

Scanning modulated photothermal radiometry

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Abstract

In this work the photothermal radiometry technique is developed in order to observe the thermal properties of anisotropic materials at micro-metric spatial resolution. The use of modulated excitation in addition to the synchronous detection allows explore frequencies up to 100 kHz. In addition a scanning configuration has shown the capability to perform image acquisition of thermal behavior with micro-metric resolution.

1. Introduction

The thermal properties of materials at micro-metric scale are of key importance. In fact, their knowledge enables to design and optimize new microstructured materials with enhanced thermal properties for a wide range of applications, from building thermal insulation to microelectronic applications.

One distinguishes two main classes of methods for thermal characterization at micro-nanoscale: the contact and contactless methods. The main drawback of contact methods like SThM, is the presence of the additional unknown parameters relating to the contact itself and the significant thermal inertia of the probes which limits its application to low frequency region. On the other hand, the contactless methods are carried out without interaction probe-sample, allowing the study of faster dynamics. However, the contactless methods are not suitable to achieve absolute temperature measurements. In an effort to improve the thermal analysis of the material at micro-metric scale the modulated photothermal radiometry (MPTR) has been taken as the starting point for the development of a new scanning configuration setup.

2. Photothermal radiometry

Photothermal radiometry [1] is based on monitoring of the infrared radiation from the sample surface consequently to a photothermal excitation provided by a modulated laser. Considering a periodic heat flux $\varphi = \varphi_0 \cos(\omega t)$ applied at the sample surface, the temperature increase of the sample is composed of a continuous and a transient part as: $\Delta T(t) = \Delta T_0 + \Delta T_w \cos(\omega t + \phi)$. Assuming a weak enough disturbance to produce a small temperature increase, the variation of the emitted radiation can be linearized as: $\Delta M \approx 4\epsilon\sigma\Delta T_0^3 \Delta T_w \cos(\omega t + \phi)$. In this way it is possible to monitor the sample surface temperature ΔT by measuring the radiant emittance ΔM with an infrared detector.

3. Experimental setup

The experimental setup is presented in figure 1. The optical arrangement exploits three bands of the light spectrum. First, the 1.064 μm in wavelength CW laser beam is frequency modulated by an acousto-Optic modulator and conducted to the microscope objective which focus the laser beam on the sample surface. The infrared radiation from the sample surface (maximum power centered around $\lambda \approx 10 \mu\text{m}$), consequently to the periodic photothermal excitation at the same surface, is collected and conducted to the infrared detector. Finally the visible spectrum is used by a visible camera to manage the position of the sample.

The microscope objective produces a spot over the sample surface with radius r_0 . The radius has been measured through the knife edge method [2]. This technique consists in moving a knife edge perpendicular to the direction of propagation of the laser beam and record the total transmitted power as a function of the knife edge position. Assuming a Gaussian beam propagation, a spot radius $r_0 = 3.3 \mu\text{m}$ was obtained. The measurement area is given by the optical magnification of the system. The sensing optical system is composed of two elements, the microscope objective with a focal length $f_o = 5 \text{ mm}$ and the off-axis parabolic mirror with a focal length $f_m = 200 \text{ mm}$. Thus, the magnification is given by $m = f_m/f_o = 40$. The infrared detector is a HgCdTe sensor with an square active area of 500 μm width, and applying the magnification factor, the resulting sensing area over the sample surface at focal plane is a square with 12.5 μm width.

The sample is mounted over a 3-axis piezoelectric translation stage (P-611K101 from PI) with travel range to 100 $\mu\text{m} \times 100 \mu\text{m} \times 100 \mu\text{m}$. This precision displacement system is equipped with position sensors which allows to know the position of the plate while a feedback loop guarantees the exact and well repeatable positioning of the plate, avoiding thus a possible phenomenon of hysteresis. The translation stage allows to locate the sample on the focal plane and drive the lateral displacement of the sample to perform a scan over the sample surface. In the sweep process the sample surface is displaced at constant velocity in the focal plane, perpendicular to the fixed laser beam propagation axis. In this way, the image is made



