PCLT/P(VDF-TrFE) Nanocomposite Pyroelectric Sensors

Qing-qi Zhang, Helen Lai Wa Chan, Bernd Ploss, and Chung Loong Choy

Abstract—**Thin composite films consisting of 12 vol% of nanosized lanthanum and calcium-modified lead titanate (PCLT) powder embedded in a vinylidene fluoride-trifluoroethylene copolymer [P(VDF-TrFE)] matrix were deposited on silicon (Si) substrates to form pyroelectric sensors with three different configurations. The influences of a thermal buffer layer and back etching of the silicon substrate on the current and voltage responsivities of the sensors were investigated theoretically and experimentally. The specific detectivity of the sensors was also calculated from the measured voltage responsivity and noise.**

I. Introduction

Studies on ferroelectric ceramic/polymer composites were initiated in the 1970s, but most of the composites investigated consist of a passive polymer matrix, such as silicone rubber or epoxy [1]–[5]. P(VDF-TrFE) is a ferroelectric copolymer that has attracted special attention in integrated pyroelectric sensor and array applications [6]–[9]. When the VDF content is in the range of 20 to 50 mol % , the copolymer crystallizes in a polar β form when prepared from the melt or from solution. Hence, P(VDF-TrFE) thin films can be deposited on substrates by spin-casting and then poled in situ to yield high pyroelectric activity. Compared with the sintering of ceramic films, fabrication of P(VDF-TrFE) films is a low temperature process that is more compatible with Si technology. By incorporating nanosized ferroelectric ceramic powder in a P(VDF-TrFE) matrix, composite thin films with both phases ferroelectric can be produced. The advantage of these nanocomposites is that the established low-temperature technology for P(VDF-TrFE) can be directly applied to fabricate pyroelectric sensors. The pyroelectric coefficients of the ceramic and copolymer phases have like sign, and the piezoelectric coefficients have opposite sign; therefore, when the two phases are poled in the same direction, the pyroelectric activities of the two phases are reinforced, and their piezoelectric activities are partially cancelled [10], [11], reducing the vibration-induced electrical noise in the sensor. PCLT was reported to have better pyroelectric properties than lead titanate [12]; hence, PCLT-nanosized powder was prepared by a sol-gel process [13] and used in the fabrication of composites.

In this study, thin PCLT/P(VDF-TrFE) nanocomposite sensors with 12% volume PCLT were deposited on Si substrates by the spin-coating method, and the PCLT and P(VDF-TrFE) phases were poled in the same direction to obtain reinforced pyroelectric properties. Because the Si substrate has a high thermal conductivity and heat capacity compared with the thin composite film, it served as a heat sink, thereby reducing the pyroelectric signal amplitude. To improve the performance of the sensor, it is necessary to reduce the heat flow from the pyroelectric layer to the Si substrate, either by inserting a polyimide thermal buffer layer between the pyroelectric layer and the Si substrate or by back etching the Si wafer.

II. PCLT Powder Prepared by the Sol-Gel Technique

To ensure that the composite films are homogeneous, the inclusions should have as small a size as possible. The conventional mixed oxide method cannot be used, as the resulting powder size is too large. Hence, nanosized $[(Pb_{0.88}Ca_{0.04}La_{0.08})Ti_{0.98}O_3]$ powder was prepared by the sol-gel technique using lead acetate trihydrate, lanthanum nitrate, and calcium acetate as the precursors [13]. The size of the powder can be controlled by varying the annealing temperature T_a because the size increases as T_a increases. However, if the size of the powder is lower than a critical value, the ceramic powder will not have good pyroelectric properties. A study of the X-ray diffraction (XRD) patterns of PCLT powder annealed at various temperatures revealed that the (002) and (200) peaks were clearly separated when the powder was annealed at 850°C, indicating that the powder is in a ferroelectric tetragonal phase. Hence, in this work, PCLT powder annealed at 850°C was used. The powder had an average crystallite size of 45 nm as determined by XRD and an average particle size of 80 nm as determined by a particle size analyzer.

III. Fabrication of PCLT/P(VDF-TrFE) Composites

P(VDF-TrFE) 70/30 mol % copolymer [Curie transition temperature (T_{ch}) = 105°C upon heating and $T_{cc} = 70°C$ upon cooling] supplied by Piezotech (St. Louis, France) was used as the matrix. One gram of P(VDF-

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The authors are with the Department of Applied Physics, The Hong Kong Polytechnic University, Hunghom, Kowloon, Hong Kong (e-mail: apahlcha@polyu.edu.hk).

