

**PROFESSOR DR. MOHD MAAROF H. A. MOKSIN**

# METROLOGY AT NANOSCALE

*Thermal Wave Probe Made It Simple*

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## ABSTRACT

A major hurdle facing nanotechnology implementation is in how samples of nano-scale dimensions can be probed. Parts of the problems include sample mounting, making contact with the sample; the possibility that the act of measuring alters the sample, repeatability and accuracy of measurement and referencing reference metrology to calibrate various tools to perform required measurements.

The present attempted solution is certainly not a one-size-fits-all matter and no more than a complement to existing tools available such as SEM, TEM, AFM etc, which are no match for the simplicity of the thermal wave probe.

Initially the thermal wave probe was not intended for nano-scale applications. The discovery of the converging thermal wave mechanism in thin subsurface layers opened up avenues for stand alone thin layer probing even for materials of high thermal conductivity and diffusivity like copper and silver. This came in tandem with the advent of nano-materials whereby electronic packaging materials could include thin layers with thermal diffusivity exceeding that of copper to alleviate problems associated with overheating.

From the time the converging thermal wave technique was introduced in the mid eighties until very recently, it could only be performed with the availability of massive and expensive ultra-short lasers even though the thinnest material that could be measured was 30  $\mu\text{m}$ . With the birth of the CTWaveProbe™ this myth should no longer exist. Within the limitations of standard samples available the CTWaveProbe™ has measured samples of thickness as low as 0.75  $\mu\text{m}$ .

In the near future everyone can perform nano-scale measurement like everyone now can fly!



## INTRODUCTION

Since time immemorial beauty and the love of it has not been merely skin deep. Man's extensive perceptions and obsession with beauty motivates him to look much deeper by not altering outer appearances which are already part and parcel of beauty. Scientists and engineers showed the way by using non-destructive testing (NDT) tools. In a nutshell the tool sends information carriers which non-destructively interact with materials beneath the skin and carry the results of the interaction back to the tool to be analyzed for matters of interest that remain hidden from unaided eyes.

Different information carriers interact differently and therefore conveys different information that may represent some of the material characteristics and features. The standard information carriers that are conventionally in use include the electron beam in SEM, ESEM and TEM; the atomic force in AFM and x-rays in XRD which produce images of the sample at high resolution at atomic and molecular level. These images provide strong justification for the established or new characteristics of the sample that still have to be measured by other suitable tools like the thermal wave probe. The characteristics of the materials or products are direct indications of its ability, suitability and sustainability to perform under specified conditions.

As the world view shrinks from huge big-bang matter to a mere global village, so do consumer products in the market become smaller and thinner. For instance, multilayers of even thinner films are now deposited on substrates in preparation of MEMS (micro-electromechanical systems). The trend of miniaturization also results in large dissipative power density in the devices that are usually operated at ambient temperatures such as computers and cell phone microchips. The power density in Intel Pentium chips may be equivalent to that of a hot plate of  $10 \text{ Wcm}^{-2}$  (Haque and Saif, 2005). Size and shape always matters and material properties of thin layers (attached or stand alone) are expected to vary with thickness. Thin materials' behavior deviates from



its bulk characteristics and properties due to its different structure or simply because information carrier interaction with and other interactions at boundaries become prominent as materials become thinner. Thermal properties such as thermal diffusivity and thermal conductivities are no exception.

The following pages describe how the CTWaveProbe™ found the way and the opportunities in simple nano-scale measurement.

## **THERMAL DIFFUSIVITY**

We are used to experiencing overheated arguments which have the tendency to deviate from the topic of discussion which eventually puts us in danger of a breakdown in communication. Thus we may end up with undesired results if we allow ourselves to be trapped in a web of overheated environments. The same problems may also occur in products and tools if its components and parts become overheated forcing them to operate at temperatures higher than has been specified. Overheating of components in controlled ambient conditions has its roots in the design which partly involves choice and availability of materials with suitable thermal properties to efficiently remove excess heat before temperatures rise above the threshold.