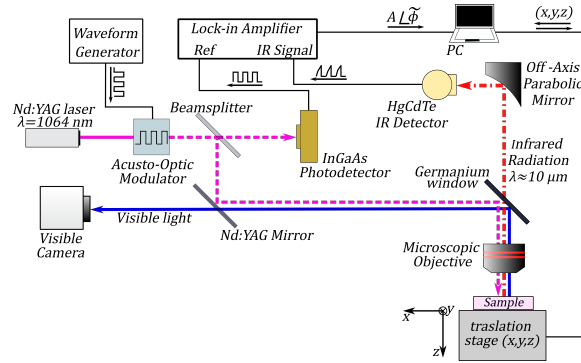


Fig. 1. Experimental setup

from pixel by pixel measurement with the infrared sensor at different sample positions. The signal from the infrared detector is measured with a lock-in amplifier which directly retrieve the amplitude and phase of the signal simultaneously.

4. Scanning

In order to show the capabilities of the setup, a silica/pyrocarbon composite has been scanned. The sample is composed of silica fibers with a diameter around $8\mu\text{m}$, surrounded by a pyrocarbon matrix, as shown in figure 2. The figure 3 shows the image of the sample surface, obtained with the visible camera, where the red rectangle encloses the scanned area, a fiber structure is easily identified close to the right edge of the scanning zone and that is the circular structure one expects to appear in the scanning.

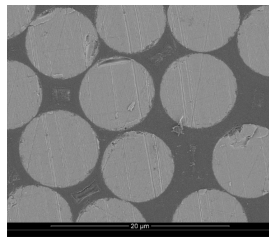


Fig. 2. Silica/Pyrocarbon sample (SEM image)

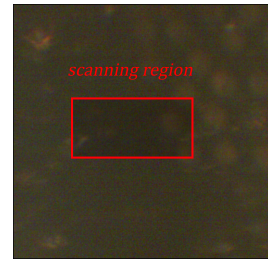


Fig. 3. Silica/Pyrocarbon visible image of scanning region

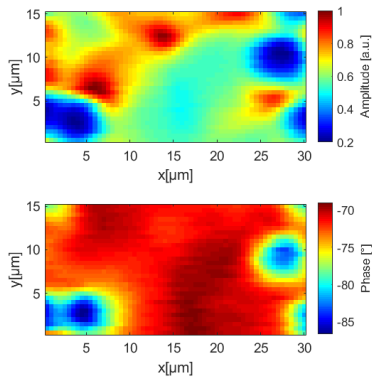


Fig. 4. Scanning at 99.013 kHz

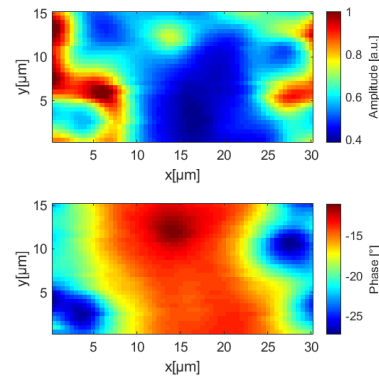


Fig. 5. Scanning at 1.125 kHz

In figure 4 one can observe the amplitude (top) and phase (bottom) of the infrared response from the sample consequently to the excitation flux modulated at 99.013 kHz. In both, amplitude and phase, is clearly visible the contrast between the silica fiber and pyrocarbon matrix and the circular structure belonging to the target fiber is easily identified close to the right edge of the images. Figure 5 shows the scanning at 1.125 kHz, one can note that at this frequency the fiber structure is less defined, this behavior was expected because the diffusion length is higher at low frequencies. The same phenomena explain the structures that appear in the infrared images but no in the visible image, this is because the fiber are behind a thin matrix layer.

5. Conclusion

The scanning configuration of the modulated photothermal radiometry makes it possible to obtain images of thermo-physical properties of composite materials at the micro-scale. The scanning of silicon/pyrocarbon sample shows the capability to perform studies at higher frequencies that the achieved by the infrared cameras [3, 4]. However, more work is necessary in the data post-processing and the coupling of the scanning results with 3D models to achieve quantitative characterization of thermal properties of the sample under study. The developed setup has potential application in the study and identification of thermal properties of micro structured materials and thermal boundary resistances.

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