shows first a decrease and then an increase. The decrease is observed for the sample with a weak GK-

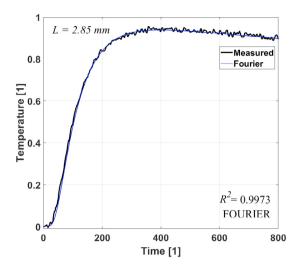


Fig. 12. Fourier evaluation for Szarsomlyo limestone formation $L=2.85~\mathrm{mm}$ sample.

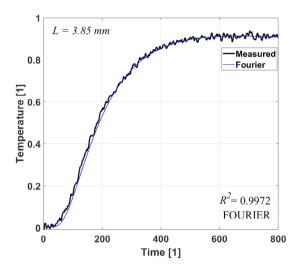


Fig. 13. Fourier evaluation for Szarsomlyo limestone formation L=3.85 mm sample.

effect, and then the thermal diffusivity increases again as the non-Fourier effect disappears.

5. CONCLUSION

We develop an algorithm to efficiently evaluate room temperature heat pulse experiments in which a non-Fourier effect could exist. This is called over-diffusive propagation and detunes the thermal diffusivity, even when the deviation is seemingly small or negligible for the rear side temperature history. The presented method is based on the analytical solution of the Guyer-Krumhansl equation, including temperature-dependent convection boundary condition, thus the heat transfer to the environment can be immediately included in the analysis.

We believe that this procedure lays the foundations for the more practical engineering applications of non-Fourier models, especially for the best candidate among all of them, the Guyer-Krumhansl equation. It sheds new light on the classical and well-known flash experiments, and we provide the necessary tools to find additional thermal parametres to achieve a better description of heterogeneous materials. It becomes increasingly important with the spreading of composites and foams and helps characterize 3D printed samples with inclusions. With continuing the experimnets, our goal is to find a relationship between the non-Fourier coeficients and the material structure. For instance, we aim to analyze multiple foam samples with different inclusion sizes, expectedly connecting production parameters to the non-Fourier effects.

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