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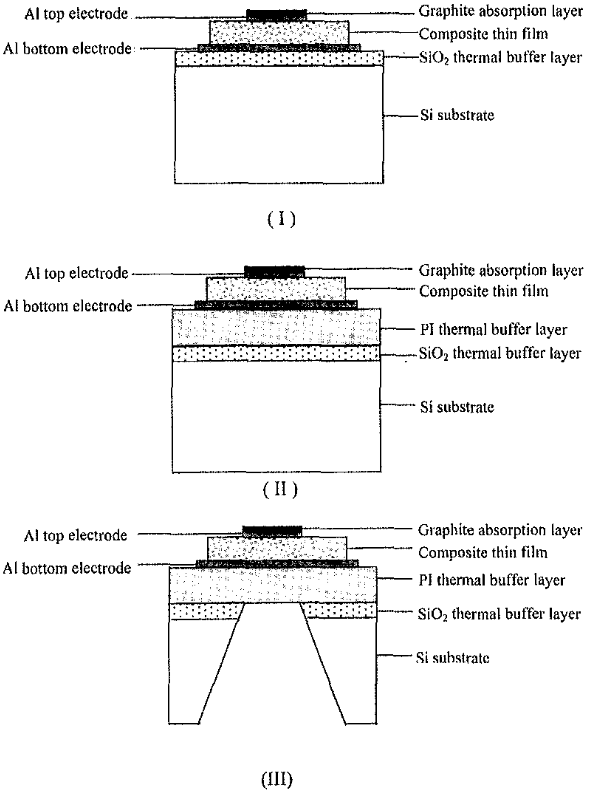

Fig. 1. Schematic diagrams of PCLT/P(VDF-TrFE) nanocomposite pyroelectric sensor configurations.

TrFE) was dissolved in 9 mL of MEK/ethanol mixture to form a solution with about 10 wt% of P(VDF-TrFE). A mixture of methyl-ethyl-ketone (MEK) and ethanol (66/34 vol%) was used instead of MEK because this resulted in a better dispersion of the PCLT powder [14]. A suitable amount of PCLT powder was then added, and the mixture was ultrasonically agitated for several hours to break up the agglomerates and to disperse the ceramic powder uniformly in the copolymer solution. A sensor with 0.12 volume fraction of PCLT and thickness 3.5 μm was prepared by spin-coating (1600 rpm for 1 min) the composite suspension onto a ⟨100⟩ Si substrate previously coated with an aluminum (Al) electrode (~0.6 μm thick). The film was annealed at 130°C for 2 h to remove the solvent. An Al top electrode (~0.6 μm thick) of area 8 mm^2 was then thermally evaporated onto the top surface of the film to produce the desired capacitor structure.

The composite film was subjected to a dc field of 50 MV/m for 1 h at 115°C, (above T_{ch}) to polarize the ceramic phase [15]. The electric field was kept on when cooling to room temperature so that the copolymer phase was polarized in the same direction as the ceramic phase. The composite was reheated to 95°C and poled under a dc field of 50 MV/m for another 2 h to polarize the copolymer fully. The poled sample was short-circuited and annealed at 60°C for 12 h to reduce the contribution of thermally stimulated current in subsequent pyroelectric measurements.

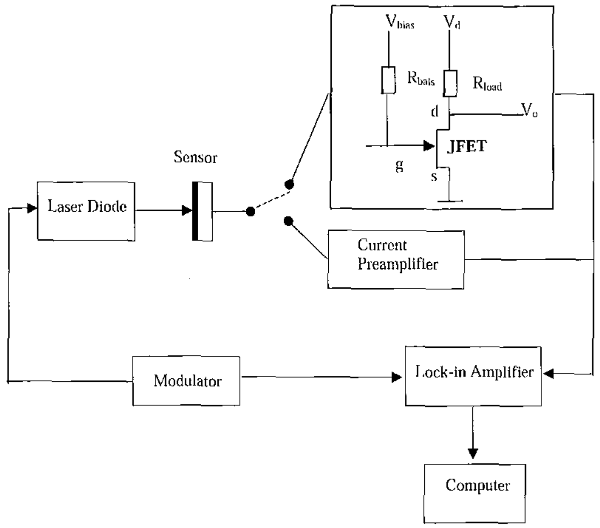

Fig. 2. Schematic diagram of the experimental setup for current or voltage responsivity measurements.

