A Simplified Low Cost Converging Thermal Wave Technique for Measuring Thermal Diffusivity of Thin Foils

M. S. Husin¹, M. M. Moksin¹, S. Jibrin¹, B. Z. Azmi¹, W. M. M. Yunus¹, M. Waziri²

¹ Department of Physics, Faculty of Science, Universiti Putra Malaysia, Serdang, Selangor, Malaysia ² Department of Mathematics, Faculty of Science, Universiti Putra Malaysia, Serdang, Selangor, Malaysia

ABSTRACT: Ultrashort pulsed lasers have been widely used in measuring thermal diffusivity of thin solid specimens where the output temperature is detected by using fast radiation detector. One of the techniques used for measuring thermal diffusivity of submicron thin foils mostly for high conducting materials is done using converging thermal wave technique which is based on ultrashort pulsed laser. This article reported a moderate simplification of converging thermal wave technique is successfully done by using low cost camera's flash lamp and the temperature at the rear surface is detected using thermocouple. The theory of the present work is presented and the results obtained from the curve fitting are discussed. In addition simulation of heat propagation and temperature distribution in the samples is performed using visual finite element analysis as a guidance purpose regarding the present work. The results obtained from the present work have the deviation less than 5% compared to the standard converging thermal wave technique.

Keywords - converging thermal wave technique; thermal diffusivity; thin foils; flash lamp

I. INTRODUCTION

In new era of nanometers and quantum dots fabrication technologies, characterizing thermo physical properties for the decreasing size dependent materials intensively need to reconsider for the new requirements of measurement techniques as they are categorized in low dimensional properties instead of bulks. Many researchers have tried to maintain their measurements of axial thermal diffusivity with negligible heat loss assumption using ultrashort pulsed laser exposed on the specimen of the intermediate thickness within a few millimeters thick samples [1-3]. These requirements of techniques are not suitable for the system which used a low power heat source such as low cost flash lamp with a pulse width of a few ten milliseconds.

Thermal diffusivity measurement by using flash method as introduced by Parker et al. [4] is widely accepted in industries and research institutes since early 60's. Using this method, the entire front surface is exposed to the short pulsed laser and thermal diffusivity is measured at the rear surface in the axial direction. Heat loss is neglected for the small temperature rise and only a single point of half time (the time corresponding to one-half of the maximum temperature evolution) is employed to evaluate the parameter of thermal diffusivity. The flash method requires fairly simple specimen, furnaces, and associated control, but it is unsuccessful to measure thermal diffusivity of ultra thin sample especially for high diffusivity materials without the present of ultrashort pulsed laser. Kehoe et al.[3] has been successful measured the thermal diffusivity of high conducting metal of foils using the same method proposed by Parker using nanoseconds pulsed laser, but alternatively detecting the temperature evolution at the front surface.

Cielo et al. [5] have developed a new method called converging thermal wave (CTW) technique which originated from the concept of radial heat flow method [6]. This method reversed the radial heat flow method by exchanging the central annular short pulse energy to the specimen with the circular ring departs from the central part of the specimen, with the detector located at the centre of the rear surface. Converging thermal wave technique provides a much stronger detected signal compared to the diverging heat flow [6], as it was a collection of energy that converged to the centre of the rear surface of the specimen.

CTW technique is a powerful technique for characterizing thermal properties of thin and high diffusivity materials [3, 6-7, 9, 12], but somehow it is still uncomplimentary low cost technique as it requires a ultrashort pulsed laser as the heating source and IR (infrared) camera to monitor the temperature. CTW can be applied for a small specimen of solids if the diameter of the specimen is two times larger than that of the heat source ring [12]. Along with the CTW technique, a simple algorithm has been developed by Murphy et al. [7] to treat heat loss parameter in the analytical solution of the CTW technique.

In this paper we report the CTW photoflash technique is moderately simplified by using a low cost technique within reliable accuracy of measurement which composes a low power heat source using 10 milliseconds pulse width of camera flash instead of ultrashort pulsed laser in order to induce heat and generate thermal wave signal in the specimen and temperature is monitored using an ordinary thermocouple. Since

ultrashort pulsed laser is no longer necessary (as the heating source), by making suitable adjustment modification on the theory and the setup, we manage to measure thermal diffusivity of thin foils using a low cost CTW technique exclusively as a non-destructive technique.

