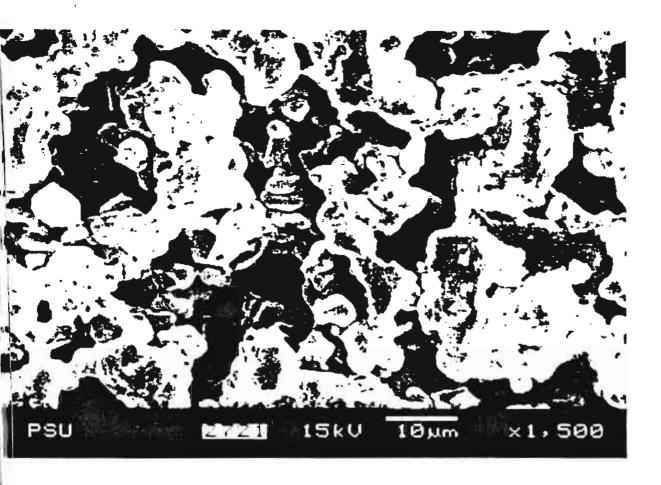
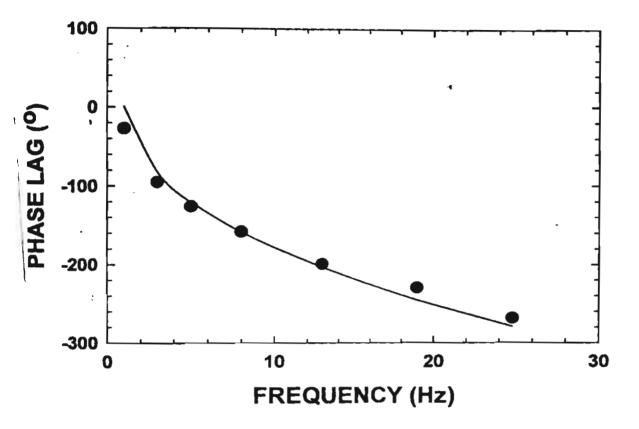
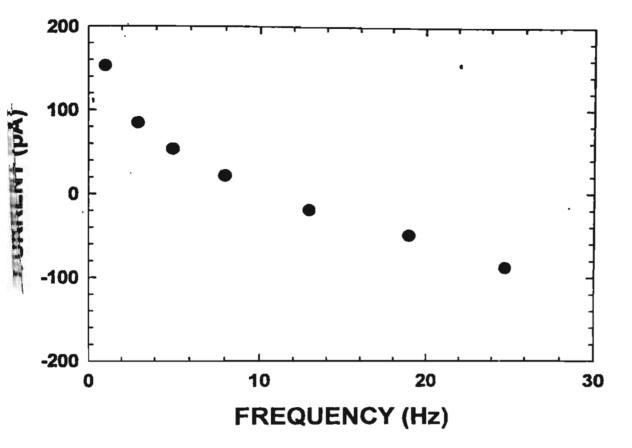


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PIEZOELECTRICITY OF 1-3 PZT/EPOXY COMPOSITES

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Abstract

The piezoelectric and pyroelectric properties of 1-3 type PZT/epoxy composites have been investigated. These materials have important advantages for a wide range of applications. The piezoelectric strain d coefficient for the poled samples was determined using the interferometric techique. It is found that the d_{33} values are 190 and 188 pm/V for the 0.4 and 0.6 volume fractions of PZT, respectively. The pyroelectric coefficient for samples with 0.4 and 0.6 volume fractions of PZT was measured and compared with that of the PZT. It is found that the coefficients of the composites increased with the ceramic content and reached values as large as 54 μ C/m²/°C. Because these materials have good promise for applications such as pyroelectric detectors, the thermal diffusivity of the composites is also being determined. The data reported in this work might serve as a guide for development of specific applications of the composite.

Introduction

There have been a numbers of investigations of the piezoelectric and pyroelectric properties of ceramic/polymer composites with different connectivities and volume fractions of the ceramic phase. For the 1-3 type PZT/epoxy, both the piezoelectric and pyroelectric activities depend on the polarization of the ceramic phase. Inhomogeneous displacement profiles under an electric field for a single-rod ceramic embedded in the epoxy matrix were theoretically studied and the effective piezoelectric coefficients have been derived [1]. The most commonly used piezoelectric coefficient d is defined by

$$d = \left(\frac{\partial S}{\partial E}\right) \tag{1}$$

where S is the strain and E the electric field.