To calculate the current and voltage responsivities of the sensor, the specific heat and thermal diffusivity of the sensor material must be known. Because the thickness of the sensor is too small for accurate thermal diffusivity measurements, a series of composite samples with thickness of about 0.2 mm and ceramic volume fraction $\phi = 0.09$–0.36 were prepared for density, specific heat, and thermal diffusivity measurements. To fabricate these samples, a composite suspension was poured onto a petri dish and allowed to dry; then, it was compression molded at 170°C into thick films.

IV. PHYSICAL PROPERTIES OF PCLT/P(VDF-TRFE) COMPOSITES

The specific heat c of the thick composite films was measured by a Perkin Elmer DSC7 differential scanning calorimeter. The thermal diffusivity D was measured by the flash radiometry technique [16], and the density ρ was measured by hydrostatic weighting. These data, together with the volume-specific heat (ρc) and thermal conductivity (κ = ρcD), are given in Table I. It is seen that the density of the composites increases, while the specific heat decreases, as ϕ increases, resulting in a relatively constant volume-specific heat. Both the thermal diffusivity and thermal conductivity increase as ϕ increases. The thermal data required for calculating the response of the thin film sensor ($\phi = 0.12$) was obtained by interpolating the data for the thick films. These are given in Table II together with other data taken from literature.

As polyimide (PI) was used as a thermal buffer layer in the pyroelectric sensor, its thermal properties were also required in the calculation of sensor response. The poly-

TABLE I

DENSITY ρ, SPECIFIC HEAT c, VOLUME-SPECIFIC HEAT (ρc), THERMAL DIFFUSIVITY D, AND THERMAL CONDUCTIVITY κ (ρcD) OF P(VDF-TRFE) 70/30 MOL % COPOLYMER AND PCLT/P(VDF-TRFE) 0-3 NANOCOMPOSITES. ALL DATA WERE OBTAINED AT ROOM TEMPERATURE.

PCLT volume fraction ϕ	0	0.09	0.15	0.23	0.36
Density ρ (kgm^{-3})	1920	2395	2725	3165	3880
Specific heat c (Jkg^{-1}K^{-1})	1040	888	796	697	585
Volume-specific heat (10^6 Jm^{-3}K^{-1})	2.00	2.13	2.17	2.21	2.27
Thermal diffusivity D (10^{-6} m^{2}s^{-1})	0.101	0.103	0.120	0.148	0.202
Thermal conductivity κ (Wm^{-1} K^{-1})	0.202	0.218	0.261	0.327	0.459

TABLE II

MATERIAL PARAMETERS USED IN THE MODELING OF PYROELECTRIC SENSORS. PARAMETERS FOR THE COMPOSITE ARE INTERPOLATED FROM THE DATA IN TABLE I.

Material	Thermal conductivity κ (Wm^{-1}K^{-1})	Specific heat c (Jkg^{-1}K^{-1})	Density ρ (kgm^{-3})	Thickness (μm)
Al*	237	896	2707	0.6
Composite	0.239	840	2560	3.5
PI	0.253	981	1356	2 ~ 8
SiO$_2$*	1.3	750	2200	1.5
Si*	149	703	2330	380

*Taken from [18]–[21].

imide used in the present study was supplied in liquid form by Shanghai Jiaotong University, China. A PI sample of diameter 12 mm and thickness 0.2 mm was used in the thermal diffusivity measurement. The thermal data for PI are also given in Table II.

The relative permittivity ε at 1 kHz and the pyroelectric coefficient p of the poled composite with $\phi = 0.12$ were measured by a HP 4194A impedance analyzer and by the charge integration method, respectively [17]. The relative permittivity and pyroelectric coefficient were found to be 14 and 50 μC/m^{2}K, respectively.