Heat transport within solids is governed by conduction and diffusion at rates determined by its thermal conductivity and diffusivity. Even though heat conduction depends on material thermal conductivity, the rate of rise (Haydari, 2004) and fall (Moksin *et al*, 1997) in temperature, in particular in high conductivity materials, depends on thermal diffusivity (Ozisik, 1993) which is known as the ratio of heat conducted to heat stored. Higher thermal diffusivity means that the material is better able to transfer heat than to absorb heat and is therefore less likely to trap heat and cause overheating. The best heat dissipating materials are thought to be prepared from graphene (hexagonal lattice of carbon atom). Japan Fujitsu Laboratories Ltd for example has very

recently successfully created a new nano-scale composite of self-organizing structure by combining carbon nanotubes and graphene. Graphene also could replace silicon in semiconductors since electrons travel more than 100 times faster in graphene than in silicon.

Being a derived quantity thermal diffusivity ( $\alpha$ ) is not a primary material physical property but is directly related to other physical properties of the material or its micrograph features. In the relation ( $\alpha = k/(\rho c)$ ) thermal diffusivity measures the final effect of collective changes in thermal conductivity ( $k$ ), density ( $\rho$ ) and the specific heat capacity ( $c$ ) of the material. On other hand, for phonon conductors, thermal diffusivity can also be expressed as (Bruls *et al*, 2005),  $\alpha = \frac{1}{3} \nu_s \ell_{tot}$  where  $\nu_s$  is the average phonon velocity and  $\ell_{tot}$  is the total mean free path of the phonons. Since  $\nu_s$  is weakly dependent on temperature,  $\alpha$  is determined by  $\ell_{tot}$ . Furthermore if there are no secondary phases taken into account  $\ell_{tot}$  is determined by lattice characteristics, defects and grain boundaries present in the materials. At low temperatures where lattice vibration has little effect, the thermal diffusivity indicates changes in the grain size (Haydari *et al*, 2004) besides defects that may exist in the materials.

Since thermal diffusivity is the most relevant thermal property, the following description focuses on issues related to the measurement of its quantity.

## THERMAL WAVES

One of the important information carriers of thermal properties is the thermal wave. Thermal wave, also known as heat wave, is a diffusion wave. Diffusion waves differ from other generally known waves by not obeying the wave equation:

$$\nabla^2 \psi = \frac{1}{v^2} \frac{\partial^2 \psi}{\partial t^2}$$

Instead, it obeys the diffusion equation:

$$\nabla^2 \psi = \frac{1}{\alpha} \frac{\partial \psi}{\partial t}$$

$\psi$  is an oscillating temperature resulting from the optical modulated heating of medium,  $\alpha$  is the thermal diffusivity and  $v$  is the velocity. Its highly damped nature which travels not further than a diffusion length before being severely attenuated makes thermal waves suitable candidates to probe surfaces, subsurfaces and thin layers for thermal properties. Further, thermal waves obey a linear law rather than a square law such that when they encounter interfaces, thermal waves obey an accumulation-depletion law rather than the reflection-refraction law of normal waves (Mandelis, 2000).

The solution to the last differential equation forms the major part of data analysis to extract thermal diffusivity of material from experimental data. The solution has many forms that can be tailored to the mechanism of thermal wave generation and detection other than the shape and size of the sample.

## **THERMAL WAVES GENERATION & DETECTION**

To have better control over thermal waves and more importantly to be able to extract information carried by the waves, it can be generated according to one's specific requirements in terms of thermal diffusion length, transparency or the specific temperature profile in the sample. In most cases, it is generated remotely following absorption of pulsed or modulated optical radiation. For choice of optical radiation, there are options from the most expensive and convenient such as lasers (Grozescu, 1998) to the very cheap and simple such as the camera flash (Haydari, 2004 and Husin 2007) that may also be very convenient for some types of work. For detection, the choice ranges from direct detection by using a remote IR detectors (Grozescu, 1998) such as liquid N<sub>2</sub> cooled CMT, thermally attached detectors such as PVDF film (Haydari, 2004),

thermocouples (Husin 2007) or laser probes (Grozescu, 1998 and Grozescu *et al*, 2004); to indirect detection by using a simple microphone (Yunus *et. al.* 2001 & 2002).