A pyroelectric material exhibits a spontaneous polarization in the absence of an electric field [2]. A variety of new pyroelectric materials can be made by combining a ferroelectric ceramic with a passive polymer phase. A ceramic/polymer composite of the 1-3 type is an important structure, providing a new range of material properties. Moreover, by selection of the ceramic, polymer, proportions, and spatial scales, these material properties can be tailored to specific device requirements such as infrared sensors and thermal imaging devices [3,4]. Pyroelectricity originates from the temperature dependence of the spontaneous polarization. Variations of spontaneous polarization produce a displacement current parallel to the polar axis described by

$$\rho = \frac{I}{AdT \mid dt} \tag{2}$$

where p is the pyroelectric coefficient. A is the surface area normal to the polar axis, and $dT \cdot dt$ is the rate of change of temperature with time. An alternative form of (2) is [5]

$$\rho = \frac{(\Delta Q / A)}{\Delta T} \tag{3}$$

where ΔQ is the change of total charge due to a change in sample temperature ΔT . Equation (3) is more appropriate for the case of polymer pyroelectrics in which a change in surface area may be caused by a temperature change. Therefore, when the sample is heated or cooled at a constant rate, the pyroelectric coefficient can be found from a measurement of the charge produced.

Preparation of 1-3 PZT/epoxy composites

The 1-3 composites were fabricated from PKI 502 PZT powder and Araldite LY5138-2/HY5138 epoxy using the dice-and-fill technique [6]. The PZT powders were shaped into 10.5-mm diameter disks with thicknesses of 1.5 mm. The disks were sintered at 1285 °C. The disks were diced to make a set of grooves and filled with the epoxy. The second set of grooves was cut normal to the first and also filled with the epoxy. The samples were left at 50-60 °C for about half an hour, allowing the epoxy to dry. They were poled under an electric field of 10 MV/m at room temperature for 15 minutes [7]. In order to check if the rods were sufficiently far from each other, a SEM investigation was performed a n d a n e l e c t r o n m i c r o g r a p h s i s s h o w n i n F i g u r e 1.

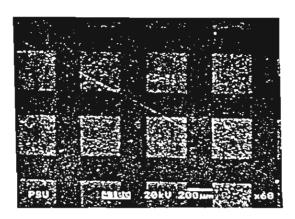


Figure 1 SEM micrograph for 1-3 PZT/epoxy composite with 0.4 volume fraction of ceramic.

Piezoelectric measurements

The piezoelectric strain was measured using a single-beam Michelson interferometer, consisting of a laser, a beam splitter and two mirrors. The incoming laser beam is divided into two beams: a probe and a reference beam. The reference beam was reflected from a reference mirror mounted on a piezoelectric transducer, which was driven by a feedback signal derived from an optical detector. The probe beam was reflected from the sample whose surface was made reflective by a metallized coating. A voltage applied across the sample produced a change in thickness. For a small displacement of the sample surface, the piezoelectric coefficient is the ratio of the strain to the field. By using this interferometric technique, the piezoelectric coefficient becomes [8]

$$d_{ii} = \frac{d_{aC}}{V} \tag{4}$$

where V is the rms voltage driving the sample, d_{ac} is the small displacement and d_a the measured piezoelectric coefficient. It is found that the d_{33} values are 190 and 188 pm/V for the 0.4 and 0.6 volume fraction of PZT, respectively (Figure 2). These values agreed closely with the modeling results [9]. It was noticed that the smaller ceramic content sample possessed a slightly higher d value. This may be related to a thermal expansion in the material, in particular, the epoxy [10].

