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Frequency modulated infrared imaging for thermal characterization of nanomaterials

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HIGHLIGHTS

- Reports frequency modulated thermal wave imaging (FMTWI) as a novel technique for thermal characterization.
- Active infrared (IR) thermography based photo thermal technique.
- Fast and noncontact technique with improved operational time.
- The proposed approach may improve all well established lock-in thermography based material characterization techniques.

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ABSTRACT

The paper presents frequency modulated thermal wave imaging (FMTWI) as a fast and efficient non-contact technique for in-plane thermal characterization of thin plate nanomaterials. A novel excitation signal in the form of an up-chirp is applied and the thermal response is monitored using an infrared (IR) thermography based temperature sensing system. The in-plane thermal diffusivity of any sample can be measured using the multiple phase information extracted from a single run of the experiment. This feature provides a time efficient approach for thermal measurements using infrared thermography techniques. The theoretical background and experimental details of the technique are discussed, with practical measurement of thermal diffusivity of an empty anodic alumina (AAO) template in direction perpendicular to the nanochannel axis, in support.

Thermal characterization techniques using contactless and fast measurement principles have fetched considerable global attention in recent past. Established non-contact techniques such as laser flash method [1,2] and mirage techniques [3,4] have their own limitations. Accuracy of laser flash method reduces considerably in case of materials having anisotropic thermal properties and very thin specimen dimension [5]. Also the mirage effect is largely used for observing temperature distribution near sample surface. Photothermal techniques such as Infrared (IR) lock-in thermography [6,7] phase method [8] and Surface Thermal Wave (STW) [9] have also emerged. In all of these techniques intensity modulated optical excitation produce a thermal wave response, whose propagation is captured using an infrared detector. The thermal diffusivity can then be retrieved from the phase or amplitude information corresponding to different excitation frequencies, as explained in Angstrom's method [10]. In situations of one dimensional (1D) heat propagation, thermal diffusivity can also be derived from the product of slopes of both the phase $m_{\varnothing}$ and the natural logarithm of the amplitude of the oscillating temperature $m_{\ln(T)}$ using the relation $m_{\varnothing} \times m_{\ln(T)} = \omega/2D$ [6,11,12] where ω is the angular excitation frequency and D is thermal diffusivity. This approach works very well for good thermal conductors like metals and alloys but it fails when dealing with thin films and filaments of low thermal conductivity. Our earlier work [13] reports measurement of thermal diffusivity of anodic alumina (AAO) templated nanocomposites using well established IR lock-in thermography based approach. Most of these conventional techniques demand experiments to be done at different excitation frequencies separately, which in turn arrogates longer experimental time.

The current study presents recently proposed frequency modulated thermal wave imaging (FMTWI) [14–17] as an efficient tool for in-plane thermal diffusivity measurement of materials, having anisotropic thermal properties and extremely thin specimen dimensions. Here, the idea is not to increase the spatial resolution directly to nano meter range. Measurements are done at mm length scale from which properties of nanostructures may be inferred using suitable model [18,19]. FMTWI, in essence, can be

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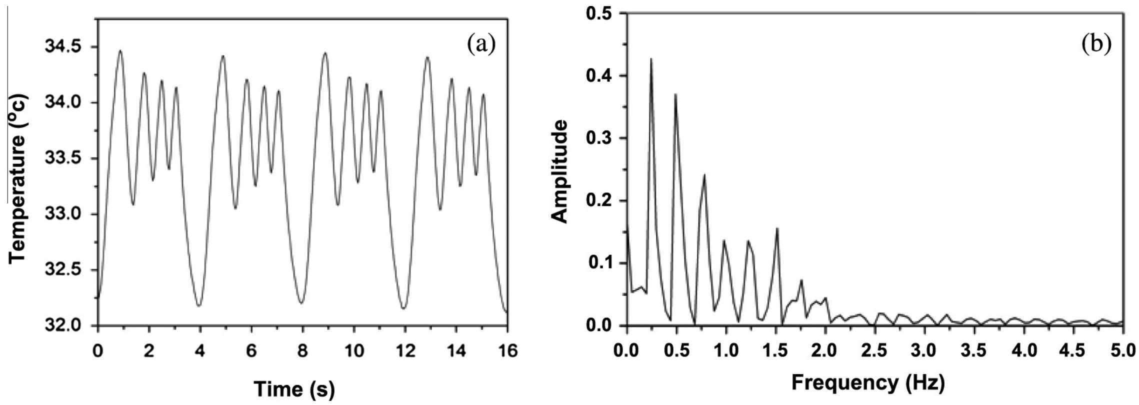

Fig. 1. (a) IR thermal response from 100 nm pore empty AAO template with an applied chirp excitation, (b) Fast Fourier Transform (FFT) analysis of the chirp temperature response, showing fundamental frequency of 0.25 Hz and its harmonic components.