These options can be weighted in terms of fulfillment of some or all of the following requirements:

1. Rapid non-contact & non-destructive material evaluation.
2. Surface and subsurface characterization.
3. Bulk characterization.
4. Options for cheap and simple methods of measurement.
5. Ability to zoom on smaller details in the sample.
6. On-line measurement.
7. Sample properties to be determined.
8. Affordable, portable and reliable

Rapid non-contact & non-destructive material evaluation fulfills the need to have real time measurement with the luxury of flexible working distances, shielded from exposure to hazardous environment and having no physical contact in order to prevent exposure to possible contamination or infection. Additionally, no elaborate sample preparations are necessary. This is limited to the measurement of temperature of the surface from emitted gray-body radiation. The induced thermal wave in the sample can be directly detected as the change in the emitted radiation which depends on the sample thermal and optical properties it is associated with (Imhof *et al*, 1991)

- Surface emissivity
- Heating radiation penetration
- Thermal wave transparency

Thus the non-contact and non-destructive measurement (Moxsin *et al*, 1999 and Moxsin and Almond, 1995) in retro-geometry arrangement can be performed or adapted for

- Single access approaches as in the case of semi-infinite samples
- Continuous assessment of on-going processes such as paint drying, changes in thickness during coating process, sample sintering, surface coating contact condition and degradation during exposure to harsh and corrosive environment etc.

Even though thermal waves cannot be launched in a particular direction, smaller details of the sample can still be examined by using tightly focused laser heating and point detection of reflecting microscope objective signal collecting units as described by Grozescu and Moxsin (1998), Moxsin (1993) and Moxsin (1995).

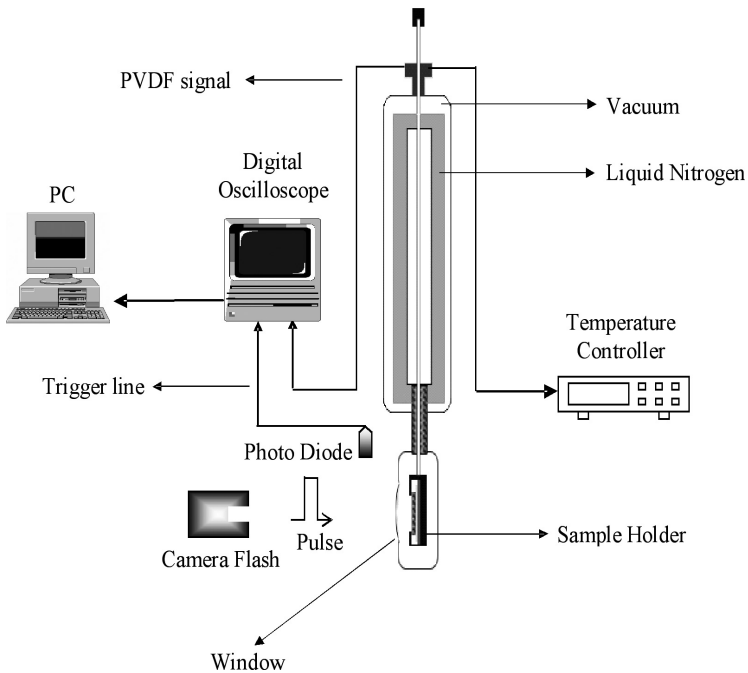
Some samples of high reflecting surfaces may not absorb enough optical radiation to generate an appreciable thermal wave signal which can be detected. In this case even though thermal wave is too weak to be directly detected it may still cause localized surface thermo-elastic deformation. A laser probe can be used instead to monitor any appreciable deformation on the sample surface as it undergoes temperature change. The shift in the reflected probe beam spot on a position detector depends very much on the slope of the deformed surface and hence on the thermo-elastic property of the deformed portion of the sample (Grozescu, 1998). From this single measurement, it has the capability to examine the formation and reduction of induced surface deformation and describe it in terms of three important thermal constants i.e. expansion coefficient, conductivity and diffusivity. However, extra precautions have to be taken in this pump-probe technique whenever the temporal profile of the heating laser is crucial in the measurement (Grozescu *et al*, 2000).