V. FABRICATION OF NANOCOMPOSITE PYROELECTRIC SENSORS

Three configurations of single-element pyroelectric sensors have been fabricated (Fig. 1). The sensing element is a 3.5-μm thick PCLT/P(VDF-TrFE) nanocomposite with ceramic volume fraction $\phi = 0.12$. Configuration I consists of a sensing element deposited on a 380-μm thick Si wafer previously coated with 1.5-μm thick Si dioxide and 0.6-μm thick Al electrode. Configuration II is similar to I, except that an additional 8-μm thick PI layer is deposited between the sensing element and the Si substrate. Configuration III has the same structure as configuration II, but with the Si substrate etched away (at 70°C in a 20% KOH solution). An absorption layer was produced by spraying graphite on the top electrode of the sensor to increase the absorption of incident energy.

VI. CURRENT AND VOLTAGE RESPONSIVITIES OF THE SENSORS

The current and voltage responsivities R_i and R_v of the single-element pyroelectric sensors were measured in the range of 1.1 Hz to 10 kHz using the experimental setup shown in Fig. 2. The graphite absorption layer was irradiated by a modulated beam from a laser diode (wavelength $\lambda = 684.4$ nm) equipped with a modulator. The amplitude of the incident power (P_0) was 1.7 mW, and 90% absorption by the graphite layer was assumed. The output of the sensor was connected either to a current amplifier (for current measurement) or to a JFET (for voltage measurement) and fed to a lock-in-amplifier and then to a computer. The voltage noise ΔV_N of the sensors was also measured using the same experimental setup but with the laser switched off.

The current and voltage responsivities R_i and R_v of a single-element pyroelectric sensor are given by [19], [22], [23]:

$$R_i = \frac{I_0}{P_0} = \frac{pA\omega\overline{T}}{P_0} \tag{1}$$

$$R_v = \frac{V_0}{P_0} = \frac{g_m R_{load} R_E}{(1 + \omega^2 R_E^2 C_E^2)^{1/2}} R_i \tag{2}$$

where I_0 and V_0 are the amplitudes of the output current and output voltage, respectively; P_0 is the amplitude of incident power on the sensor area A; $\overline{T}$ is the spatially averaged temperature amplitude in the sensor layer; ω is the modulation angular frequency; and p is the pyroelectric

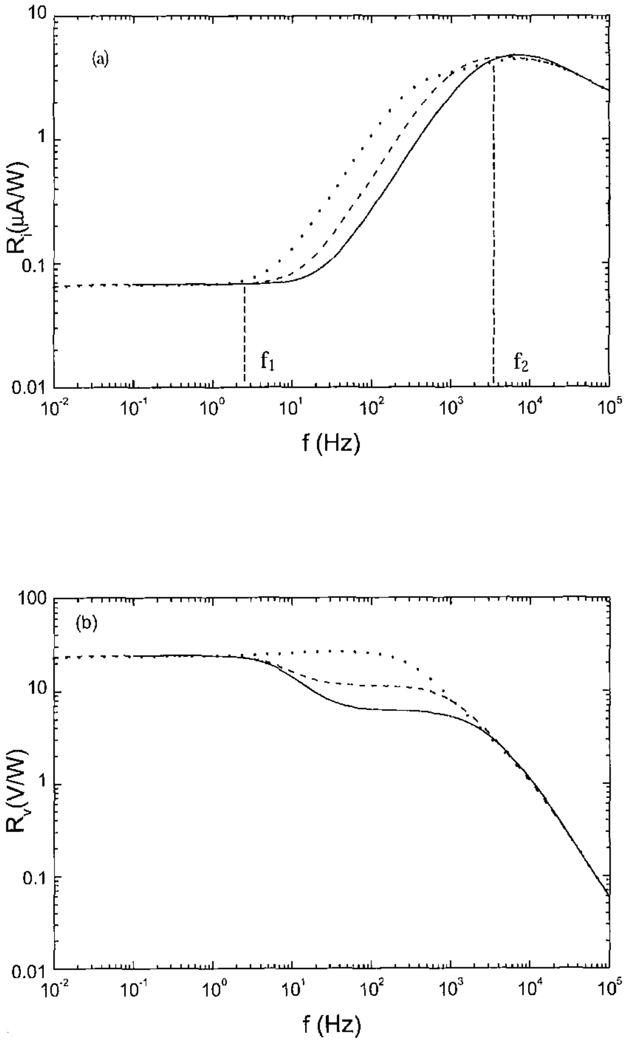

Fig. 3. Calculated R_i (a) and R_v (b) of the sensors with configuration I and II. Solid curve: configuration I; dash curve: configuration II, thickness of PI = 2 μm; dotted curve: configuration II, thickness of PI = 8 μm.

