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LASER INDUCED TRANSIENT PHOTOTHERMAL RADIOMETRY IN OPAQUE SAMPLES

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LASER INDUCED TRANSIENT PHOTOTHERMAL RADIOMETRY IN OPAQUE SAMPLES

By

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TABLE OF CONTENTS

Page

APPENDIX

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

a	absorbance
a	radius of gaussian beam
B_o	bulk modulus (N/m^2)
c	velocity in free space (m/s)
C_V	heat capacity at constant volume (J/mol/K)
C_P	specific heat at constant pressure
D_m	minority carrier diffusion coefficient (m^2/s)
D	distance from the sample to the detector (m)
$h\nu$	photon energy (J)
e	dielectric constant ($1.602177\ 10^{-19}$ C)
E_G	semiconductor gap energy (J) (eV)
$g(r,t; r't')$	Green function
I_m	maximum incident irradiance
k	imaginary part of index of refraction, extinsion coefficient
k_B	Boltzmann's constant ($1.380658\ 10^{-23}$ J/K)
K	thermal conductivity (W/m/K)
K_L	lattice thermal conductivity (W/m/K)
K_e	electronic thermal conductivity (W/mK)
K_r	radiation thermal conductivity (W/mK)
J	heat flux ($J/m^2/s$)
l_r	mean free path
s	surface recombination velocity (m/s)
N_A	Avogadro's number
n	real part of index of refraction
r	reflectance
R	reciprocal thermal conductivity value
$r_{1,2}$	radius of the beam spot (m)
T	absolute temperature (K)
T_∞	ambient temperature (K)
$u(r,t)$	transient surface displacement (m)
V	molar volume (m^3/mol)
$w_{1,2}$	laser beam waist (m)
α_{th}	thermal expansion coefficient (ppm/K)
β	absorption coefficient for thermal radiation (m^{-1})
$\delta(t)$	Dirac delta function
γ_G	Gruneisen parameter
ε	effusivity
ε_s	surface emissivity

ε_λ	spectral emittance
ε_t	total emittance
σ	electrical conductivity
μ_o	permeability of the free space (N/A^2)
μ	relative permeability
ρ	specific resistance (Ω)
ρ	density (Kg/m^3)
ν	Poisson ratio
τ_l	minority carrier lifetime (sec.)
τ_α	opto-thermal decay time (sec.)
τ_p	pulse duration (sec.)
σ	Stefan's constant ($5.67 \cdot 10^{-12}\ W/cm^2/K^4$)
$\sigma_{r,z}, \sigma_{zz}$	normal stress components
$\phi(t)$	light flux
γ	detector constant
γ	deflection angle (rad)
Ψ	Love function
rms	square root of a sum of squares of the data values divided by the number of points
AD	analog to digital
CMT	Cadmium Mercury Telluride
CCD	Charge-Coupled Device
EMATs	electro-magnetic acoustic transducers
IR	infrared
NDE	non-destructive evaluation
OBD	optical beam deflection
OS	optical system
PA	photoacoustic
PT	photothermal
PDT	photothermal deflection technique
TPTD	transient photothermal displacement
TEM_{oo}	laser transversal mode
PTR	photothermal transient radiometry
TL	thermal lensing
PBRS	probe beam referential system
HgCdTe	cadmium mercury telluride
NEP	noise equivalent power

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Doctor of Philosophy.

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IONEL - VALERIU GROZESCU

February 1998

Chairman : **Associate Professor Mohd. Maarof Moksin, Ph.D**

Faculty : **Faculty of Science and Environmental Studies,**
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A radiometer system has been designed and constructed for measurements of optical, thermal and related properties of two distinct categories of opaque samples. It was used exclusively for highly light absorbing and/or diffusing samples (protective coatings and ceramics) and highly reflecting samples (metals and its alloys).

In the measurements, the signals were initially generated by localised pulsed laser heating of the sample. The signals were then monitored directly by detecting changes in infrared radiation or indirectly by measuring the deflection of the reflected probe beam.

The underlying theory behind the technique and the design criteria for the radiometer are discussed. The theory has been described for the case of direct signal detection and as well as for the case of indirect signal detection.

The performance of each type of detection was duly evaluated and found that the indirect detection displayed much higher signal-to-noise ratio for the respective intended application.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia bagi memenuhi syarat untuk ijazah Doktor Falsafah.

