Quantitative photopyroelectric out-of-phase spectroscopy of amorphous silicon thin films deposited on crystalline silicon

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A photopyroelectric spectrometer with real-time and(or) self-normalization capability was used for both conventional transmission and thermal-wave spectroscopic measurements of amorphous Si thin films, deposited on crystalline Si substrates. Optical-absorption-coefficient spectra were obtained from these measurements and the superior dynamic range of the out-of-phase (quadrature) photopyroelectric signal was established as the preferred measurement method, owing to its zero-background compensation capability. An extension of a photopyroelectric theoretical model was established and successfullly tested in the determination of the optical absorption coefficient and the thermal diffusivity of the sample under investigation. Instrumental sensitivity limits of $\beta t \approx 5 \times 10^{-3}$ were demonstrated.

Un spectromètre photopyroélectrique avec capacité de temps réel et/ou d'auto-normalisation a été utilisé pour des mesures spectroscopiques, par transmission conventionnelle et par ondes thermiques, sur des couches minces de Si amorphe déposées sur des substrats de Si cristallin. Des spectres du coefficient d'absorption optique ont été obtenus à partir de ces mesures, et la partie dynamique supérieure du signal photopyroélectrique déphasé (en quadrature) a été établie comme méthode de mesure préférée, à cause de sa capacité de compensation du fond. Une extension d'un modèle théorique photopyroélectrique a été établie et testée avec succès dans la détermination du coefficient d'absorption optique et de la diffusivité thermique de l'échantillon étudié. Des limites de sensibilité instrumentale de $\beta t \approx 5 \times 10^{-3}$ ont été atteintes.

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1. Introduction

As is well known, during the last 20 years researchers and semiconductor industries have shown a great interest in the development of amorphous semiconducting materials. Many experimental techniques have already been used for the characterization of noncrystalline materials and several transport theories have been established (1). However, the interest in improving the experimental techniques, as well as the desire to obtain more information concerning the structural, electrical, and optical properties of amorphous materials has led to the development of new characterization techniques affording specific advantages over conventional methods.

Recently, frequency-domain photopyroelectric spectroscopy (PPES) has been used for the characterization of thin semiconducting films. This technique is capable of out-of-phase thermal-wave lock-in detection with complete suppression of synchronous background signals owing to direct optical transmission to the pyroelectric sensor (2, 3). Mandelis *et al.* (4) first performed spectroscopic PPE measurements of an a-Si: H thin film on quartz. Christofides *et al.* (5, 6) have extended the use of PPES to spectroscopic measurements of thick crystalline Ge and Si wafer substrates.

In this work we present the first application of PPES on very thin absorbing amorphous layers deposited on absorbing crystalline Si substrates using the geometries designed for microelectronic device fabrication. In Sect. 2 of this paper we present a PPE theoretical model pertinent to such geometries. In Sect. 3 the experimental set-up for PPES will be discussed briefly, since the spectrometer has already been presented elsewhere in detail (7). Experimental results and comparisons between PPE and conventional transmission spectra will be presented in Sect. 4. Section 5 includes our preliminary assessment of the dynamic range of PPES with respect to the product of βt (β is the optical absorption coefficient, and t the thin-film thickness), in comparison with that reported for photothermal beam deflection spectroscopy (PDS), a related zero-background technique. Finally, we present some conclusions and future perspectives of the photopyroelectric technique.

2. Theoretical model

Figures 1*a* and 1*b* present a one-dimensional geometry of the photopyroelectric system. A crystal substrate of thickness L assumed entirely transparent, on which an amorphous layer of thickness t is deposited, is irradiated with monochromatic light of wavelength λ, the intensity I of which is modulated at frequency f. As is shown in Fig. 1*a*, where the sample is in contact with the pyroelectric polyvinylidene fluoride (PVDF), the induced signal, S, is the sum of a thermal component, S_T, and an optical component, S_O. If the substrate is assumed transparent, the surface reflectance is included in the form $1-R_1(\lambda)$, and the pyroelectric detector surface is blackened (i.e., photothermally saturated) then the normalized detected total PPE signal S, in this case is given by (2, 4)

$$[1] \quad S(f,\lambda) = S_T(f,\lambda) + S_O(f,\lambda)$$
$$= (1 - R_1)\{\eta(\lambda)[1 - \exp(-\beta t)]\exp[-(1+i)qL]\} + S_O(f,\lambda,R_1,R_2,R_3)$$

