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A composite layered sub-system for front detection calorimetric instrumentation



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ABSTRACT

A stacked three layer monolithic sub-system comprising a pyroelectric (PE) sensor, a thermal coupling layer and an external plate (EP), intended to detain a rigid, corrosion resistant external surface and to be used in front detection configuration for liquid samples' thermal parameters measurement is proposed herein. The thermal waves (TWs) are generated on the irradiated surface of the PE material and after propagating through the sensor and the coupling layer reach the EP. The EP, positioned in contact with the liquid sample is intended to act as a buffer layer (protective against mechanical and corrosion agents) while successfully transmitting the probing TWs to the investigated semi-infinite sample. The normalized signal can be described by means of an analytical model obtained by assuming one-dimensional TWs propagation through the five layered system: air/proposed sub-system and backing liquid sample. The theoretical signal is firstly analyzed by studying the numerical behaviour of the normalized amplitude and phase and the behaviour of the sensitivity coefficients specific to these components when the thermal effusivity of a semi-infinite liquid is aimed to be extracted. The experimental results obtained by performing frequency scanning measurements using a composite sub-system with a brass EP and different backing sample liquids are presented.

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1. Introduction

Calorimetric methods that use thermal waves (TWs) in order to extract the thermal parameters of a sample material have been developed as an alternative to classical methods [1,2]. The photothermal (PT) techniques assume the generation of the TWs by chopped (periodically modulated) light irradiation. These methods are characterized by an efficient thermal and mechanical isolation between the excitation system and the measurement cell. Because the irradiated surface can be controlled, in order to obtain a heated region easily to approximate with a planar source, a one-dimensional (1-D) heat diffusion model usually describes the thermal field distribution into the measurement cell (a multilayered system containing also the investigated sample [3]). PT based calorimetric methods have been used for the measurement of one or two of the four material specific thermal parameters: heat capacity, thermal conductivity, thermal diffusivity and thermal effusivity, two of these material properties being independent.

Photopyroelectric (PPE) calorimetric methods, a sub-class of PT methods make use of a pyroelectric (PE) material, in order to quantitatively evaluate the TWs' propagation through the measurement cell. These techniques proved to be an effective tool for in situ measurements [4]. PPE methods have been mostly developed in order to investigate fluid samples: both liquids [5,6] and gases [7,8] since in this case the usage of a supplementary coupling layer is not required. There are two general types of PPE calorimetric measurement configurations [9], compatible with 1-D heat diffusion approximation: a front photopyroelectric (FPPE) one which presumes irradiation of the PE sensor which then feeds heat diffusively to the other layers and a back detection configuration (BPPE) which presumes irradiation of a light absorbing material, diffusive heat propagation through the sample material and detection of the transmitted TWs by the PE sensor [10,11]. The FPPE measurement configuration provides some advantages over the BPPE one, the most important of these being: **i.** there is a frequency domain for which the signal to noise ratio (SNR) has a high value, independent of the number or the thicknesses of the stacked layers, **ii.** this configuration is fitted for TWs probing of a semi-infinite medium, **iii.** the opaque PPE sensor acts as a separation layer between the modulated light excitation and the rest of the

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Nomenclature

A_{N_in}	initial normalized theoretical amplitude vector	N	total number of experimental data
A_{N_fin}	final normalized theoretical amplitude vector	R_{kl}	TWs' reflection coefficient at two successive layers' interface in case of the known configuration; $(k,l) \in \{(a,1), (1,2), (3,4)\}$
b_{kl}	ratio between the thermal effusivity of the k th and l th consecutive layers	$Sens_{\varphi}$	phase specific sensitivity coefficient [°]
e_k	thermal effusivity of the k th layer [$W s^{1/2} m^{-2} K^{-1}$]	$Sens_A$	amplitude specific sensitivity coefficient [°]
e_{4_in}	initial value of the thermal effusivity [$W s^{1/2} m^{-2} K^{-1}$]	V_n	theoretical FPPE normalized signal
e_{4_fin}	final value of the thermal effusivity [$W s^{1/2} m^{-2} K^{-1}$]	V_m^{exp}	experimental data point defined by the m index
e_{4_change}	thermal effusivity variation [$W s^{1/2} m^{-2} K^{-1}$]	V_m^{fit}	theoretical data point defined by the m index
e_{4fit}	best fit value of the sample's thermal effusivity [$W s^{1/2} m^{-2} K^{-1}$]	x_k	physical to thermal thickness ratio of the k th layer
Err_{s4}	thermal effusivity fit error calculated from amplitude or phase data; $s = [A, \varphi]$	<i>Greek letters:</i>	
EF_{tot}	thermal effusivity fit error calculated from both amplitude or phase data	α_k	thermal diffusivity of the k th layer [$m^2 s^{-1}$]
f	modulation frequency [Hz]	φ_{N_in}	initial theoretical normalized phase vector [°]
j	imaginary unit ($j = \sqrt{-1}$)	φ_{N_fin}	final theoretical normalized phase vector [°]
L_k	physical thickness of the k th layer [m]	μ_k	thermal thickness [m]
		ρ_{kl}	TWs' reflection coefficient at two successive layers' interface in case of the configuration containing the investigated layer

