Thermal Characterization of Clay Roof Tile Using Photothermal Deflection Technique

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Abstract. In this research, a non-destructive, simple and rapid method, photothermal deflection technique or the so-called “mirage effect”, is setup. A flat and smooth sample is heated by a modulated 532 nm 14 mW pump beam on the surface. The heat flow induced by the surface layer is detected by the 632 nm 0.14 mW probe beam. The frequency-dependent signal in the range of 1 - 800 Hz is measured by lock-in amplifier in term of amplitude and phase. The clay roof tile with and without the waterproof glaze layer on top are the measured samples. The results give the thermal diffusivities of the clay roof tile and the waterproof glaze layer of 0.67 mm²s⁻¹ and 2.32 mm²s⁻¹, respectively.

1. Introduction
Photothermal deflection (PTD) technique, which is non-destructive and non-contact method, is used to determine the thermal properties of a variety of the sample. It can be used to measure the thermal diffusivity of bulk materials [1], thin solid films [2], etc. This technique, so called mirage effect, was developed by Fournier, et al [3]. By placing a sample on a sample holder and heating it by the pump laser beam with a wavelength in the direction perpendicular to the surface of the sample. Then the probe laser beam passed skimming the sample surface and detected by the quadrant photodetector. The signal was obtained from photodetector can be determined the thermal diffusivity of the sample. In this research, we are interested in a clay roof tiles and a waterproof glaze, which was developed from the traditional roof of Thai-style house. The clay roof tiles were used to thatch the Thai-style house instead of the thatch. The humidity is the main cause of less life time. In the recent year, the clay roof tile was developed by coating a waterproof glaze above the clay tiles for useful life.

The mirage signal analyzed using various methods for evaluate the thermal diffusivity. This study has been carried out by the use of photothermal deflection technique or mirage effect by amplitude method [4], in order to determine the thermal diffusivity of the clay roof tile and also waterproof glaze layer which cannot be measured by the thermal constant analysis (TCA) by using the amplitude of transverse component of probe beam deflection.

2. Methodology

2.1. Experimental setup
The experimental setup of the mirage method used for this research shown in figure 1. The sample must have one flat surface, not polished and toward the incoming pump laser. It is placed on the
translated stage allows for micron adjustment in both directions, the horizontal (z) and vertical (y) directions. The heating beam (pump laser) is 14 mW, 532 nm diode laser. The heating laser beam was modulated by the Acousto-Optic Modulator (AOM). Only the first order of the modulated heating beam was chosen to pass through the diaphragm [5]. The pump laser beam is focused to small spot size using a 75 mm focal length double-convex lens and thus a time variant temperature profile in sample and the overlying ethyl-alcohol. The beam waist diameter is calculated by 

$$\text{R}_{\text{waist}} = \left( \frac{2}{\pi} \right) \left( \frac{\lambda}{f} \right) \left( \frac{D}{f} \right)$$

, \( f \) is focal length of a lens, \( \lambda \) is wavelength of laser, \( D \) is the diameter of the light entering the lens. The beam waist diameter of the pump laser beam is estimated 25.40 \( \mu \text{m} \) as shown in the table 1.

| Table 1. The input beam parameters for the calculation of the beam waist diameter of both pump laser beam and probe laser beam. |
|---------------------------------------------------------------|
| \( \lambda \) (nm) | \( D \) (mm) | \( f \) (mm) | \( R_{\text{waist}} \) (\( \mu \text{m} \)) |
|-------------------|---------|---------|------------------|
| Pump beam         | 532.0   | 2.0     | 75.0             | 25.40             |
| Probe beam        | 632.8   | 2.0     | 110.0            | 44.31             |

The probe laser is 0.14 mW 632.8 nm He-Ne laser, and focused using a 110 mm focal length achromatic lens. The diameter of the probe laser beam is approximately 44.31 \( \mu \text{m} \). The focused probe laser beam was aligned parallel and closely above the sample surface. The probe laser beam passed through temperature gradient above the sample and deflected away from the original path. The deflection of probe laser beam is detected by quadrant photodiode (QPD) and the signal was collected by the lock-in amplifier (LIA) and the computer, respectively.

The line of probe laser beam is kept parallel to the surface of the sample. It is translated in y-direction, called y-offset is the transverse position of the probe laser above sample surface is passing into the temperature gradient, by step motor with resolution of 1.25 \( \mu \text{m} \). The vertical height \( z_0 \) between the sample surface to the center of the probe beam can be adjusted by micrometer screw.

![Figure 1](image-url)
2.2. Theory

We have to solve the heat diffusion equation in all media (fluid, sample and backing) to know the temperature distribution in the sample and in the surrounding area.

Figure 2 shows the different media concerned by the heat diffusion. The sample is heated by a modulated light beam which amplitude at the frequency, \( \nu \), is \( I_0 \). The modulated sample surface temperature can be written

\[
T_s = \frac{I_0}{2k_s (1+g)} \frac{(r-1)}{(\alpha - \sigma_s^2)}
\]

(1)

where \( g = k_f \sigma_f (k_s \sigma_s)^{-1} \), \( r = (1-j)(0.5\alpha \mu_t) \), \( \sigma_s = (1+j) \mu_t^{-1} \), \( \mu_t = (D_j)^{1/2} (\pi \nu)^{-1/2} \), \( j \) is a complex number, \( \mu_t \) is the thermal diffusion length, \( D_j \) is the thermal diffusivity of region \( i \) (f, s, b), \( \alpha \) is the optical absorption coefficient, \( k_s \) is thermal conductivity of the sample, \( T_s \) is the periodic temperature in the sample.

