Photopyroelectric scanning microscopy

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A new pyroelectric technique for the thermal wave scanning microscopy is presented. The potentiality of the method for probing and characterizing defects in solid samples is tested for surface and subsurface defects.

In recent years there has been considerable interest in the development of thermal wave microscopy techniques as tools for nondestructive testing of materials. Basically, these techniques consist of generating a localized periodic heating in a sample, due to the absorption of an intensity-modulated focused laser or electron beam, and scanning the heating position across the sample. The thermal waves resulting from the absorption of the periodic incident beam propagate from the heated regions and undergo reflection and scattering when they encounter regions of different thermal and geometrical characteristics. In this way, as the heating beam is scanned across the sample any surface or subsurface change in the thermal characteristics of the sample can be monitored as a change in the thermal wave signal. In this letter we propose another alternative method for the already rich arsenal of thermal wave microscopy techniques. The method is based upon the recently developed photopyroelectric spectroscopy (PPS). This new spectroscopy technique consists of using a thin pyroelectric film (e.g., polyvinylidene difluoride, PVF_{2}) in intimate contact with a solid sample on which monochromatic light beam whose intensity is sinusoidally modulated at a frequency \( \omega \) is incident. Following the absorption of the incident light, the nonradiative de-excitation processes within the solid cause the sample temperature to fluctuate, and through heat diffusion to the surrounding pyroelectric film, the sample-pyroelectric film interface temperature fluctuates. As a result of this temperature fluctuation, a pyroelectric signal is produced which is proportional to the temperature change in the film. This novel photopyroelectric approach for probing surface and subsurface structures is schematically shown in Fig. 1. The light from a 4-mW He-Ne laser beam after being modulated by a mechanical chopper is focused on the sample. To detect the modulated thermal fluctuation of the sample a 28-\( \mu \)m-thick PVF_{2} film (Pennwall) is mounted on its back surface. The whole sample-detector ensemble is mounted on a micrometer positioner so that it can be moved back and forth perpendicularly to the heating laser beam. The output voltage from the PVF_{2} detector is fed into a lock-in amplifier and recorded as a function of the illumination position \( x \). A similar pyroelectric imaging using electron beam heating rather than light beam heating has been previously reported by Bauman et al.\(^{16}\)

As a first test of the photopyroelectric microscopy (PPM), a simple "surface defect" was examined consisting of a machined slot, 250 \( \mu \)m wide, on the surface of a silicon wafer 600 \( \mu \)m thick. The He-Ne laser beam was focused by a cylindrical lens, providing a focused strip less than 50 \( \mu \)m wide on the surface having the defect. The resulting photopyroelectric signal as a function of the heating laser beam position is shown in Fig. 2 for a modulation frequency of 28 Hz. As expected an increased signal is observed over the slot where the sample is thinnest. Since the real advantage of the thermal wave microscopy lies on its ability to image "subsurface" defects which cannot be observed optically, we have next investigated this type of defect. A prototype subsurface defect was simulated by using the same silicon sample with the 250-\( \mu \)m-wide slot, illuminating, however, the sample at its smooth surface; i.e., we simply inverted the sample position relative to the incident laser beam. The result of the beam scanning over the sample surface is presented in Fig. 3.

To further explore the potentialities of the PPM we have next investigated whether this technique could not only point out where the defect is but also give us information about the physical nature of the defect, as manifested, for instance, by a change in the thermal diffusivity in the defect region. To answer this question we have performed a lateral heating experiment as schematically shown in Fig. 4. The light from a chopped laser beam is focused by means of a cylindrical lens along one face (normal to the \( y \) axis, cf. Fig. 4) of the sample, thereby generating at a lateral distance \( x_{0} \) from the pyroelectric film-sample border \( (x = 0) \) a localized periodic heat source. As a result of the localized heating at \( x = x_{0} \), a thermal wave is set in the back face (at the \( y = 0 \) plane) of our sample. This thermal wave diffuses along the \( x \) direction and eventually reaches the PVF_{2} detector at \( x = 0 \) attenuated exponentially as \( \exp\left(-a_{x}x_{0}\right) \), where \( a_{x} = (\pi f / a_{s})^{1/2} \) is the thermal diffusion coefficient. Thus, by measuring the pyroelectric signal, at a fixed modulation frequency, as a function of the heating beam offset \( x_{0} \), the thermal diffusivity \( a_{s} \) is readily obtained from the coefficient of \( x_{0} \) in the exponential. To ensure that along the \( x = x_{0} \) plane