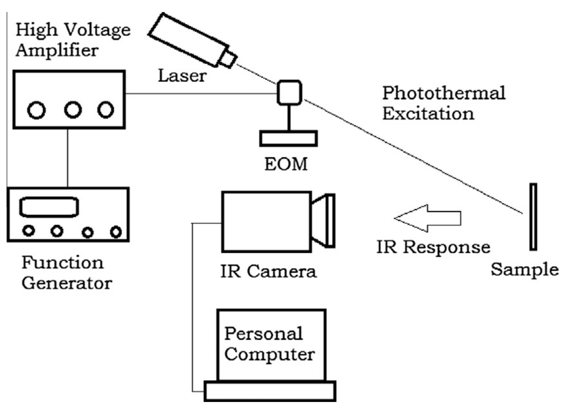

Fig. 2. Schematic diagram of the experimental setup.

termed as superposed lock-in thermography, which facilitates extraction of multiple phase and amplitude images from a single run, without having done the experiment at different excitation frequencies.

In this technique, a photothermal point source excitation comprising a linear frequency modulated signal (up-chirp) is applied close to the centre of the sample surface and the thermal response is monitored using an infrared (IR) camera system. As per Fourier theorem, a chirp signal may be viewed as a superposition of multiple sinusoidal signals, having frequencies which are integral multiples of the fundamental frequency. As the thermal waves propagate radially outwards, a change in phase and attenuation of wave amplitude are observed [10]. Out of these two parameters, the phase information is used solely for thermal diffusivity measurements in the present approach, as it is more immune to the local variations in surface temperature and emissivity of the radiating surface. In our proposed method, phase images corresponding to different excitation frequencies are extracted from a single run of the experiment and finally the in-plane thermal diffusivity is calculated from the variation of square of phase image slope with the angular excitation frequency. This reduces the operational time for the complete thermal diffusivity measurement experiment as compared to the conventional lock-in thermography approach where experiments need to be done at different excitation frequencies separately.

The mathematical model of the proposed technique is based on the equation of heat flow due to conduction. For any point source, periodic heating $Q_o e^{i\omega t}$ under steady state condition, the thermal response may be expressed as [20],

$$T(r,\mu) = \frac{I_o}{4\pi r\rho c D}\, e^{-\frac{r}{\mu}+i\left(\omega t-\frac{r}{\mu}\right)} \tag{1}$$

where $T(r,\mu)$ is the temperature, ω is the angular excitation frequency, r is the radial distance, μ is the thermal diffusion length, ρ is the density, c is the specific heat, and D is the thermal diffusivity. Here, μ is a measure of wave attenuation and is related to both ω and thermal wavelength λ as

$$\mu = \sqrt{\frac{2D}{\omega}} = \frac{\lambda}{2\pi} \tag{2}$$

From Eq. (1), it can be said that the phase $\varnothing$ of the thermal wave is a function of the radial distance r and can be expressed as,

$$\varnothing = \frac{r}{\mu} = r\sqrt{\frac{\omega}{2D}} \tag{3}$$

Eq. (3) is a very significant expression relating thermal diffusivity with excitation frequency and the phase image slope ($\varnothing/r$). Clearly, thermal diffusivity can be extracted from the slope of the plot of ω and $(\varnothing/r)^2$, without involving the amplitude information [13]. This approach of extracting D needs the phase images and the ($\varnothing/r$) slopes, corresponding to different excitation frequencies.

An efficient solution to this need is FMTWI. Here, a suitable frequency modulated (up-chirp) signal is applied as the photothermal excitation expressed as,

$$Q(r=0,t) = Q_o Sin\left\{2\pi\left(f_i + \frac{\beta t}{2\tau}\right)t\right\} \tag{4}$$

where f_i is the initial frequency, β is the bandwidth, and τ is the chirp duration. The thermal response from any sample, on application of this chirp excitation (Eq. (4)) can then be deduced as follows

$$T(r,\omega) = \sum_{\frac{1}{\tau}}^{\frac{n}{\tau}} \frac{I_o(\omega)}{4\pi r\rho c D}\, e^{-\frac{r}{\mu(\omega)}} Sin\left(\omega t - \frac{r}{\mu(\omega)}\right) \tag{5}$$

where n is an integer. Eq. (5) represents the cumulative response of the discrete excitation frequencies within the chirp bandwidth. As the chirp repeats itself after every τ interval, Fourier transform of the same produces $1/\tau$ as the fundamental frequency and $f_i \leqslant n/\tau \leqslant f_o$ as the significant harmonics. Here f_o is the final frequency of the chirp at $t = \tau$ and any harmonics beyond this frequency are highly attenuated and are not significant. It is also noticed that in situations of radial heat flow from a point source the thermal wave attenuates more rapidly following both exponential and inverse distance function, unlike the situation of one dimensional heat flow where the decay follows only exponential function. It becomes evident that the observed thermal response is well within one thermal