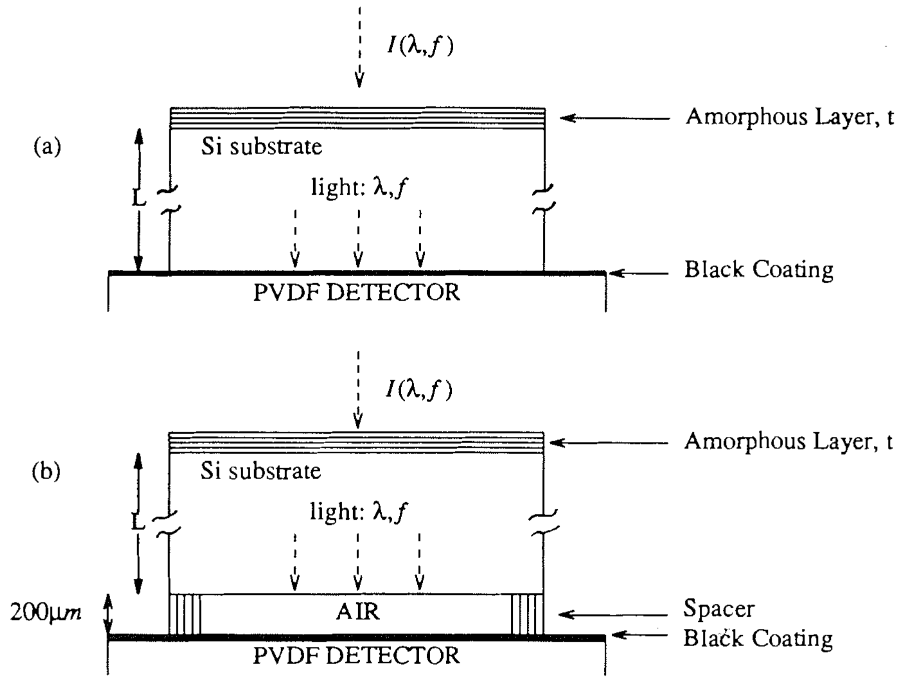

FIG. 1. One-dimensional geometry of a photopyroelectric system: (*a*) Configuration for thermal-wave spectroscopy. (*b*) Configuration for transmission-mode spectroscopy.

where $\eta(\lambda)$ is the nonradiative quantum efficiency for absorption and

$$q = \sqrt{\frac{\pi f}{\alpha}}$$

with α the thermal diffusivity of the substrate. Finally R_1 and R_2 are the reflectivities of film–air and film–substrate interfaces, and R_3 is the reflectivity of the substrate–air interface. As usual, these reflectivities at normal incidence are given by (8)

$$[2a] \quad R_1 = \frac{(n-1)^2 + k_0^2}{(n+1)^2 + k_0^2}$$

$$[2b] \quad R_2 = \frac{(n-n_1)^2 + k_0^2}{(n+n_1)^2 + k_0^2}$$

and

$$[2c] \quad R_3 = \left(\frac{n_1 - 1}{n_1 + 1}\right)^2$$

where n and n_1 are the refractive indices of the film on substrate, respectively; k_0 is the extinction coefficient of the thin film on the substrate.

In the case of the configuration shown in Fig. 1*b*, between the sample and the detector there exists an air gap of an approximate thickness 200 μm. An air gap of this thickness is thermally thick. Therefore, the PPE signal is due only to the direct transmission and is equal to S_0 (9)

$$[3] \quad S_0(f, \lambda, R_1, R_2, R_3) = \frac{(1 - R_1)(1 - R_2)(1 - R_3)\exp(-\beta t)}{(1 - R_2R_3)\{1 - [R_1R_2 + R_1R_3(1 - R_2)^2]\exp(-2\beta t)\}}$$

Theoretically, this equation describes the fraction of the incident power after propagating a distance t through the absorbing material (10). However, it is impossible to obtain a quantitative spectrum of the optical absorption coefficient because of the dependence of S_0 on the hard-to-determine coefficient R_2. This is one reason why a combination with thermal-wave measurements is needed.