The Laser itself is no doubt a source of inspiration to perform many types of measurements (e.g. Woolsey *et al*, 1982). Besides the laser, which is synonymous with light applications in expensive research, other light sources such as cheap camera flash with a combination of cheap PVDF detection techniques (Josephine *et al*, 2007, and Haydari *et al*, 2008) have found many

applications even at temperatures measuring as low as 80 K (Haydari *et al*, 2004, 2005a & 2005b) as shown in Figure 1. Another cheap detection method is by using a microphone which detects the elastic effect in the medium following modulated light absorption (Yunus *et al*, 1999).

There are a few other varieties of thermal wave generation and detection methods each of which has its own advantages as reported by Azmi *et al* (2008) and Azmi *et al* 2004.

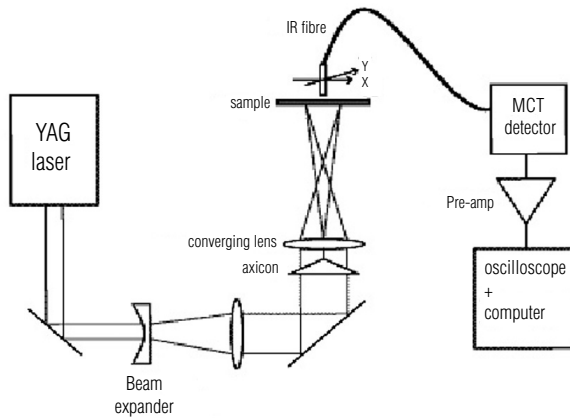
Another important example is presented in the following section. This only serves to emphasise further the great potential that the thermal wave technique has to offer in cases which embrace multidisciplinary work.



**Figure 1** Schematic diagram of measurement at low temperature (Haydari *et al*, 2004)

## CONVERGING THERMAL WAVE TECHNIQUE

The advantages of having converging thermal waves as information carriers is perhaps best appreciated by looking at it from diverging points of view. In the diverging thermal wave mechanism thermal waves spread out radially from a point heat source prior to its detection at a point laterally displaced (Moksin *et al*, 1994 and Luukkala *et al*, 1982). This arrangement is useful in examining sample heterogeneity in the radial direction and a complete profile of the sample can be obtained by rotating it in small increments up to 360°. However problems arise due to difficulties in detecting the signals particularly from high reflecting surfaces or low thermal diffusivity materials. Despite the laser beam being tightly focused, to the point that it could cause damage to the sample, to induce diverging thermal waves, the collected signals remain weak. To alleviate the problem a converging thermal wave technique was introduced by Cielo *et al* (1985). The heating laser beam was focused by using an axicon lens to form a circular pattern on the sample surface. The induced thermal wave inward flow from the heated annulus is detected at the center of the annulus. This arrangement significantly increased the signal to noise ratio to a value beyond what was achievable in the diverging wave arrangement. Enguehard *et al* (1989) seized this potential in measuring thermal diffusivity in radially anisotropic materials by improving the data analysis. The inherent sources of error found in this mechanism are non-zero width and non-uniformity of the annulus, the radial extent of non-zero and non-centered detection areas. These error problems become acute in cases where the optical heating beam has a smaller annulus radius which is a pertinent measure in the case of high reflecting surfaces or small thermal diffusivity measurements. Additionally the width of the annulus is limited by diffraction, and high focusing of the laser beam would also be damaging to the sample surface.