Pyroelectric measurements

The pyroelectric measurements were performed under low pressure conditions in order to minimize the humidity in the surrounding environment. Sample temperature was varied using a Peltier element (DL12-6), sandwiched between 2 aluminum plates whose dimensions were the same as those of the Peltier element surfaces. Heating and cooling processes were achieved by varying the magnitude and direction of the current passing through the Peltier elements. The temperature was measured with a calibrated Pt-100 resistance thermometer and converted into an equivalent voltage by means of a linearised RTD module. In order to ensure uniform temperature distributions in the sample, it was necessary to vary the temperature slowly. Typical temeprature variations in the system are shown in Figure 3.

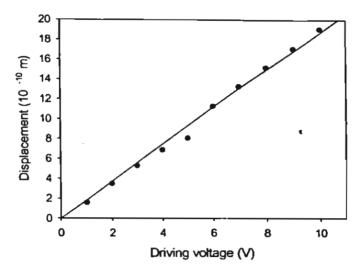


Figure 2 Piezoelectric responses in the 1-3 PZT/epoxy with 0.6 volume fraction of ceramic.

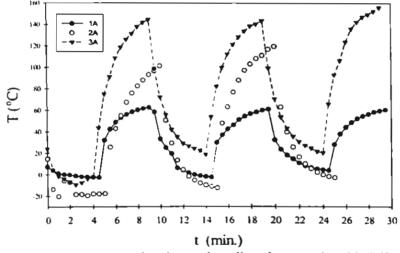


Figure 3 Typical temperature pattern upon heating and cooling the sample with different magnitudes of the current applied to the Peltier elements.

In order to check the performance of the system, measurements were also made on a piece of commercial LiNbO₃ wafer. The charge variation upon heating and cooling was measured by an electrometer. The polarization vs temperature plot was obtained and the slope of the plot is the pyroelectric coefficient. It was found that the pyroelectric coefficient was 115 μ C/m²/°C. This value was slightly higher than that reported in the literature [11]. This was probably due to a better quality of material used in this work. The values of the pyroelectric coefficient of the composites with different ceramic volume fractions are summarized in Table 1.

Table 1 Pyroelectric coefficients for the composites with different volume fractions of ceramic.

Volume fraction of PZT	p (μC/m²/°C)		
0.4	44		
0.6	54		
1	74		

Thermal diffusivity measurements

A technique developed by Lang [12] for measurements of the thermal diffusivity of samples was used in this work. Using this technique, a sample was thermally glued onto the LiTaO₃ which acted as a pyroelectric detector. The surface of the sample was exposed to a laser beam whose intensity was modulated sinusoidally at different frequencies. The thermal wave generated by the laser beam propagated through the sample to the LiTaO₃, which produced a pyroelectric current. The pyroelectric current whose frequency was the same as that of the laser beam modulator depended on the temperature variation in the LiTaO₃ and was out of phase with the laser beam. The phase shift depends only on the thermal properties of the sample and its thickness, so the thermal diffusivity was calculated.

As a check of the operation of the system, a piece of a 0.5 mm thick LiTaO₃ was used as the test sample. Its thermal diffusivity was analysed to be 1.7 x 10⁻⁶ m²s⁻¹. The result was in reasonable agreement with the reported value [13]. This technique is currently being applied to the measurement of the composite samples.

Conclusion

The interferometric measurement has been extended to the determination of the piezoelectric coefficient for 1-3 PZT/epoxy composites. The piezoelectric coefficients of 190 and 188 pm/V were obtained for the 0.4 and 0.6 volume fraction of PZT, respectively. In addition, the pyroelectric coefficients for the mentioned composites were respectively 44 and 74 µC/m²/°C. Measurements of the thermal diffusivity of the composites with different volume fractions of ceramic will be made to thermally characterize the materials.

Acknowledgement

The authors would like to thank APMRC, Hong Kong Polytechnique University for providing us an opportunity of using the facilities and valuable suggestions for sample fabrication. Thanks are due to Prof. Sidney B. Lang for helping with an analysis of the thermal data. This work is financially supported by the Thailand Research Fund (TRF).

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Dear Colleague,

It has been customary to publish a selection of papers presented at each ISE meeting in an issue of the *IEEE Transactions on Dielectrics and Electrical Insulation*. This year the journal editor, Dr. R. Hackam, has again invited us to do so. I would like to ask you to prepare an extended version of your paper, "Piezoelectricity of 1-3 PZT/epoxy composites", by Y. Phermopornsakul, S. Muensit and I.L. Guy, for this issue.