measurement cell. In case of the FPPE configuration, an analytical model that resumes to a recurrent description of the normalized signal, by means of a set of complex TWs reflection coefficients [12] has already been developed. This model can be extended for an infinite number of layers (as long as 1-D heat diffusion approximation is applicable). The FPPE measurement configuration has been employed in order to extract the thermal effusivity of liquids [13–15] and both the thermal diffusivity and effusivity of liquids, either by using a self-consistent technique [16] or by performing combined thickness/frequency scanning procedures [17].

The PPE technique is a contact one that requires direct interfacing between the sensor and the investigated system. Pyroelectric sensors are in many cases fragile (monocrystalline and polycrystalline materials) or are flexible materials with little rigidity (polymeric materials), when the surface flatness of the sensitive element, and thus the one-dimensionality of the mathematical model is difficult to maintain. The metallic electrodes and the electrical junctions between the metallic electrodes and the signal carrying conductors are usually prone to suffer time alteration, because of external chemical or physical agents that gradually worsen the SNR. These insufficiencies of the classical PE sensors have partly restrained the usage of PPE methods for laboratory applications, in which case, periodic reconditioning or replacement of the PPE detectors and/or the renewal of the electrical junctions has been performed. In order to overcome these issues a BPPE configuration that presumes the placement of a protective plate on the surface of the PPE sensor, as a buffer layer between the detector and the investigated sample (while the electrical junction has been made by adding a thin metallic layer to the buffer layer's surface closest to the PPE sensor) has been reported recently [18,19]. In order to extract either the thermal diffusivity or the optical absorption coefficient specific to the investigated liquid, a thickness scanning procedure was employed.

A stacked three layer monolithic (permanently joint together) sub-system, aimed to be used in PPE configuration, as an alternative to conventional PE sensors is described herein. The proposed sub-system is in fact a three layer system: directly irradiated PE sensor, thermally coupling adhesive layer and an external plate (EP). This configuration of stacked layers is meant as a planar PPE sensing device for which the signal collecting electrical junctions are placed on one side (the irradiated one), while the surface of the EP is meant to be contacting the investigated liquid. If the EP is a

thin electrically insulator layer then total insulation of the PPE signal can be achieved, hence the signal can be rendered immune to otherwise perturbative chemical phenomena. The composite sub-system analyzed herein is meant to be integrated in a PPE measurement device [20] for thermal parameters measurement usable on-field. In the following, a FPPE composite sub-system with a brass EP will be described along with the analytical model referring to the 1-D TWs' diffusion through the measurement cell, applicable to the measurement of a semi-infinite liquid's thermal effusivity. The numerical behaviour of the normalized phase and amplitude is presented. A comparison between the behaviour of the composite sub-system and that of a simple PPE sensor has been performed using sensitivity coefficients. Experimental results obtained for different semi-infinite sample liquids, with a brass EP sub-system is presented and discussed.

2. Theoretical expressions of the analytical model associated with the multilayered configuration

The proposed sub-system necessitates compliance with the main requirement to detain a rigid EP, thick enough to ensure a long-lasting corrosion and mechanical wear resistance. The surface contacting the investigated liquid is required to be as smooth and as plain as possible, in order to comply with 1-D heat diffusion approximation. A three layered topology (PE material with deposited metallic electrodes), thermally coupling layer and external plate is analyzed in the following. The thermally coupling layer is also necessary in order to obtain a good electrical contact and a good mechanical bonding.

For a certain modulation frequency range, a layered structure can be described by means of a 1-D heat diffusion model if the thermal field's distribution is not influenced by any boundary effects that may alter the TWs' propagation and thus induce a difference in comparison with the ideal case when all the stacked layers are infinite along the two axes perpendicular to the layered measurement cell [3,21]. Assuming the 1-D approximation, the proposed layered sub-system is schematically represented in Fig. 1.

In the simplest case, the sample liquid is a semi-infinite (backing) layer, contacting the surface of the EP. In order to estimate the thermal behaviour of the proposed sub-system, a study of the mathematical model associated with 1-D TWs' diffusion through the stacked structure can be performed. The PPE

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