coefficient of the composite sensor ($p = 50\ \mu Cm^{-2}K^{-1}$), $R_{load} = 10\ k\Omega$, and $R_{bias} = 68\ M\Omega$ are the external resistors used in the voltage measurements [Fig. 2]); R_E is the equivalent resistance of the composite and R_{bias} ($R_E \approx R_{bias} = 68\ M\Omega$); C_E (283 pF) is the equivalent capacitance of the composite and the JFET (2N4861); and g_m (0.525 mA/V) is the mutual conductance of the JFET.

A. Calculated Current and Voltage Responsivities

The current responsivity R_i and voltage responsivity R_v were calculated in terms of (1) and (2) using the temperature change $\overline{T}$ calculated by solving a one-dimensional heat diffusion equation for a multi-layer system [18], [20], [21].

R_i and R_v are affected very little by the SiO_2 layer because it is very thin and has a high thermal conductivity compared with the composite (Table II). The dependencies of R_i and R_v on the modulation frequency are shown (as solid lines) in Fig. 3(a and b), respectively. From the R_i curve, three frequency regimes separated by f_1 and f_2 can be identified. The thermal diffusion length in the composite layer L is given by

$$L = (D/\pi f)^{1/2} \tag{3}$$

where D is the thermal diffusivity of the composite. In the frequency range $f < f_1$, L is so large that the sensor film and substrate are heated uniformly. R_i is determined mainly by the thermal capacity of the substrate and is therefore constant. In the frequency range $f > f_2$, L is smaller than the thickness of the sensing layer, so the thermal wave does not fully penetrate the sensing layer. As the frequency increases further, the decrease in L above about 10 kHz implies that the relative amount of thermal energy remaining in the Al top electrode increases; therefore, R_i decreases. In the frequency range $f_1 < f < f_2$, which is usually used for infrared detection, R_i falls off with decreasing frequency.

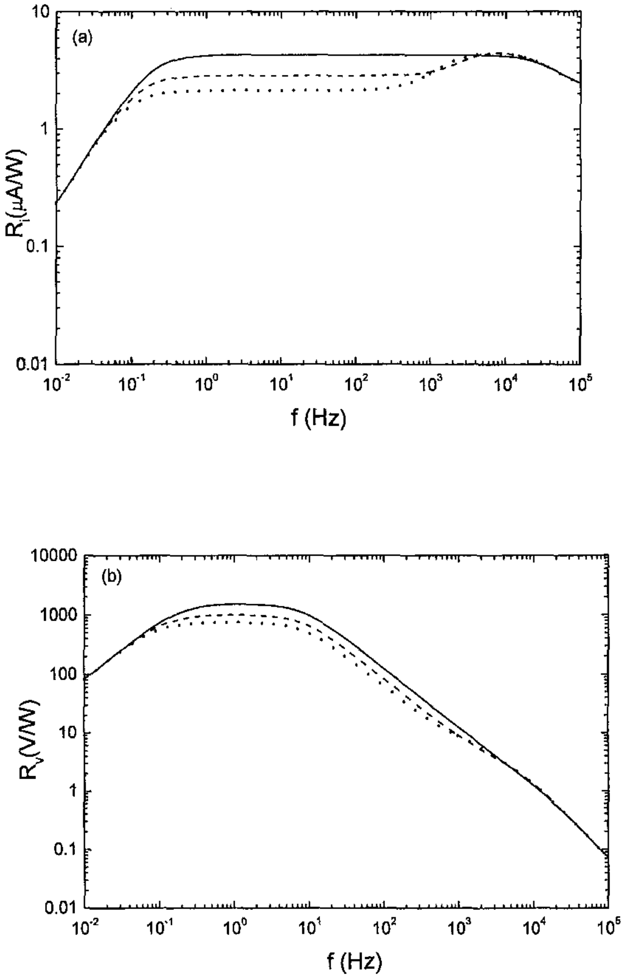

Fig. 4. Calculated R_i (a) and R_v (b) of the sensor with configuration III. Solid curve: thickness of PI = 0 μm, dashed curve: thickness of PI = 4 μm; dotted curve: thickness of PI = 8 μm.