RADIOMETRI FOTOTERMA FANA TERARUH LASER DALAM SAMPEL LEGAP

Oleh

IONEL VALERIU GROZESCU

Februari 1997

Pengerusi: **Profesor Madya Mohd. Maarof Moksin, Ph.D**

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Satu sistem radiometer telah direkabentuk dan dibina untuk pengukuran sifat-sifat optik, terma dan yang berkaitan dengannya bagi dua kategori bahan legap yang nyata berbeza.Ia telah digunakan secara eksklusif untuk bahan yang sangat menyerap dan/atau membaur cahaya (lapisan perlindungan dan seramik) dan bahan yang sangat memantulkan cahaya (logam dan aloinya).

CHAPTER I

INTRODUCTION

A survey of the scientific literature of last five years shows an increasingly interest in non-destructive and non-contact analysis techniques. Besides the traditional spectroscopies, photoacoustic and photothermal phenomena-based techniques play an important role in the field of materials characterisation.

The world-wide search for improved technological materials has produced a large variety of new composite structures. Most of them are opaque and/or diffusing for optical radiation and therefore inaccessible by conventional optical analysis methods. This determines the necessity and inevitability of using new techniques to evaluate their thermal, optical and its associated properties.

Although a large variety of new applications of photothermal (PT) and photoacoustic (PA) techniques are being reported in specific publications, the field is still not completely understood, particularly in the case of the PA and PT phenomena induced by short pulse heating sources. This is mainly due to the mathematical difficulties involved in taking into consideration the large bandwidth of the pulsed heating source in modelling the temperature distribution induced into the sample immediately after heating.

The goal of this work is to reduce the existing gap in the field of pulsed PT phenomena by studying the case of opaque materials of two extreme cases: i.e. highly absorbing/diffusing and highly reflecting samples.

Since a comprehensive analysis of all existing materials and various pulse heating sources will takes years to complete, we are restricting ourselves to the set of following objectives:

i) To determine the 3-D temperature field in a bulk opaque sample subsequent to the pulsed laser heating of the surface, considering that the transient temperature distribution represents the basis of any pulsed photothermal technique.

ii) To design and construct a radiometer system able to perform non-destructive and noncontact thermal waves analysis on highly absorbing and/or diffusing opaque materials.

iii) To determine the suitability of the radiometer system by investigating paint coatings and ceramic composites of present interest due to their industrial applications. Paint coatings are used as protective agent for metallic and non-metallic elements against the environment aggression. Ceramic composites such as silicon carbide and silicon nitride because of their relatively light weight (density 2.83-3.18 Kg/m^3 for SiC and 2.99-3.26 Kg/m^3 for Si_3N_4), excellent oxidation and thermal shock resistance and good high temperature strength are suitable for the engine hot-section components and heat exchangers. Boron carbide ceramic, due to its high neutron capture cross section, is a good candidate in nuclear power engineering as a neutron shield.

iv) To establish the thermoelastic surface displacement response based on induced temperature distribution for the particular case of pulsed laser heating.

v) To design and construct a new sensitive instrument for evaluation of the highly reflective opaque materials based on pump and probe technique for the pulse regime.

vi) To verify the validity of the theoretical model developed for transient photothermal displacement radiometry by using data from test samples of known mechanical and thermal properties.

In Chapter II, the principles for direct and indirect detection photothermal transient radiometry techniques are presented. The literature review on related work is also included.

Chapter III is dedicated to the theory of TPTR including the mechanism of the heat transfer in solids, heat conduction equation, calculated temperature profile for thermally thick and layered samples, and thermal displacement of the surface due to pulsed laser heating.

Chapter IV presents the experimental design used in transient photothermal radiometry. For highly laser absorbing materials, thermal waves generation and collection systems has been described. Some methods for noise reduction and suppression were analysed. For the case of transient photothermal displacement technique, pump and probe beam positioning system and position sensitive detection method have been presented in detail.

Experimental techniques i.e. direct signal detection with a wideband infrared detector, indirect signal detection using pump-probe measurements, signal amplitude estimate, effect of noise on device sensitivity, effect of the probe beam misalignment on the TPTD signal and sample surface requirement have been discussed in Chapter V.