The temperature profile in the medium above the sample surface causes the deflection of the intensity of probe beam. This phenomenon is mirage effect, was already described in detail [1, 7]. The deflection of the probe beam is passing the temperature gradient. The deflection has been divided into two component, that is a normal component \( \phi_n \), which is normal direction with the sample surface, and a transverse component \( \phi_t \), which is parallel direction with sample surface. Only a transverse component is respected. The expression of the probe beam deflection in the photothermal deflection technique is given by:

\[
\phi_t = \frac{L}{n} \frac{d \sigma_f T_s}{dT} \exp(-\sigma_f z_0)
\]

(2)

Where \( n \) is the fluid refractive index, \( L \) is sample length, \( z_0 \) is distance between sample surface and the probe beam axis. We can calculate the thermal diffusivity by theoretical model in the case of high thermal diffusivity by amplitude method.

2.3. Sample

In this research, a Sigradur-G (SGG), a clay roof tile (CT) and a clay roof tile with a waterproof glaze layer on top (WG) are measured. The CT can be regarded as homogeneous material. The thickness of the waterproof glaze layer was about 470 µm. The compositions of the waterproof glaze layer were analysed by Energy Dispersive X-ray analysis (EDX). The result shows in table 2. A waterproof glaze layer was thermally thick sample, which is thermal diffusion length (\( \mu_t \)) is smaller than the sample thickness, and transparent.
Table 2. The compositions of a waterproof glaze layer measured by EDX.

| Composition | Atomic Percentage (At%) |
|-------------|------------------------|
| SiO₂        | 48.78                  |
| PbO         | 31.07                  |
| Other       | 20.19                  |

3. Results and discussion
The mirage effect technique is used to determine the thermal diffusivity of the sample. In this research, we obtained the deflection signals, amplitude and phase, from LIA. These signals were considered for the range of y-offset in proper fitting theoretical model. In the case of the measurement at 39.9 Hz, the y-offset range of 0.200 – 0.475 mm was chosen for determining the thermal diffusivity of SGG. This range is considered from the stable phase signal as shown in figure 3b and the amplitude signal with the exponential decay shown in figure 3a. The logarithm of amplitude versus y-offset in the chosen range was plot. The thermal diffusivity was determined by the slope of this plotting and calculated by:

\[ D = \frac{\pi \nu}{s^2} \]  

where \( s \) is the slope of the fitting. The results of SGG, CT and WG are shown in figure 4a, figure 4b and figure 4c, respectively. The samples were examined and summarized the results in table 3.

![Figure 3](image-url)
Figure 4. The plot of log of amplitude versus y offset (mm) of SGG (a), CT (b) and WG (c) for the modulated frequency 15.85 Hz and 39.9 Hz.

Table 3. Thermal diffusivity for samples measured by mirage effect.

| Sample | $D$ (mm$^2$s$^{-1}$) | $D$ measured (mm$^2$s$^{-1}$) |
|--------|----------------------|------------------------------|
| SGG    | 6.00$^a$             | 6.20±0.39                    |
| CT     | 0.66$^b$             | 0.72±0.21                    |
| WG     | -                    | 2.32±0.02                    |

$^a$The result is literature value [8].
$^b$The result from thermal constant analysis (TCA).

The results of slope of SGG and CT for frequency 15.85 and 39.9 Hz are about the same but the value of slope is not the same. For the slope of SGG for 15.85 and 39.9 Hz is 2749.9 m$^{-1}$ and 4646.4 m$^{-1}$, respectively. The slope of CT for 15.85 and 39.9 Hz is 9920.5 m$^{-1}$ and 11644 m$^{-1}$, respectively. We have calculated the thermal diffusivity by equation 3 and shown the thermal diffusivity of all samples in table 3.

The mirage effect technique result of SGG is great agreement with the literature value. We calculated the thermal diffusivity of SGG by theoretical model. The result of CT is good agreement with the result from thermal constant analysis (TCA). Furthermore, this technique can be measured the thermal diffusivity of WG as a thin layer. So the thickness of WG was greater than the thermal diffusion length, which the heat could not distribute to CT layer. However, TCA technique could not be measured for thin layer. Consequently, the mirage effect technique has high accuracy and precision in measurement of various materials, bulk and thin film materials.
4. Conclusion

In this work we succeed to determine the thermal diffusivity of Sigadur-G, clay roof tile and waterproof glaze layer by using photothermal deflection technique. This technique is the powerful, accurate and precise method. The thermal diffusivity was calculated by theoretical model. We found that the thermal diffusivity is great agreement with the literature and the result measured by TCA. Moreover, this technique can measure various materials, which TCA could not apply. The mirage effect method provides a way to measure the thermal diffusivity of the porous material and the thin layer on top.

References

[1] Salazar A, SánchezLavega A and Fernández J 1989 Journal of Applied Physics 65 4150-6
[2] Ghrib T, Yacoubi N and Saadallah F 2007 Sensors and Actuators A 135 346-54
[3] Boccara A, Fournier D and Badoz J 1980 Applied Physics Letters 36 130
[4] Ravi J, Lekshmi S, Nair K and Rasheed T 2004 Journal of Quantitative Spectroscopy and Radiative Transfer 83 193-202
[5] Frohlich B, Lahaye T, Kaltenhauser B, Kubler H, Muller S, Koch T, Fattori M and Pfau T 2007 Review of Scientific Instruments 78 043101
[6] Roger J, Fournier D, Boccara A and Lepoutre F 1989 Journal de Physique Colloques 50 (C5) C5-295-310
[7] Kuo P et al 1986 Canadian Journal of Physics 64 1165-7
[8] Salazar A and Oleaga A 2012 Review of Scientific Instruments 83 014903