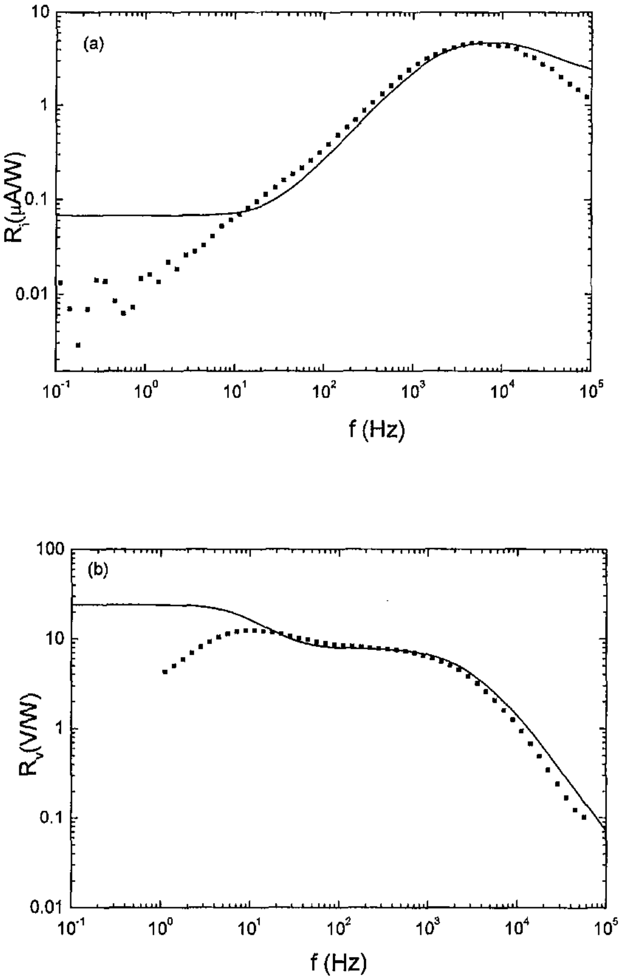

Fig. 5. Measured (points) and calculated (line) R_i (a) and R_v (b) of the sensor with configuration I.

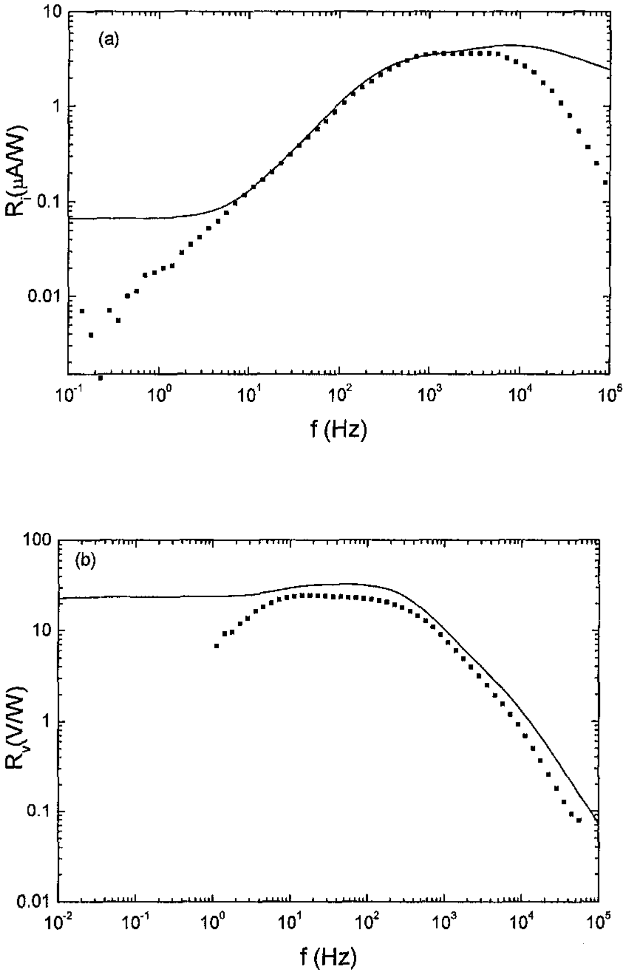

Fig. 6. Measured (points) and calculated (line) R_i (a) and R_v (b) of the sensor with configuration II . Thickness of PI = 8 μm.