![FIG. 1. Experimental arrangement for the photopyroelectric microscopy. The thermal waves generated on one side of movable sample S is detected on the opposite side by a PVF_{2} detector. The sample-detector ensemble is moved back and forth by the translator T.](http://apl.aip.org/about/rights_and_permissions)
an optically opaque sample is being uniformly heated, we have worked with thermally thin samples such that the thermal diffusion length \((a_s/\pi f)^{1/2}\) is much longer than the sample thickness \(t\). This important case of optically opaque samples is the one we address ourselves to in what follows. For a thermally thin sample it can be shown that the periodic temperature fluctuation \(\phi_s\) in the \(x = x_0\) plane is given by

\[
\phi_s(x_0, t) = -\frac{\beta' I_0}{t_s k_s \sigma_s} e^{-\omega t},
\]

(1)

where \(\beta'\) is the surface absorption coefficient of the sample, \(I_0\) is the heating laser intensity, \(k_s\) is the sample thermal conductivity, and \(\sigma_s = (1 + \rho) a_s\), where \(a_s = (\pi f / \alpha_s)^{1/2}\). Using Eq. (1) as a boundary condition in the heat flow equation along the \(x\) direction and neglecting the heat diffusion into the surrounding air, one can calculate the temperature rise \(\Delta T\) in the PVF\(_2\) film by taking the average temperature fluctuation in the detector-sample region \((-d < x < 0)\). For \(d \alpha_s / \sigma_s \gg \max 1\) (large detector-sample contact region) one gets

\[
\Delta T \approx \frac{\beta' I_0}{d t_s k_s \sigma_s} e^{-\omega t} e^{\omega x_0}.
\]

(2)

Equation (2) tells us that the pyroelectric signal should decay exponentially as a function of the beam offset \(x_0\), with the coefficient of \(x_0\) in the exponential being the thermal diffusion coefficient \(a_s = (\pi f / \alpha_s)^{1/2}\). Thus, when crossing regions of different thermal diffusivities, the slope of the semilog plot of the photopyroelectric signal as a function of \(x_0\) should also change thereby indicating the presence of a different medium.

To test the above method we have performed a lateral heating experiment using a He-Ne laser modulated at 26 Hz. The sample consisted of a thin Cu strip, 5000 Å thick and 200 μm wide, deposited on one side of a Si wafer 250 μm thick. The laser beam in this case was focused on the smooth surface so as to simulate a subsurface defect. In positioning the sample we have kept the Cu strip close to the PVF\(_2\) detector edge (cf. Fig. 4). In Fig. 5 we show the semilog plot of the

FIG. 5. Lateral heating image of a subsurface defect consisting of a 5000-Å thick and 200-μm-wide Cu film deposited on a 250-μm-thick silicon wafer. The clear surface of the silicon wafer was facing the heating beam, and the Cu film was positioned close to the PVF\(_2\) film.
amplitude of the pyroelectric signal as a function of $x_0$ for this subsurface defect. As expected, apart from exhibiting the discontinuity at the defect edge, Fig. 5 also shows a change in the slope of signal as a function of $x_0$. This change of slope is, of course, due to the different nature of the material in the defect region. A least-squares fitting of the experimental data to an expression of the form $S = S_0 \exp(-\alpha_x x_0)$ [cf. Eq. (2)] yielded $\alpha_x = 1.19$ cm$^2$/s in the region $0 < x_0 < 200 \mu$m and $\alpha_x = 0.87$ cm$^2$/s in the region $x_0 > 200 \mu$m, indicating that indeed the defect region is made of Cu (literature value $\alpha_x = 1.17$ cm$^2$/s).

In conclusion, we believe we have demonstrated the usefulness of the photopyroelectric technique to perform scanning microscopy. Its extreme simplicity, sensitivity, and minimal preparation requirement are some of the aspects to be taken advantage of in future works, as compared with some other detection techniques. The basic limitation of the PPM regards the fact that it is a thermal transmission technique which, in turn, restricts its use to low modulation frequencies.

References: