DETECTION OF THERMAL DIFFUSIVITY OF THIN METAL FILM USING PHOTOACOUSTIC FREQUENCY MODULATION LOCK-IN TECHNIQUE

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In a Frequency Modulation (FM) Photoacoustic Spectroscope (PAS) a Lock-in Analyzer is used as both correlator and vector analyzer, then the phase response of PA signal in the range of modulation bandwidth is obtained quickly and precisely. The thermal diffusivity of a sample can be detected by this method. The experimental results of thermal diffusivity for Ti films of several different thicknesses are presented and show that this technique is very efficient for measuring thin film's thermal diffusivity.

Dans le Spéctroscope Photoacoustique (PAS) de modulation par frequence, un analyseur de serrure en phase est utilisé en même temps comme corrélateur et analyseur de vecteur, ainsi on peut obtenir vitement et précisement la réponse de phase de signale photoacoustique dans la domaine de frequence de modulation. Utilisant cette methode la diffusivité thermale d'un echantillon peut être détecté. Le resulta experimentale pour les feuilles de Ti avec epaisseurs differente montre que cette techique est très efficace pour detecter la diffusivité thermale de feuille mince.

INTRODUCTION

In the field of today's microelectronics, surface physics, thin-film physics and functional layer material, there exists an increasing demend for detecting thermal diffusivities of thin film and sheet materials. The photoacoustic effect has recently received considerable attention as a promising tool for detecting thermal diffusivity, as the photoacoustic signal provides information about the optical and thermal properties of the sample. Various methods have been developed to measure thermal diffusivity by means of photoacoustic spectroscopy (PAS) [1-4]. However, these methods suffer from certain limitations, particularly in the case of thin-film samples. In this paper, we report a new method for detecting thermal diffusivity by using photoacoustic frequency modulation lock-in technique. Our experimental results have shown that this method is very

efficient for measuring thin film thermal diffusivity.

THEORY

Fig. 1. Diagram of a photoacoustic cell
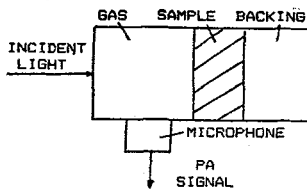
In Fig. 1, a periodically modulated light beam is incident on a sample enclosed in gas-filled cell. Optical absorption in the sample produces periodic heating, causing a pressure variation in the gas. The pressure fluctuation is detected with a microphone and the photoacoustic signal of the sample is obtained.

For samples having a large optical-absorption coefficeint, the theoretical expression for the relative phase-lag can be obtained from the Rosencwaig-Gersho theory[m], applied to front-surface illumination[m]. The expression is

$$\phi - \phi_r = -tg^{-1} \left[\frac{2RSin(2x)}{e^{2x} - R'e^{-2x}} \right] \qquad (1)$$

In Eq. 1, ϕ is the phase of the photoacoustic signal from the sample and ϕ_r is the phase of the photoacoustic signal from the reference sample. A sheet material with a roughly known low charactoristic frequency fc (fc= α / l^2, where α and l are the thermal diffusivity and thickness of the sheet respectively) is used as reference because its theoretical photoacoustic phase is constant for modulation frequency greater than 2.5fc and equal to $-\Pi/2$. x is defined by

$$x = ls \left(\frac{\Pi f}{\alpha s} \right)^{1/2} \qquad (2)$$

where f is the modulation frequency(f>2.5fc), ls is the sample thickness, and αs is the sample thermal diffusivity. R is defined by

$$R = \frac{\rho_b C_b \rho_s C_s \alpha_s' - K_b'}{\rho_b C_b \rho_s C_s \alpha_s' + K_b'} \qquad (3)$$

where ki, ρi and Ci are the thermal conductivity, the density and the thermal capacity at constant pressure of material i, for backing material (i=b) and for sample (i=s). For air backing, R is approximate to 1.

In fact, Eq. 1 implies that identical non-sample related parameters are needed for sample and reference sample. In an actual photoacoustic measurement, there exist some experimental uncertainties such as the geometry of the photoacoustic cell for different sample. The uncertainty causes a phase translation of photoacoustic signal (this has been demonstated by our experiment). Therefore, the actual measured phase-lag satisfies the following equation:

$$\phi - \phi_r = -tg^{-1} \left[\frac{2RSin(2x)}{e^{2x} - R'e^{-2x}} \right] + C \qquad (4)$$

where C is the numerical constant of phase translation, X and R are the same as that in Eq. 1.

When K_b, C_b, ρ_b, C_s, ρ_s and ls are known, from the experimental values of $(\phi - \phi_r)$ for several modulation frequencies we can obtain αs by data fit to Eq .4 by optimizing free parameter αs and C. When K_b, C_b, ρ , C_s. and ρ_s are unknown, let $K_b{}^s/(\rho_b C_b P_s C_s)=K($ namely $R=(\alpha s^s-K)/(\alpha s^s+K))$, and αs can be obtained by using data fit to Eq. 4 by optimizing free parameter αs, K and C.

EXPERIMENT

Our experimental setup is schematically shown in Fig. 2. A He–Ne laser is used as an excitation light source. A acousto–optic modulator drived by a rf driver is used to modulate the laser beam. The first–order laser beam with great modulation depth provided by the acousto–optic modulator is directed to the front side of the photoacoustic cell. By feeding the rf driver a frequency linear–modulated sine wave signal provided by a function generator (HP.MODEL 3325A), the frequency linear–modulated photoacoustic signal of sample is produced. The photoacoustic signal is detected by a lock–in analyzer (EG&G.PAR.MODEL 5204) whose reference signal is the same sine wave signal provided by the function generator. The lock–in analyzer is used as both correlator and vector analyzer which outputs the phase of the photoacoustic signal. The phase signal is fed into a data acquisition system consisting of a microcomputer and an analog – digital converter. In order to precisely record the phase response of the photoacoustic signal in the range of frequency modulation bandwidth, the data acquisition system is interfaced to the sweep–initiating impulse of the function generator.
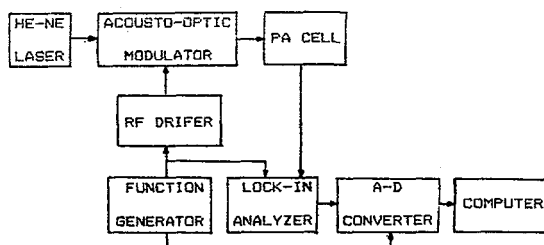

Fig. 2. Experimental set–up

In our experiment, the power output of the He–Ne laser is 15mW, and the first–order laser beam has a power of 8 mW. A 2.22–mm–thick pure Cu sheet (fc=22Hz)is used as the reference sample. The samples are thin Ti films of thicknesses 30, 50 and 70 μm (ρ s=4.54×10' kg/m', Cs=522 J/kg.'k) . To prevent from the drum effect in photoacoustic cell, the sample is fixed to a glass backing (K,=0.9w/m.s.'k, ρ ,=2.5×10'kg/m', C,=0.84×10'J/kg.'k).The
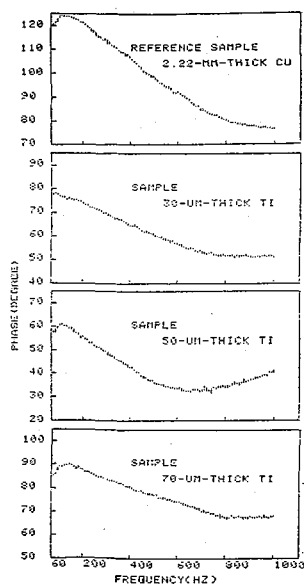
Fig.3.Phase of PA signal vs modulation frequency

FM bandwidth is from 60Hz to 1000 Hz .The sweep time is 9s.The measured phase response of the photoacoustic signal over the modulation range is shown in Fig.3.

A computer program based on Eq.4 is developed to fit the experimental data of the phase-lag.The results of the fit are listed in Table 1,where the literature value of α, is also listed for comparison.

Table 1.Experimental results of Ti filmsamples

ls(μm)	C	$\alpha s(cm^2/s)$	Fitting Error
30	6.9140×10^{-1}	0.0945	17.6575×10^{-1}
50	0.4543	0.1067	6.6665×10^{-1}
70	8.7950×10^{-1}	0.1055	1.3833×10^{-4}

Literature value of αs :0.08cm^2/s$^{(9)}$

CONCLUSIONS

The good agreement between our obtained values of Ti film thermal diffusivity and the literature value and the reproducibility of our results have shown that the photoacustic frequency modulation lock-in technique is a very effective techique for detecting thin film thermal diffusivity. In addition to the well-known properties of the photoacoustic technique (its nondestuctive aspect,its sensitivity,its capability of allowing the study of opaque samples and its advantage of being contactless),with our data acquisition and handling, the present technique have the ability of obtaining the thermal diffusivity of a thin metal film quickly and precisely. Moreover, the technique used here is also suitable for other film and sheet materials.

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