Chapter Vi presents the results of the investigation of highly absorbing samples. In the case of black and metallic graphite coatings on mild steel, the results revealed that the predicted linear dependence of the decay time on coating thickness is valid up to a certain limit. The ratio of the thermal diffusivities of black and metallic paint was found to be 0.83. The analysis of the test samples of the black paint on glass fiber phenolic resin composite shows a value of 2.46×10^{-7} m^2/s for the thermal diffusivity. For the SiC/B_4C ceramic composites the effect of the B_4C content on the transient photothermal signal has been studied. In order to demonstrate the feasibility of the pulsed photothermal displacement radiometry, two types of highly reflecting solid samples were tested: high purity metals (gold silver and copper) and highly reflecting alloys (aluminum, brass, and stainless steel). Pulsed induced photothermal signals for highly reflecting samples were in good agreement with the developed theoretical model.

The summary of the results and future developments of Photothermal Transient Radiometry are discussed in Chapter VII.

It is hoped that this work, beside of having an importance in consolidating the knowledge in the photoacoustics and photothermal field, particularly in transient phenomena, will also create new direction and further development.

CHAPTER II

LITERATURE REVIEW

Photothermal radiometry is a technique whose information carrier is the induced degraded heat wave in the sample. The heat is first deposited on the sample by using a CW modulation or pulsed excitation. The change in the sample temperature is then directly detected, for example by a wideband infrared detector or indirectly detected by using a probe beam as in this work. The optical absorption of the radiation and the interaction of the heat waves with the thermal properties of the sample affect the surface temperature and hence, the emitted infrared radiation and the surface profile. Therefore, the photothermal signals as obtained from either direct detection of the grey body radiation or indirect measurement of the surface deformation can be analysed to extract the properties of the sample.

Direct Detection Photothermal Transient Radiometry

Introduction

The principle of photothermal transient radiometry (PTR) is the following. A pulsed laser excitation is used to produce localised transient heating of the material being studied. The subsequent thermal emission transient from the region of the excitation is directly measured by using a wideband infrared detection. The time dependence of the decay of the grey body emission signal can be used to measure the properties of the materials.

The temporal shape of this thermal emission transient from the homogeneous semi-infinite sample depends upon (see Chapter III):

a) the penetration of the excitation radiation into the sample;

b) the thermal diffusivity of the materials, and

c) the transparency of the sample to the emitted thermal infrared radiation.

If two of the above properties are known, or remain constant throughout a series of measurements, then the third property or the change in that property can be measured.

Penetration of the excitation radiation

When a semi-infinite homogeneous sample is excited by an instantaneous and uniform flux of radiation, $E_o\delta(t)$, the energy density absorbed within the sample at initial time (t = 0) is:

$$E(z)=E_o\alpha(\lambda)\exp(-\alpha(\lambda)z) \tag{2.1}$$

where E_o is energy absorbed per unit area in the sample, z is depth into the sample, $\alpha(\lambda)$ is absorption coefficient, λ is wavelength of the excitation, and $\delta(t)$ is Dirac delta function.

Since the absorption of the radiation is wavelength-dependent, the penetration depth of excitation also has similar characteristic.

The initial induced temperature rise at t=0 within the sample, in absence of any nonlinear effects, is given by :

$$\theta(z)=\theta_o\exp(-\alpha(\lambda)z) \tag{2.2}$$

where θ_o is the initial temperature rise at the sample surface.

The temperature change, in turn, produces a modification in the grey body heat flux leaving the sample which is also dependent on the excitation wavelength.

It can also be inferred that any change in the temporal shape of the thermal transient signal due to the variation of the excitation wavelength can only be attributed to the selective absorption of the excitation (variation of absorption coefficient with excitation wavelength). This feature offers the possibility of measuring absorption spectra of opaque samples.

Thermal Diffusivity

As shown in Chapter V, subsection Signal Amplitude Measurement of this work the recorded thermal transient signal can be parameterised by using the decay times. The decay time is inversely dependent on the absorption coefficient of excitation (or the absorption coefficient of the emitted infrared signal) and thermal diffusivity of the sample. This property gives the opportunity to monitor any change in the thermal diffusivity of the sample due to, for example, environmental exposure or operation at high temperatures.

By choosing a suitable excitation and infrared wavelengths, variation in thermal diffusivity as a function of depth below the surface can also be explored

Infrared Transparency

Equation (2.1) shows that the excitation radiation penetration into the sample is wavelength-dependent. Thermal waves generated below the surface are highly