II. THEORETICAL

Let T(r,t) represent the temperature of a semi infinite specimen at a point r, at time t. As the specimen is very large (∞) in radius, r as compared to the thickness z, the ideal case of a two dimensional case can be applied to solve the heat equation problem.



Fig. 1: Schematic diagram used in this work for measuring thermal diffusivity.

In Fig. 1, a surface of homogenous specimen instantaneously irradiates on a 'ring' region by a concentric heat pulse with energy Q (Joule) at time t=0 and $T(r,t)=T_1=10$ K (maximum value of the thermal gradient). The thermal wave generated on the surface then diffuses to the centre (origin) of the coordinate, $r = \sqrt{x^2 + y^2} = 0$. Assuming the entire specimen is uniformly homogenous with constant thermal properties, the heat equation for this problem can be written as:

$$\frac{\partial T}{\partial t} = \alpha \frac{1}{r^2} \frac{\partial}{\partial r} \left(r^2 \frac{\partial T}{\partial r} \right)$$
(1).

The temperature T(t) that satisfying the heat conduction equation (for the semi infinite disc with 2 D approximation) with the boundary at the edge $(r=\infty)$ $T = T_0 = 0$ K, at t=0 s (equilibrium state) is given by [5]:

$$T(r,t) = \frac{Q}{4\pi\alpha t^p} \exp(-\frac{r^2}{4\alpha t})$$
(2).

Equation (2) can be simplified by normalization approach which can be achieved by dividing equation (2) with the maximum value of $T_{max}(r,t_p)$, therefore Q/($4\pi\alpha$) is vanished and equation (2) become simplified with the maximum amplitude 1 (dimensionless) and only dependent on the radius (*r*) and thermal diffusivity (α). Constant *p* is related to the geometry of the samples which correlated to the half of the respective physical dimension structure. For example the value of *p* in this work is 1 (as two divide by two) as 2 dimensional is considered where the thickness of specimen, *z* is infinitely small compare to the length of radius, *r*.

$$\frac{T(r,t)}{T_{\max}(r,t_{p})} = \frac{t_{p}}{t} \exp[\frac{r^{2}}{4\alpha}(\frac{1}{t_{p}} - \frac{1}{t})]$$
(3).

If equation (2) is differentiated, the peak time at which the amplitude reaches a maximum, t_p , can be used to calculate the initial value of the thermal diffusivity, then this value is used in the curve fitting evaluation in order to obtain more accurate thermal diffusivity. After differentiate, equation (2) becomes as [8]:

$$\alpha = r^2 / (4 p t_p) \qquad (4).$$

The standard converging thermal wave technique [5, 7-8, 9, 12] are using a heat pulse width of less than 10 nanoseconds and sub micrometers ring of irradiation. This measurement has been conducted by Kehoe et al. [9] and has recorded the temperature up to 80 microseconds of smoothed data Cu foil (2 mm thick) and taken up

more than 200 microseconds for the temperature to achieve the equilibrium state at the ambient temperature. Since the present work is using the heat pulse width of 10 milliseconds (1000 times longer), theoretically it shall expanded the temperature signal 1000 times longer to achieve the equilibrium state. Therefore equation (3) will be expanded 1000 and can be written as:

$$\frac{T(r,t)}{T_{\max}(r,t_p)} = \frac{t_p}{t} \exp[1000\frac{r^2}{4\alpha}(\frac{1}{t_p} - \frac{1}{t})]$$
(5)

and this expansion is also applied to equation (4) appropriately:

$$\alpha = 1000[r^2 / (4 p t_p)]$$
 (6).



Fig. 2:(a) Characteristic curve of the equation (3) and (b) Characteristic curve of the equation (5).

Fig. 2 shows the two curves are similar to each other after plotting equation (3) and (5) but they are different in the time scale of microseconds and milliseconds respectively.