This is because, as the frequency decreases, the thermal wave penetrates more and more into the substrate. In this frequency range, the introduction of a thermal buffer layer can improve R_i.

Fig. 3(a and b) also shows the effects of the addition of a PI thermal buffer layer. In the frequency range between f_1 and f_2, R_i and R_v increase significantly with increasing PI thickness. This arises because the PI layer can effectively decrease the heat flow into the Si substrate.

The influence of the Si substrate can be eliminated by back etching the silicon (configuration III in Fig. 2). For this case, R_i [Fig. 4(a)] and R_v [Fig. 4(b)] have been calculated for three different thicknesses of PI layers (solid line: 0 μm, dash line: 4 μm, dotted line: 8 μm). R_i decreases with increasing thickness of the PI layer because part of the thermal energy is absorbed by the PI layer. However, an appropriate thickness of PI provides good support to the pyroelectric sensing layer, thereby increasing the robustness of the sensor and preventing the membrane structure from being damaged.

B. Measured Responsivities and Specific Detectivity

Fig. 5 through 7 show a comparison of the measured and calculated responsivities. It is observed that the measured values agree well with the calculated curves except at low and high frequencies. In the calculation, lateral heat flow was neglected, and a one-dimensional thermal diffusion equation was used. The discrepancy between theory and experiment at low frequencies is due to the neglect of the lateral heat flow. Because of the small thermal capacity and thickness of the absorption layer, it was also neglected in the calculation. This results in considerable error at high frequencies because the thermal diffusion length is very small in this frequency range.

The specific detectivity D^* $(A^{1/2}R_v/\Delta V_N)$ has been calculated from the measured voltage responsivity R_V, voltage noise ΔV_N, and area of the pyroelectric element A; the results are shown in Fig. 8. It is observed that D^* has a maximum value of 1.3×10^7 cmHz$^{1/2}$/W at about 1 kHz for the sensor with configuration I, and 2.1×10^7 cmHz$^{1/2}$/W at about 300 Hz for the sensor with configuration II. It

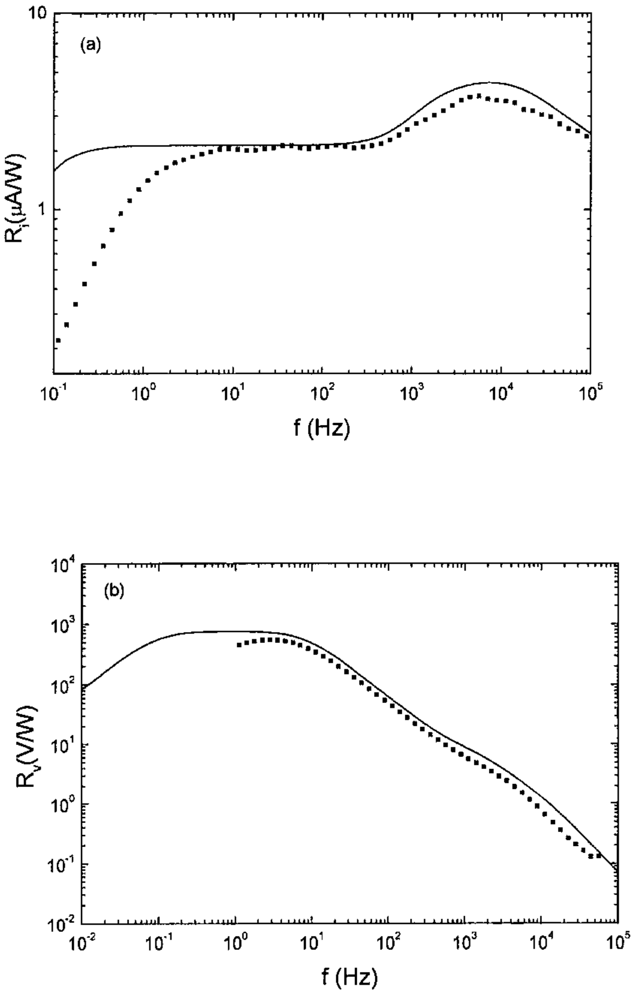

Fig. 7. Measured (points) and calculated (line) R_i (a) and R_v (b) of the sensor with configuration III . Thickness of PI = 8 μm.