The manuscript should be submitted to me before 15 January 2003. The manuscripts will be sent out to referees as customary with this journal. The accepted manuscripts will then be published in a special issue, probably in December 2003. The papers must be extended versions of those published in the Proceedings of ISE11, preferably with additional new material. The material should not have been published elsewhere. Manuscripts that are essentially the same as those published in the Proceedings of ISE11 are unacceptable. Instructions for the preparation of the manuscript are available on the Internet at http://tdei.sju.edu/tdei/formats.html. Please send your manuscript directly to me rather than to Dr. Hackam as described in the instructions.

Please send a confirmation of your intention to submit a manuscript to Sidney Lang by e-mail at lang@bgumail.bgu.ac.il.

Thank you for your cooperation.

R. J. Fleming

DETERMINATION OF THE PIEZOELECTRIC AND PYROELECTRIC COEFFICIENTS AND THE THERMAL DIFFUSIVITY OF 1-3 PZT/EPOXY COMPOSITES

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ABSTRACT

PZT/epoxy composites with 1-3 connectivity were prepared using the dice-and-fill technique. The samples were poled with an electric field of 10 MV/m for 30 minutes at room temperature. The piezoelectric and pyroelectric coefficients for the composites were measured. From the laser interferometric measurements, it was found that the piezoelectric d_{33} coefficients for the composites were independent of the volume fraction and averaged (190 \pm 10) pm/V, which was about half of the measured value of lead zirconate titanate (PZT) ceramic. Measurements of the pyroelectric coefficient showed that the coefficients increased with the ceramic content and reached values as large as 54 μ C/m² °C. The thermal diffusivity of the composites was also determined using a technique based on the measurement of the phase retardation of a thermal wave passing through the material. The average value for the composites was $(2.15 \pm 0.05) \times 10^{-7}$ m²/s.

1. INTRODUCTION

There have been a number of investigations of the piezoelectric and pyroelectric properties of ceramic/polymer composites with different connectivities and volume fractions of the ceramic phase. For the 1-3 type PZT/epoxy, both the piezoelectric and pyroelectric activities depend on the polarization of the ceramic phase. Inhomogeneous displacement profiles under an electric field for a single rod of ceramic embedded in an epoxy matrix have been analysed and the effective piezoelectric coefficients derived [1]. The most commonly used piezoelectric coefficient d is defined by

$$d = \left(\frac{\partial S}{\partial E}\right) \tag{1}$$

where S is the strain and E the electric field.

A pyroelectric material exhibits a spontaneous polarization in the absence of an electric field [2]. A variety of new pyroelectric materials can be made by combining a ferroelectric ceramic with a passive polymer plase. A ceramic/polymer composite of the 1-3 type is an important structure, providing a new range of material properties. Moreover, by selection of the ceramic, polymer, proportions, and spatial scales, these material properties can be tailored to specific device requirements such as infrared sensors and thermal imaging devices [3-5].

The pyroelectric coefficient p is defined by [6]

$$p = \frac{(\Delta Q / A)}{\Delta T} \tag{2}$$

where Q is the charge, A is the electrode area perpendicular to the polar axis and T is the temperature. An alternative form of (2) is:

$$p = \frac{I}{AdT/dt} \tag{3}$$

Equations (2) and (3) indicate that the coefficient p can be measured either by measuring the current generated for a known rate of change of temperature, or by measuring the charge generated for a known temperature change. In either case, the change in electrode area resulting from thermal expansion needs to be kept in mind. This effect could be especially significant in polymers where the thermal expansion coefficient may be relatively large.

2. SAMPLE PREPARATION

The ceramic used for the composite was lead zirconate titanate (PZT), supplied by Ultra-sonic Powders Ltd., type PKI 502. The epoxy used was Araldite type LY5138-2/HY5138. The composites were fabricated using the dice-and-fill technique [7] The PZT powders were shaped into 10.5-mm diameter disks with thicknesses of 1.5 mm. The disks were sintered at 1285 °C and diced with a diamond

saw to create a set of the grooves which were then filled with the epoxy. Each diced ceramic disk was placed in a glass petri dish in a low pressure chamber and a solution of the epoxy was poured over the disk. The samples were left for 15 minutes to allow the epoxy to fill the grooves and bubbles in the grooves to dissipate. The samples were left at 50°C to 60°C for half an hour, allowing the epoxy to cure. The second set of grooves was cut normal to the first and also filled with the epoxy. Ceramic content in the epoxy was varied as shown in Table 1. Final finishing of the composites to the desired thickness was done by polishing using abrasive papers of various grades.