From [1] one can write the in-phase synchronous signal

$$[4a]\quad \mathrm{Re}\,[S\,(f,\lambda)] = (1 - R_1)\,\{\eta(\lambda)\,[1 - \exp(-\beta t)]\,\exp(-qL)\cos(qL)]\} + S_0\,(f, \lambda, R_1, R_2, R_3)$$

The out-of-phase (quadrature) signal can be given by the relation

$$[4b]\quad \mathrm{Im}\,[S(f,\lambda)] = -(1 - R_1)\,\{\eta(\lambda)\,[1 - \exp(-\beta t)]\,\exp(-qL)\sin(qL)\}$$

At infrared wavelengths, such that the photon energy is less than the gap energy, E_g, one can show that for the absorbing substrate the derivative of [4*b*] with respect to the modulation frequency is

$$[5a]\quad \frac{\mathrm{d}}{\mathrm{d}f}\mathrm{Im}\,(S) = J\frac{\mathrm{d}}{\mathrm{d}f}[\exp(-qL)\sin(qL)]$$

where J is a constant. The Im (S) passes through an extremum with respect to the modulation frequency if

$$[5b]\quad [\sin(qL) - \cos(qL)]\exp(-qL) = 0, \qquad qL = \frac{\pi}{4}$$

The second derivative of [4*b*] leads to the conclusion that the extremum is a minimum. By choosing $f_{\min}$ to correspond experimentally to condition [5*b*], then using the definition of $q(f)$:

$$[6]\quad \alpha = f_{\min}\left(\frac{16\,L^2}{\pi}\right)$$

and knowing the thickness of the sample, L, one can deduce the thermal diffusivity of the substrate from this relation.

By taking the ratio of the real and imaginary parts of S presented in [4*a*] and [4*b*] one can write

$$[7]\quad \frac{\mathrm{Re}\,[S\,(f,\lambda)]}{\mathrm{Im}\,[S(f,\lambda)]} = \cot(\Phi)$$

$$= -\cot(qL) - \frac{S_0\,(f, \lambda, R_1, R_2, R_3)}{\eta(\lambda)\,(1 - R_1)\,[1 - \exp(-\beta t)]\,W(f)}$$

where Φ is the synchronous signal phase (the difference between the sample and a suitable reference).

$$W(f) = \exp(-qL)\sin(qL)$$

depends on the working frequency and $\eta(\lambda)$ is taken as equal to unity, which is realistic when photon energies below the optical gap are involved, assuming sub-band-gap absorption to be primarily due to nonradiative defects (1, 4, 11). One can finally show that the optical absorption coefficient can be found from the following equation:

$$[8]\quad \beta(\lambda) = -\frac{1}{t}\ln\left[1 + \frac{S_0(\lambda)}{(1 - R_1)\,\{\cot[\Phi(\lambda)] + \cot(qL)\}\,W(f)}\right]$$

Note that in the spectral range of this study $\cot[\Phi(\lambda)]$ has a negative value. Equation [8] leads to the limit of zero β when Φ becomes zero, as expected. We note that $S_0(\lambda)$ can be measured from the transmission measurements (Fig. 1*b*) and $\Phi(\lambda)$ from the thermal-wave ones (Fig. 1*a*).

3. PPE spectroscopic instrumentation and methodology

In this section, the method of spectroscopic measurement that was used to study the composite amorphous silicon–crystalline silicon samples will be described. White light from a 1000 W Xe lamp was passed through a monochromator housing an infrared-blazed grating with a resolution of 16 nm, which was adjusted using a stepping motor (Step motor 2 in Fig. 2) (7) under IBM PS/2 computer control, to produce a beam with variable wavelength. The spectral cut-off filters associated with the monochromator were changed automatically using an electromechanical switch. Then the beam was focused and directed towards the sample or reference detector if so needed. Using an oscillating mirror (M) sometimes, a swept beam was alternately focussed on two spots on a plane where the sample and detector would ultimately be placed, if an independent reference cell for spectral throughput normalization were desired. At those spots the beam sizes were approximately 1 × 0.5 mm^2 each in area. For self-normalized spectra, an EG&G chopper (Model 192) was placed before the mirror to provide the modulation. The data acquistion software ensured that the intensity variation of the lamp during successive measurements of sample and reference did not contribute a considerable error. It is important to note that the two Al–Ni metallized PVDF photopyroelectric detectors were covered with black paint to absorb maximum optical flux (blackbodies) and exhibit saturated PPE spectra (12). The output signals were band-pass-filtered and preamplified by low-noise Ithaco preamplifiers (Model 1201). An IBM computer equipped with a mathematical coprocessor for fast calculations interacted with the two lock-ins through a general purpose interface bus (GPIB) board, and upon reading