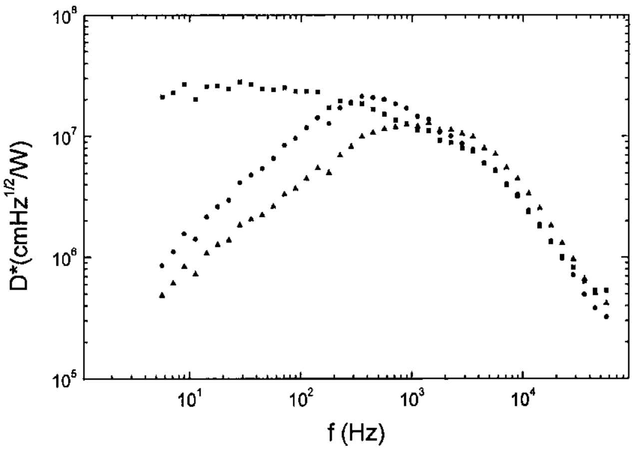

Fig. 8. Specific detectivity D* of the sensors with configuration I (triangle), II (circle), and III (square) calculated from the measured voltage responsivity and noise.

is also observed that, between 5 and 100 Hz, the sensor with configuration III has a constant D* value of 2.8×10^7 $cmHz^{1/2}/W$, which is much higher than those of the sensor with configuration I or II. It has been reported that D* for a PVDF sensor element in an array on a Si substrate at a modulation frequency of about 25 Hz lies between 10^6 and 2×10^7 $cmHz^{1/2}/W$ [7], [8], [24]. The comparatively high D* values of our sensors indicate that PCLT/P(VDF-TrFE) nanocomposites have good potential for use as pyroelectric sensors.

VII. CONCLUSIONS

Single-element pyroelectric sensors with three different configurations have been fabricated using PCLT/P(VDF-TrFE) nanocomposites as sensing elements. The measured voltage and current responsivities of the pyroelectric detectors show good agreement with the theoretical predictions obtained by solving a one-dimensional thermal diffusion equation for a multi-layer system.

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Qing-qi Zhang was born in Wuhan, China in 1962. She received her B.Sc. and M.Sc. degrees in physics from Hubei University in 1984 and Wuhan University in 1990, respectively, and her Ph.D degree in applied physics from the Hong Kong Technical University in 1999. She is currently working as a postdoc in the Materials Research Laboratory of The Penn State University. Her research fields are ferroelectric films and their applications in MEMS.

Helen Lai Wa Chan was born in Hong Kong in 1948. She received the B.Sc and M.Phil. degrees in physics from the Chinese University of Hong Kong in 1970 and 1974, respectively, and received the Ph.D. degree in physics from Macquarie University, Australia in 1987.

Dr. Chan worked as a research scientist at CSIRO Division of Applied Physics in Sydney, Australia for 4 yr and was responsible for setting up the standards for medical ultrasound in Australia. She then worked at GEC-Marconi Pty. Australia for a year as a senior acoustic designer before she returned to Hong Kong in 1992. She is currently a professor in Applied Physics at the Hong Kong Polytechnic University.

Bernd Ploss was born in Calw, Germany in 1955. He received the Dipl.-Phys. and Dr. rer. nat. degrees from the University of Karlsruhe in 1981 and 1987, respectively, and, in 1997, he became Privatdozent. He worked as an academic staff at the Institute of Applied Physics, University of Karlsruhe from 1982 to 1997, and, since 1987, has lead a research group in the field of ferroelectric materials and their application in sensors.

Dr. Ploss was a guest professor at the Ecole Nationale Superieure de Physique de Grenoble, France in 1996. From 1995 to 1999, he was the Chairman of the association "Dielectric Materials" of the German Physical Society (DPG). He is presently Associate Professor in the Department of Applied Physics at the Hong Kong Polytechnic University.

Chung Loong Choy was born in Malaysia in 1938. He received the PhD in physics from Rensselaer Polytechnic Institute in 1968 and then worked as a research associate for 1 yr at Cornell University. He was a visiting scientist at the University of Leeds and the University of Massachusetts in 1974 and 1981, respectively. His main research interest is in the structure and physical properties of polymers and composites.

Dr. Choy is presently Chair Professor and Head of Applied Physics at the Hong Kong Polytechnic University.