In order to check the separation of the ceramic rods, the composites were investigated by a JEOL JSM-5200 scanning electron microscope (SEM) and the electron micrographs are shown in Figure 1. In order to enhance the piezoelectric and pyroelectric activities, each sample was electroded on the top and bottom and poled with an electric field of 10 MV/m at room temperature for 15 minutes.

3. PIEZOELECTRIC MEASUREMENT

The piezoelectric d_{33} coefficient was measured using a single-beam Michelson interferometer, based on the design developed by the PennState group [8]. In the system the incoming laser beam from a Uniphase 1135P He-Ne Laser was divided into two beams: a probe and a reference beam. The reference beam was reflected from a reference mirror mounted on a piezoelectric transducer. The probe beam was reflected from the sample whose surface was made reflective by a metallized coating. The two beams were combined to produce an interference pattern, which was directed to a pin photodiode detector. A sinusoidal driving voltage was applied to the sample to produce a change in sample thickness, which in turn resulted in a change in the light intensity of the interference pattern at the detector. The component of the detector voltage related to the displacement of the sample surface was measured by a SR530 Lock-in Amplifier (Stanford Research Systems, Sunnyvale, CA, USA). For a small displacement d_{ac} of the sample surface, the piezoelectric coefficient d_{ii} is given by [8]

$$d_{\bar{s}} = \frac{d_{ac}}{V} \tag{4}$$

where V is the driving voltage.

For comparison purposes, the piezoelectric responses of the PZT and composites are shown in Figures 2 and 3, respectively. All of the responses showed a linear relation between the displacement and driving voltage. The measured d_{33} coefficient of the PZT in a frequency range of 1- 5 kHz was (360 \pm 10) pm/V. Measured values for the composites having 0.4 and 0.6 volume fractions of ceramic were 190 and 188 pm/V, respectively. These values were measured in the frequency range 1 - 3 kHz in order to avoid problems arising from heat generation in the composites at higher frequencies. The d_{33} values for the composites agreed closely with the modeling results [9], but were only about 50% of the value for PZT. This difference may be due to incomplete poling of the PZT or to heat accumulation in the epoxy.

The dielectric constants of samples with various ceramic volume fractions were measured using an HP 4263B LCR meter and an HP 16451B Dielectric Test Fixture. These values increased linearly with increasing ceramic content. The piezoelectric g_{33} coefficient of the samples was calculated using a relation: $g_{33} = d_{33} / \varepsilon_0 \varepsilon_3$, where ε_0 was the permittivity of free space and ε_3 the dielectric constant [3]. It was found that the g_{33} value for the 40% PZT/epoxy (43x10⁻³ m²/C) was twice that of PZT (22x10⁻³ m²/C).

4. PYROELECTRIC MEASUREMENT

The pyroelectric measurements were performed at a reduced pressure of 10⁻³ Pa in order to minimize the humidity in the surrounding environment. Sample temperature was varied using a Peltier element, sandwiched between 2 aluminum plates. Heating and cooling were achieved by varying the magnitude and direction of the current passing through the Peltier elements. The temperature was measured with a Pt-100 resistance thermometer and converted into an equivalent voltage by means of a linearised RTD module. The measurement system is schematically shown in Figure 4. In order to ensure uniform temperature distributions in the sample, it was necessary to vary the temperature slowly. Typical temperature variations in the system are shown in Figure 5.

In order to check the performance of the system, measurements were made on a piece of commercial LiNbO₃ wafer. The charge variation upon heating and cooling was measured by an electrometer. The slope of the polarization vs temperature plot

gave a pyroelectric coeffficient. of 115 μ C/m² °C, slightly higher than that reported in the literature [10]. The difference could be due to the better quality of material us ed in this work. The measured pyroelectric coefficients for the composites having 0.4 and 0.6 volume fraction of ceramic were respectively 44 and 54 μ C/m² °C, and 74 μ C/m² °C for PZT.