III. NUMERICAL ANALYSIS

Finite element analysis (contour image and curve plot of 2D heat transient using VisualFEA) is performed in order to visualize heat propagation and temperature distribution in the specimen simultaneously after the flash.One series of parameters input for the semi infinite specimen of a thin Cu foil (ρ =8920 kg/m³, C_p=390 J/kgK, k= 400 W/mK) have been selected for the numerical analysis. The parameters input for the numerical analysis are:

Table 1: Parameters input for the numerical analysis						
Energy of	Thickness, z	Diameter of	Radius	Width of	Ring	Ambient
pulse,	(m)	specimen,	of ring,	ring,	temperature, T_1	temperature,
Q (J)		d (m)	r (m)	w (m)	(K)	$T_0(K)$
					at t=0	(constant for
						$0=t=\infty$)
0.5	100 x 10 ⁻⁶	20 x 10 ⁻³	5 x 10 ⁻³	2 x 10 ⁻³	310	300

VisualFEA (a program for the finite element analysis) is an easy programme to execute. It is done by drawing the required objects, assigning the values of the physical properties that related to the sample and setting up the appropriate boundary conditions. This program is emphasized on the visual output only and limited on equation analysis (such as the physical model is only defined by the graphical method without presenting any mathematical expression) therefore the exact equation of heat partial differential of equation (1) is not solved in this analysis. The purpose for using VisualFEA as only a guidance regarding the present work, therefore the results from this analysis are not employed further to analyze the experimental data.

IV. EXPERIMENTAL

Three foils of high purity 99.99 % (manufactured by Advent Co. Uk.) were cut into semi infinite disc. These foils are gold (Au), Copper (Cu) and Aluminum (Al) with identical thickness have been measured using the setup in Fig. 3. All parameters input that have been employed in the experiment are shown in Table 2.

Table 2: Parameters input that have been utilized in the experimental work						
Energy of	Thickness, $\pi (10^{-6} \text{ m})$	Diameter of specimen, $d_{1}(10^{-3}m)$	Radius of ring, $r(10^{-3}m)$	Width of ring, $(10^{-3} m)$		
Q (J)	2 (10 III)	a (10 m)	r (10 m)	w (10 III)		
0.5	125	20	3.5	2		

Thermal diffusivity of the specimens is obtained from the normalized temperature (at the rear surface) respect to time. Thermal diffusivity of the material can be determined by fitting the experimental data with the analytical solution given by equation (5).



Fig. 3: Experiment setup for the present work to measure the thermal diffusivity of the foils.

In order to facilitate the measurement, a LabVIEW GUI (Graphical User Interface) application has been developed in order to evaluate the thermal diffusivity of each samples and the chronology of this procedure is shown in the flow chart below: Examples for this GUI application are shown in the Appendix.



Fig. 4: Flow chart of the evaluation steps used in the present work.



Fig. 5: Curve simulation generated using Mathematica (software) to approximate the best thermal diffusivity value of the samples as the value is to be implemented in the curve fitting evaluation. Theoretical curve (continues line) and experimental data (unfilled circle) for Cu sample are plotted as for example of the simulation.



V. RESULTS AND DISCUSSION

Fig. 6: Images of temperature for t=0 s and t=5 s across the specimen, d (normalized diameter).



Fig. 7: Images of temperature for t=15 s and t=30 s across the specimen, *d* (normalized diameter).



Fig. 8: Images of temperature for t=45 s and t=60 s across the specimen, d (normalized diameter).



Fig. 9: Images of temperature for t=75 s and t=90 s across the specimen, d (normalized diameter).



Fig. 10: Images of temperature for t=105 s and t=150 s across the specimen, d (normalized diameter).



Fig. 11: Temperature curve created using the numerical data (t = 0, 1, 2, 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, 90, 100, 110, 120, 130, 140 and 150 s) originated at the centre of the specimen (a) full scale (160 s) and (b) half-scale (80 s). The curve (b) shows a similarity with the curve reported in the literature [8] for the Cu sample (2 mm thick) but in different time scale (as one in seconds and the other in microseconds of time scale).

Temperature distribution in the sample for 0 s and 5 s (as shown in Fig. 6) show that the flow of heat propagates to the centre (r=0) and to the outer edge of the sample. In the numerical analysis, we expect the temperature at the 'ring' of the irradiation region is small and not less than 10 K at t=0 s after the flash Then slowly the strength of heat is decayed exponentially and continuously distributed to the entire surface enforced by the thermal gradients. The temperature at the centre of the specimen is exponentially increased and reached the maximum 307.6 K at t=30 s as shown in Fig. 7 and Fig. 11. The equilibrium temperature (300 K) is reached at about 150 s where heat is totally departed from the sample as shown in Fig. 10.