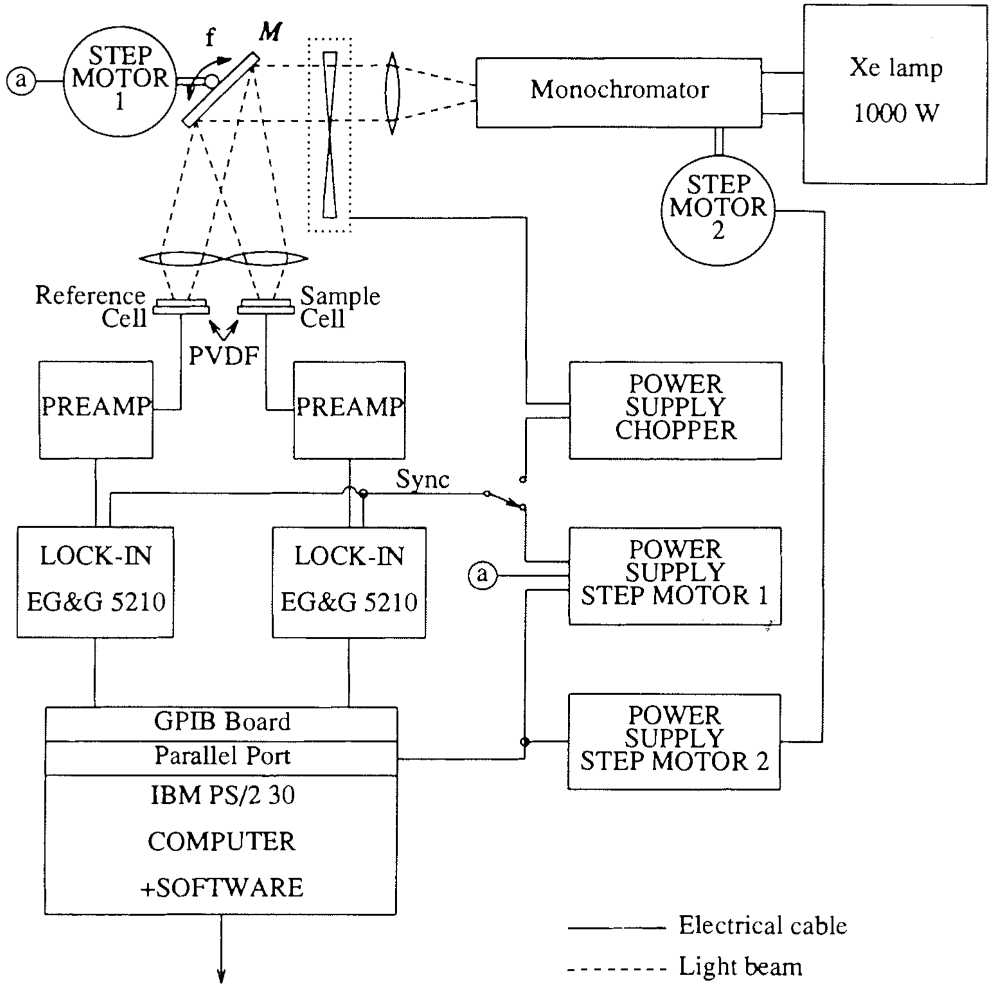

FIG. 2. Dual-channel photopyroelectric spectrometer instrumentation.

their output, it calculated in-phase and quadrature-signal components. Using only the two black PVDF films the quadrature channels were zeroed and the normalized magnitude signal was further calibrated (the amplitude ratio of the two signals: $S \rightarrow 1$) at the beginning of each experiment by a judicious choice of gain on the preamplifiers, before placing the sample on one of the PPE detectors. Two different configurations were used in our experiments as illustrated in Fig. 1. In the first (thermal-wave) configuration the sample was placed directly on the pyroelectric PVDF detector. In this geometry the detected signal is the sum of the optical and thermal components as was shown in the theoretical part. On the other hand, in the second (optical-transmission) configuration, the sample rested on a 200 μm thick metal ring support so as to be separated from the pyroelectric PVDF detector, while the intervening gas layer was thermally thick for all measurement frequencies. Consequently, the photopyroelectric signal produced in the second case was due solely to the optical transmission, as verified by the absence of out-of-phase signals in our working modulation frequency range 15–200 Hz.