**Figure 2** Schematic diagram of converging thermal wave technique (Murphy *et al*, 2005)

Since the work done by Enguehard *et al* (1989) there have been a few other models developed (e.g. Lu and Swann, 1991; Murphy *et al*, 2005; Kim *et al*, 2006) to improve further on data analysis, which were tested against data from thin films. The Murphy *et al* (2005) model is considered the perfect one to effectively reduce off-centered and heat loss errors in addition to procedures in measuring effective radius of the annulus. Using the model in the measurement scheme in Figure 2, the thinnest sample on which they could measure thermal diffusivity was a 30  $\mu\text{m}$  copper foil which is thinner than the 50  $\mu\text{m}$  as achieved by Kim *et al* (2006). It has enhanced greatly the capability of the thermal wave as information carrier for being able to recognize thin layers. These are already been achieved and accomplished despite of the problems associated with zero width of the annulus and zero size of detection area as required in the models which has been compounded by relatively weak signal remaining unsolved. Errors originating from both sources could theoretically be reduced by increasing the radius of the annulus but in so doing it would impair the signal since heat loss and noise would also dominate. Some trade off between the need for



accurate and precise measurements and being able to get a discerning signal seems the only way out of this predicament. Bear in mind also that even at this level of accuracy of measurement and thickness of the sample an expensive ultra-short laser coupled with high speed detection is the only option. An even faster heating laser and detection method is required if the thermal diffusivity of materials increases further or the sample becomes thinner or both.

## **CTWaveProbe™: THE NEW CONVERGING THERMAL WAVE TECHNIQUE**

As stated earlier, success in thermal diffusivity measurements depends on the accomplishment of three major components i.e. thermal wave generation, thermal wave detection and data analysis to extract the thermal diffusivity value from the experimental data. The detection and data analysis parts can be simplified if strong thermal wave signals can be generated in the materials. Hence the thermal wave generation method could hold the key to simple detection and data analysis and possibly cheaper measurement & simpler operating procedures without sacrificing accuracy, precision and capability. Sometimes no matter how difficult a problem is it can be solved by a simple solution!

The novelty in the CTWaveProbe™ is in its use of a very large number of annuli of heating beams instead of a single annulus in the conventional design mentioned in section 5 above. The Laser source is no longer a necessity in this scheme of thermal wave generation and neither are the complicated beam delivery optics as shown in Figure 2. This simple innovation was successful in solving all the problems related to:

- a) Weak signal detection
- b) Annulus zero width
- c) Zero size detection area
- d) Complicated operating procedures
- e) Annulus radius measurement

- f) Expensive manufacturing and maintenance
- g) Portability and mobility
- h) Detection position centering

The large number of heating annuli obviously supplies much stronger signals than which can be possibly collected from the single heating annulus. At the same time annulus zero width requirements can be fulfilled as the number of annuli is close to infinity. Further, the scheme also provides the flexibility of having detection areas of finite size. Smaller errors may come from determination of radius involving the few outermost annuli. Since these outermost annuli signals are much smaller than the signal from the infinite number of the inner annuli, the effect of the errors on the measurements is effectively nullified. However, as precaution, the CTWaveProbe™ also specified the sample positioning procedure that takes into account the accuracy of the radius measurement. The other advantage of having a very large number of heating annuli is the elimination of the effects from non-uniformity in the beam profile that may exist by way of averaging.

This is the first time that converging thermal waves measurement can be generated in style without the expensive laser. No one has ever dreamt that all these can be accomplished by using a camera flash and a thermo-couple. The capability of the new design was proven by the measurements of high conductivity thin foils (Advent Research Materials Ltd) of thickness as low as 750 nm. It was tested against thinner sample of wider range of thickness than its competitors as presented in Table 1. There was no indication of any damage to the sample surface even for the thinnest sample as is normally present when laser heating is used.