Experimental

Curve fitting is performed in order to evaluate the value of thermal diffusivity (α) for each sample and the estimated value of thermal diffusivity is calculated using equation (6). The t_p is found from the polynomial curve fit as shown in Fig. 12 (for example).



Fig. 12: Polynomial curve fit of 5th order is drawn (continuous line) to the experimental data in order to find the specific time at the peak using QtiPlot software. In the graph, x is representing the peak time t_p which equal to 27.8 s as shown at the upper window of the application.



The outputs of curve fitting (using Qtiplot software) are shown in the Fig. 13, 14 and 15 have been used for extracting the thermal diffusivity value for each sample. Only data that fitted well to the theoretical curve is selected (where ranging starting from 16 s, 18 s, and 25 s for Au, Cu and Al respectively up to 80 s) in order to obtain high accuracy thermal diffusivity values.

Results of curve fitting have shown the starting points of fitting are different for the different samples, which Au has the starting point at 16 s which is slightly quicker compared to the others where Cu and Al have the starting point at 18 s and 25 s respectively. This explained that the theoretical curve of fitting can be slightly

improved (for better starting point which close to the early of the rising part) by using higher thermal diffusivity sample. In order to successfully fit between the theoretical model and the experimental data, the heat pulse duration and the width of the ring must be minimized to the optimum condition by using ultrashort pulse width [5, 7, 9] and a few tens of micrometers of 'ring' [12] respectively. However we expected heat loss will rather quickly dominated in the sample compared to the present work.

From the results of curve fitting, the thermal diffusivity for each sample can be extracted by fitting some part of the temperature curve (some portion at the peak up to the rest of the tail, as long as heat loss is assumed negligible at the rest of the tail). Thermal diffusivity value that obtained from the curve fitting must not deviate too much (as we obtained less than 5% as shown in Table 3) from the estimated calculation value, otherwise the temperature curve seemed to be interfering with heat loss. The advantage of the present setup, the heat loss is obviously optimized where no heat loss occurred in the measurement up to 80 s. This is because the heat exposed to the sample contained bulk of energy and remains in the sample longer since it was using longer pulse duration (10 ms) and wider width of the ring (2 mm).

The variation in the thermal diffusivity of gold films/foils is reported by Jauregui et al. [10] with thicknesses in the range 0.1 μ m to 200 μ m. They have found that there is a strong decrease in the thermal diffusivity of the gold film below 1 μ m which is attributed to both film geometry and structural disorder. From the result obtained by Jauregui [10] thermal diffusivity of gold films/foils is clarified by interpolating/extrapolating of a few measured data in the range of thickness between 100 nm up to 200 μ m. In order to report the thermal diffusivity as our reference, as it is an interpolated value, accuracy is not guaranteed. As the reason not many researchers reported on the thermal diffusivity of gold thin film/foils instead for the thermal conductivity, we considered the thermal diffusivity of Au in this work correlates to its bulk value [11] as shown in Table 4.



Fig. 13: Graphs of experimental data taken from the samples at the origin (r=0). Data is reduced from 200 to 60 points in order for better display in showing the different curves.

 Table 3: Thermal diffusivity for each sample obtained from the present work and its deviation (%) compared to the estimated calculation.

		Thermal diffusivity		
Sample	Peak time value, $T_m(s)$	Estimated calculation	Present work	Deviation, Abs. %
Au	24.4	1.255	1.267	0.9
Cu	27.8	1.102	1.133	2.8
Al	30.4	1.007	0.959	4.8

Absolute percentage value (Abs. %) is used to avoid negative sign deviation.

 Table 4: Thermal diffusivity for each sample obtained from the present work and its deviation (%) compared to the literature's value.