4. Experimental results

For this study a few a-Si samples of thicknesses 500, 2100, 3700, and 8000 Å (1 Å = 10^{-10} m) deposited on Si were used. These samples were obtained from MITEL Semiconductor Division of Bromont Quebec. The thin films were deposited at 570°C using MITEL's low-pressure chemical vapour deposition (LPCVD) fabrication process. Note that this temperature is relatively high since it is close to the critical one (670°C) where the formation of polycrystalline silicon occurs (13). Some investigations on these thin films have shown the presence of the polycrystalline silicon. The samples themselves were cut in square shapes of dimensions 4×4 mm^2 to fit into the sample carrier. The substrates were crystal silicon wafers with nominal resistivities ranging between 2 and 4 Ω cm. The samples were put on the sample-cell detector (Fig. 2), and a gentle pressure was applied in order to have a good thermal contact with the black painted PVDF in the case of configuration A (see Fig. 1*a*). In case of configuration B, the sample was put on the ring spacer over the PVDF pyroelectric film (see Fig. 1*b*). Optical measurements were performed between 1000 and 1500 nm. Modulation frequency measurements were also performed in the range of 15–200 Hz and produced thermal information.

4.1. *Frequency dependent measurements*

Figure 3 shows the configuration A in-phase and quadrature signals as a function of the modulation frequency, *f*, from 15

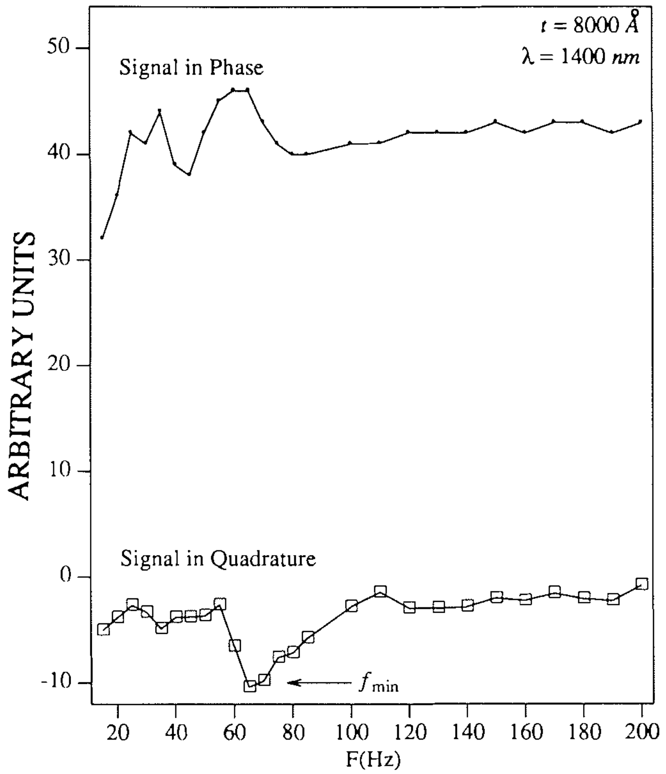

FIG. 3. Frequency dependence of the normalized in-phase and quadrature PPE signal in the configuration of Fig. 1*a*. Excitation source: 1400 nm from a 1000 W Xe lamp.

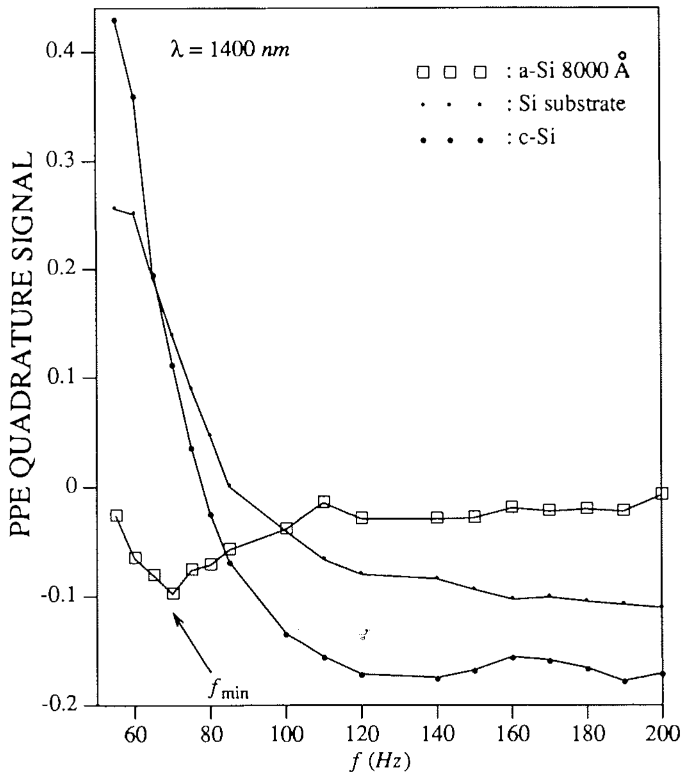

FIG. 4. Frequency dependence of the normalized photopyroelectric in-phase signal. (*a*) a-Si 8000 Å, (*b*) overturned substrate of a-Si exposed to light, (*c*) pure crystalline silicon. Excitation source: 1400 nm from a 1000 W Xe lamp.