**Table 1** Thermal Diffusivity of Aluminum, Copper, Nickel and Zinc foils (Advent Research Materials Ltd) with respective thickness (in closed bracket) as compared to other work

Sample	Thermal Diffusivity ( $10^{-5}\text{m}^2\text{s}^{-1}$ )				
	CTWaveProbe™	Kim et al (2006)	Cielo et al (1986) & Kim et al (2006)	Murphy et al (2005)	Weast (1994)
Al	9.45 (30 $\mu\text{m}$ )				
	9.80 (2.4 $\mu\text{m}$ )	9.64 (100 $\mu\text{m}$ )	8.12 (100 $\mu\text{m}$ )	9.49	9.54
	9.12 (1.5 $\mu\text{m}$ )	9.78 (50 $\mu\text{m}$ )	9.12 (50 $\mu\text{m}$ )	(645 $\mu\text{m}$ )	(bulk)
	9.66 (0.75 $\mu\text{m}$ )				
Cu	11.70 (7 $\mu\text{m}$ )				
	11.48 (50 $\mu\text{m}$ )	11.30 (50 $\mu\text{m}$ )	10.26 (50 $\mu\text{m}$ )	11.62	11.63
	11.62 (100 $\mu\text{m}$ )	11.82 (100 $\mu\text{m}$ )	11.45 (100 $\mu\text{m}$ )	(34 $\mu\text{m}$ )	(bulk)
Ni	2.31 (7 $\mu\text{m}$ )	2.33 (50 $\mu\text{m}$ )	2.06 (50 $\mu\text{m}$ )		2.29
		2.31 (100 $\mu\text{m}$ )	2.11 (100 $\mu\text{m}$ )	-	(bulk)
Zn					4.19
	4.19 (100 $\mu\text{m}$ )	-	-	4.07	(bulk)

In the absence of the sample micrograph, it is difficult to ascertain what causes thermal diffusivity to vary with thickness, as shown in Table 1. Since the thermal diffusivity value of the thicker samples is closer to their respective bulk value (Weast, 1994), it is believed that at that foil thickness the effect of thermal wave interaction with sample boundary was not significant. As the samples become thicker the thermal wave recognizes it either as thick foil or bulk samples.

## **CONCLUSION**

The superiority of the simple thermal wave probe has been demonstrated in measuring thermal diffusivity of free standing high conductivity foils down to 750 nm at room temperature. The CTWaveProbe™ is cheaper, simpler and a higher achiever than the other techniques. It should be the way forward if thermal wave application to stay relevant to the advancement in nanoscale measurement. It reaffirms my belief that simplicity can produce quality and superiority in revealing inner beauty.

Thin films are normally attached to its substrate. Converging thermal wave work on the effective thermal diffusivity of film/substrate composite is in progress.

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## **BIOGRAPHY**

**Mohd Maarof Moxsin** was born in Sabak Bernam 55+ years ago. During his childhood his parents gave him all the trust and freedom to roam around the village where man and the environment co-existed happily. He however will not extend the same freedom to his own children without monitoring and supervision.

He earned his B. Sc. (Hons) degree in Physics from UKM, M. Sc. from Brunel University and Ph. D. from Strathclyde University.

When he embarked on his career as a tutor in 1976, he didn't plan to become a lecturer which he did in 1979, and subsequently an associate professor in 1995 and a professor in late 2002, all at UPM. Now he plans to stay on at UPM to play with his many 'toys' in the laboratory.



## **ACKNOWLEDGEMENT**

**B**eyond the praise of kalimah alhamdulillah, I don't have sufficient words and deeds to express my gratefulness for His Blessings. Peace be upon Muhammad s.a.w., His servant and final messenger.

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Sincere thanks to UPM management for the opportunity to serve and to gain experience in an environment swinging between conducive and challenging. My apology is also due for all my share of shortcomings. Sincere gratitude also to all my teachers, mentors, colleagues, friends supporters and critics. I also must thank my students in particular for being my research growth engine.

Last and most important of all, I am very grateful to my late parents for giving me total trust, freedom, advice, support and guidance in whatever steps and decisions I have taken. May Allah bestow on them His Mercy even as they cherished me in childhood. Also to my beloved wife and children for accepting the inevitable reality that UPM has become my second home.



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