			1			
	Thermal diffusivity, $\alpha (10^{-4} \text{ m}^2 \text{s}^{-1})$			Deviation,	Deviation,	
Sample	Present work	Literature	Literature	Abs. %	Abs. %	
_		Ref. [9]	Ref. [11]	(Present work & Ref. [9])	(Present work & Ref. [11])	
Au	1.267	-	$1.270^{(Bulk)}$	-	0.24	
Cu	1.133	$1.182^{(100\mu m)}$	-	4.1	-	
Al	0.959	$0.964^{(100\mu m)}$	-	0.52	-	

Absolute percentage value (Abs. %) is used to avoid negative sign deviation. Superscripts in the bracket are referring to the thickness of the respective samples.

VI. CONCLUSION

We have developed a moderate simplification of converging thermal wave technique for measuring thermal diffusivity of thin solids using low cost heat source and detector. This present work is not required for ultrashort pulsed laser and fast radiation detector. We have obtained thermal diffusivity for each foil has the value of deviation less than 5% compared to the standard CTW with no heat loss signal up to 80 s, hence validating the experimental measurements. Finite element analysis is used to visualize the heat propagation and temperature distribution in the specimen as only for the guidance and better understanding purposes regarding the present work.

ACKNOWLEDGEMENTS

This work was financially supported by Ministry of Higher Education, government of Malaysia (Grant no. 5523905) and we are much thankful to the Applied Physics Group, Department of Physics, Universiti Putra Malaysia for the assistance and facilities support.

REFERENCES

- [1] K. Ramadan, W. R. Tyfour, and M. A. Al-Nimr, On the Analysis of Short-Pulse Laser Heating of Metals Using the Dual Phase Lag Heat Conduction Model. *J. Heat Transfer*, 131(11), 2009, 111301.
- [2] Mitsue Ogawa, Kazuo Mukai, Takehisa Fukui and Tetsuya Baba, The development of a thermal diffusivity reference material using alumina. *Meas. Sci. Technol.*, *12*, 2001, 2058–2063.
- [3] L. Kehoe, P. V. Kelly, and G. M. Crean, Application of the laser flash diffusivity method to thin high thermal conductivity materials. *Microsystem Technologies*, *Volume 5*, 1998, Issue 1,18-21.
- [4] W J. Parker, R J. Jenkins, C P. Butler and G L Abbott, Flash Method of determining thermal diffusivity, heat capacity and thermal conductivity. J. Appl. Phys., 32, 1961, 1679–84.
- [5] P. Cielo, L.A. Uracki and M. Lamontagne, Thermal diffusivity measurements by the converging-thermal-wave technique. *Can. J. Phys.*, *64* (9), 1986, 1172-1177.
- [6] A B. Donaldson and R E. Taylor, Thermal diffusivity measurement by a radial heat flow method. J. Appl. Phys. 1975, 46, 4584–9.
- [7] F. Murphy, T. Kehoe, M. Pietralla, R. Winfield, and L. Floyd, Development of an algorithm to extract thermal diffusivity for the radial converging wave technique. *International Journal of Heat and Mass Transfer*, *48*,(7), 2005, 1395-402.
- [8] Timothy Kehoe, Frank Murphy and Patrick Kelly, A Method for Measuring the Thermal Diffusivity of Intermediate Thickness Surface Absorbing Samples and Obtaining the Ratio of Anisotropy by the Converging Wave Flash Method. Int J Thermophys., 30(3), 2009, 987-1000.
- [9] Sok Won Kim, Jong Chul Kim and Sang Hyun Lee, Analysis of thermal diffusivity by parameter estimation in converging thermalwave technique. *International Journal of Heat and Mass Transfer*, 49, 2006, 611–616.
- [10] J. Jauregui, Z. L. Wu, D. Schafer and E. Matthias, Thermal diffusivities of thin gold films. (Springer Series in Optical Sciences Vol 69: Photoacoustic and photothermal phenomena III ed. D. Bicanic, 1992, 682-687).
- [11] J. King, Material Handbook for Hybrid Microelectronics, (Artec House, Norwood, Mass., 1988).
- [12] Y. Joo, H. Park, H.B. Chae, J.K. Lee, and Y.J. Baik, Measurements of Thermal Diffusivity for Thin Slabs by a Converging Thermal Wave Technique. *International Journal of Thermophysics*, Vol. 22, No. 2, 2001, 631-643

Appendix

Typical signal taken from control panel LabVIEW GUI used for reading and averaging data. After several averaging, the noises are diminished and the signal is greatly improved for a smoothed data obtained from the measurement.