to 200 Hz. These measurements were performed using 1400 nm excitation light to satisfy the infrared transparent-substrate condition presented in the theoretical part and were normalized by the reference PPE sensor signal. Indeed, as was expected, the quadrature signal exhibits a frequency minimum $f_{\min}$ close to 66 Hz. Use of [6] gives, for the thermal diffusivity of the substrate silicon, $\alpha \approx 0.94$ cm^2 s^{-1}. This value is in a good agreement with published values (14, 15). Before we discuss the optical information, it is important to note that this new way of finding the thermal diffusivity of semiconductors seems to be more accurate than the ones presented previously (6, 7). As was also expected from [4*a*], the in-phase signal presents a maximum at the frequency $f_{\min}$. It was noted, however, that these two curves cannot be fitted in detail by [4*a*] and [4*b*]. The in-phase signal is 45 to 60 times greater than the quadrature one, in agreement with earlier observation on powdered thin layers (3). The differences in magnitude of the signals shown in Fig. 3 are indicative of the effective background suppression afforded by quadrature detection in semiconductors. We note that the variations of these two signals at frequencies lower than 40 Hz cannot be considered, since that range approaches the thermally thin limit of the 52 μm PVDF detector. Similar experiments have been performed with pure c-Si to further test our model. In that case the in-phase and quadrature signals have also shown maximum and minimum values, respectively. These extrema, close to 66 Hz, were less pronounced ($\approx$30%) than those observed with the a-Si layer. We attributed these unexpected extrema to a possible damaged layer due to surface polishing. This hypothesis was confirmed when the experiment on the same sample was repeated after it was etched with HF for 90 s, in which case the extrema disappeared. In Fig. 4, we present the variation of the quadrature signal for three different experiments. We note that the quadrature signal presents a minimum only for the a-Si layer. This indicates that the crystalline Si and substrate-on-top (overturned sample) geometries did not conform to the response expected from a thin absorbing layer–transparent-substrate configuration, and the latter was exhibited only for the a-Si–substrate geometry.

4.2. *Transmission spectra*

Before performing any experiments, measurements were taken using only the two black PVDF films to calibrate the normalized magnitude signal and the phase difference of the two detectors. This calibration was necessary as was shown by Christofides *et al.* (7) because when one changes the working modulation frequency, the induced normalized signal may also change owing to small differences in the coatings of the two PVDF films.

Using the configuration B in Fig. 1*b*, measurements were made on two pure crystal silicon samples obtained from different wafers. At 1000 nm, no signal was produced owing to the opacity of the substrate. Consequently, we started the experiment at 1150 nm. The chosen modulation frequency was 45 Hz, to satisfy the thermally thick condition (12) for the 52 μm PVDF. Complete substrate transparency was considered to be at $\lambda > 1200$ nm, in which region no quadrature signal could be registered from a c-Si sample. As was expected, it has been found that the PPE signal S_0 increases as a function of wavelength. At a given wavelength, it was observed that the PPE signal due to transmission decreases when the thickness of the amorphous layer increases. Finally, the phase of all the

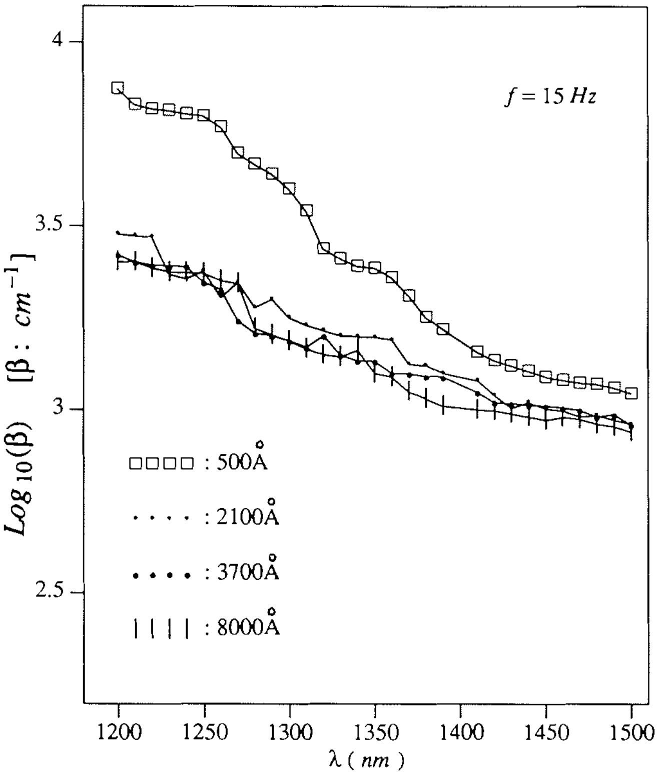

FIG. 5. Variation of the optical absorption coefficient β as a function of the wavelength for various amorphous layer thicknesses.

different pure c-Si samples was found to be constant as a function of the wavelength; i.e., there was no quadrature contribution from delayed synchronous thermal signals. As was shown before, the evaluation of the optical absorption coefficient as a function of λ cannot be found from the transmission measurements because the unknown coefficients R_2 and R_3 are present in the transmission equation.

4.3. *PPE spectra obtained from the thermal-wave mode*

Several photopyroelectric spectra have been obtained at 15 Hz. As was expected, the normalized signal S_T increased and the phase difference Φ decreased with increasing wavelength (note that Φ varies from negative values to zero). Similar spectra have been observed before (4). From close to opaque (1200 nm) to the transparent region (1500 nm) of the spectrum, the normalized signal increased ca. 1 order of magnitude and the phase difference increased from −5° to 0°. Under this condition cot (Φ) ≫ cot (*qL*) and then [8] can be written as:

[9] $$\beta(\lambda) = -\frac{1}{t}\ln\left\{1+\frac{S_O(\lambda)\tan[\Phi(\lambda)]}{(1-R_1)\,W(f)}\right\}$$

By combining thermal-wave and transmission-mode data as shown in Sect. 4.2 above, and by taking into account the calculated value for the thermal diffusivity α of silicon, the optical absorption coefficient of the amorphous layer sample was determined from [9]. In Fig. 5 we present the optical absorption spectrum of the amorphous layers obtained at 15 Hz. These spectra were obtained in a self-normalizing fashion by using the in-phase signal channel in the role of the normalizing spectrum, instead of the signal from the reference sensor. These two signals were found to be similar, as the thin surface-layer absorption, S_T, was essentially a negligible perturbation to the much stronger direct transmission, S_O. The calculated spectra β(λ) are of the same order of magnitude as the ones reported by Tanaka (16) and Freeman and Paul (17) which were obtained using different spectroscopic techniques.

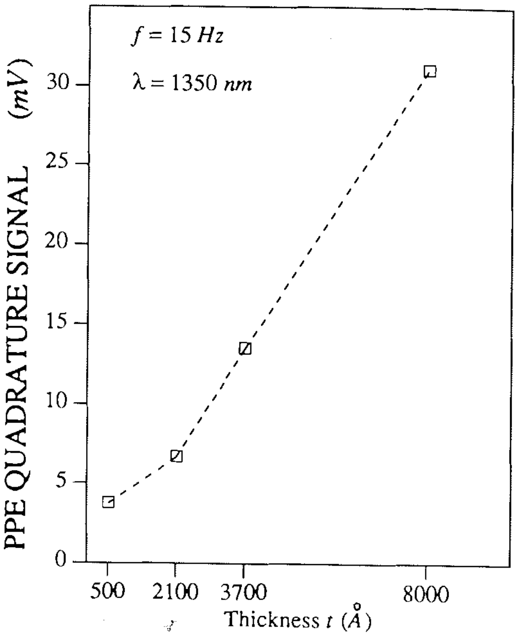

FIG. 6. PPE quadrature signal dependence on deposited layer thickness at 1350 nm irradiation. Modulation frequency 15 Hz.

We note that the optical absorption coefficient converges to the same value for all thicknesses especially at high wavelengths. The cutoff λ = 1200 nm for substrate transparency formed the criterion for treating our system in the geometry of Fig. 1*a*.

5. Discussion

As was shown, it is practically impossible to obtain the quantitative optical absorption coefficient spectrum from transmission spectra since it is not easy to determine the reflectivity between film and substrate. On the other hand, proper elimination of the large background by out-of-phase detection shows absorption spectra of the deposited thin-film amorphous Si closely resembling each other, Fig. 5. The noticeable trend to somewhat increased β(λ) with decreasing layer thickness is most pronounced with the 500 Å thick film and may be due to an increase in the importance of extraneous absorption at the interface between a-Si and the substrate. Any such effect could be due to defect densities at the interface (electronic or mechanical in nature) and appears to be less pronounced at wavelengths greater than 1360 nm. The sensitivity of the PPE quandrature technique is seen to be better than the minimum thickness detection in these measurements, for which

[10] $$[\beta(\lambda)]\,(t_{\min}) \approx 5 \times 10^{-3}$$

Figure 5 corresponds to an experimental sensitivity of Φ ≈ 0.1°, and is of the order of magnitude experimentally reported for transverse PDS sensitivities for applications to thin solid-film spectroscopy. According to Tanaka (16) with the PDS technique one can obtain sensitivities of $\beta t \approx 10^{-4}$ by immersing the sample in CCl_4. However, PDS becomes at least two orders of magnitude less sensitive if one uses air instead of CCl_4. This introduces a disadvantage for PDS with respect to PPES detection in air. Figure 6 shows the change of PPES signal with a-Si thickness for our samples and demonstrates the ability of the present technique to monitor layers even thinner than our minimum 500 Å. One advantage of PPES over PDS, in this context, lies in the avoidance of a liquid interface between the electronically sensitive microelectronic device structure and a fluid of high refractive-index gradient, $\partial n/\partial T$.

A further advantage of the present out-of-phase PPE determination of $\beta(\lambda)$ in the weak absorption regime, over earlier investigations (4, 5) is the elimination of the requirement for a reference-photon counting sensor for quadrature detection with a transparent substrate: spectral self-normalization can be efficiently performed by utilizing the in-phase signal as the normalizing reference.

6. Conclusions and future perspectives

In this work self-normalized, out-of-phase photopyroelectric spectroscopic detection has been demonstrated as a zero-background technique for characterizing amorphous layers of silicon of various thicknesses deposited on crystalline Si. We presented some results that led to the following conclusions:

(*i*) The dual channel and(or) self-normalized photopyroelectric approach has been shown to be sensitive for nondestructive evaluation of amorphous semiconducting thin-film layers of arbitrary thicknesses. The photopyroelectric spectroscopic technique seems to be promising in the quantitative characterization of amorphous layers, it is similar to PDS without the requirement for a solid–liquid interface.

(*ii*) The ability of the theoretical model presented in this paper to handle quantitative photopyroelectric spectroscopy of composite semiconducting samples was demonstrated in appropriate spectral ranges, via the extraction of $\beta(\lambda)$ spectra for thin solid films and the calculation of the thermal diffusivity of their substrates.

(*iii*) The thickness t of the amorphous layers did not influence greatly the optical absorption coefficient of our samples except at low-t values where interfacial absorption between thin layer and substrate appears to increase the average value of β at sub-band-gap wavelengths. This effect may be due to an increase in the importance of interfacial-defect densities in the overall value determination of the absorption coefficient of very thin layers.

(*iv*) PPE quadrature spectroscopy was shown to be far superior to conventional transmission spectroscopy at low energies, i.e., for $\beta(\lambda)t \ll 1$. The latter spectroscopy, however, proved useful in providing reliable values for interfacial reflection coefficients R_2, as inputs toward the quantitative determination of $\beta(\lambda)$ from out-of-phase PPES.

From the literature (15, 16) it appears that the best experimental sensitivity for transverse PDS applied to thin solid-film spectroscopy is $\beta t \approx 10^{-4}$; therefore, the present PPE technique provides a comparably powerful means for characterizing semiconducting thin films with distinct advantages. In this paper we have presented preliminary measurements on composite structures. A further enhancement of the sensitivity of PPES can be achieved, by increasing the signal-to-noise ratio via an increase in the thickness (i.e., decrease in capacitance) of the photopyroelectric detector. Given that the PPE detector can also be inserted into a cryostat to obtain combined optical and temperature information, the developed technique seems very promising for the characterization of implanted and annealed semiconductor thin layers, and the identification of de-excitation mechanisms contributing to defect-related absorption below the optical gap.

Acknowledgments

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