5. THERMAL DIFFUSIVITY MEASUREMENT

A method for measuring thermal diffusivity has been developed by Muensit and Lang [11]. The sample was attached by a very thin layer of thermally conductive glue to a pyroelectric detector, in this case, LiTaO₃. The detector was mounted on a brass substrate. A Lisa HL25 diode laser was used and the laser beam was modulated at a number of different frequencies and allowed to impinge on a blackened area on the top surface of the sample. The computer package, *Mathematica* [12], was used to find an analytic solution to the one-dimensional heat conduction equation with appropriate boundary conditions. An illustration of the calculated attenuation and phase retardation of the thermal waves for a sample having the thermal properties of the 1-3 composite with 0.4 volume fraction of ceramic is shown in Figure 6. The unknown thermal diffusivity of the sample was found by fitting the experimental frequency vs phase lag data to the theory by a nonlinear regression algorithm.

In the experiments, the lag between the phase of the modulated laser beam and that of the pyroelectric current from the LiTaO₃ was measured for 10-15 different modulation frequencies in the range of 1 - 10 Hz. In the calculations, it was necessary to know the physical properties such as the dimensions, density and heat capacity of the samples. The heat capacity of the test samples was measured using a Differential Scanning Calorimeter (Perkin Elmer, DSC7). Experimental phase lag and pyroelectric current data obtained from the composite are shown in Figure 7. Values of the thermal diffusivity for the composites having 0.4 and 0.6 volume fraction of ceramic were found to be 2.2 x 10⁻⁷ and 2.1 x 10⁻⁷ m²/s, respectively.

6. DISCUSSION AND CONCLUSION

All the experimental data obtained are listed in Table 2. In this work, the interferometric measurement was extended to the determination of the piezoelectric coefficient for the 1-3 PZT/epoxy composites. The Byer and Roundy technique [10]

for measurement of the pyroelectric coefficient was used for the composites and a new method [11] was used to determine thermal diffusivity. Because the epoxy was not ferroelectric, the piezoelectric and pyroelectric responses were considerably different from those of the poled PZT phase.

The densities and heat capacities of the 1-3 composites correspond to volumetric averages, as would be expected. Similarly, the thermal diffusivity can be modeled as polymer and ceramic elements in parallel. This also leads to a volumetric average for the composite. The situation for the dielectric constant, piezoelectric and pyroelectric coefficients is more complicated. As discussed by Chan and Unsworth [9], the d_{33} value of the composite will be higher than that predicted from a volumetric average because the elastic compliance of the polymer is higher than that of the ceramic. Experimental results in this work agreed qualitatively with this prediction. The d_{31} piezoelectric coefficient (not measured in the present work) would have a smaller absolute value than predicted from a volumetric average. This would increase the pyroelectric coefficient because of a larger contribution due to piezoelectricity (secondary pyroelectric effect). This again agreed qualitatively with the experimental measurements. For the calculated g_{33} coefficient, it is noted that this value increased considerably with decreasing volume fraction of PZT. This was due to the large decrease in the permittivity.

Qualitative differences between measurement and theoretical models may be due to incomplete poling of the composites or heat accumulation in the epoxy. It was also observed that the composites were mechanically flexible. The relatively high piezoelectric and pyroelectric coefficients combined with the flexibility suggest that the materials show good promise for applications as piezoelectric transducers and pyroelectric detectors. However, it is important that the structural designs of the transducers or detectors using the composites should take into account the dissipation of the heat generated by either an AC field or sharp temperature changes.

ACKNOWLEDGMENT

The authors would like to thank Profs. H. L. W. Chan and K. W. Kwok, Hong Kong Polytechnique University for use of their sample fabrication facilities, and valuable instruction. Thanks are due to Prof. Sidney B. Lang for useful suggestions on the thermal diffusivity analysis. This work is financially supported by the Thailand Research Fund (TRF).

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TABLE CONTENT

Table 1 Ceramic and polymer parameters used in the composite fabrication.

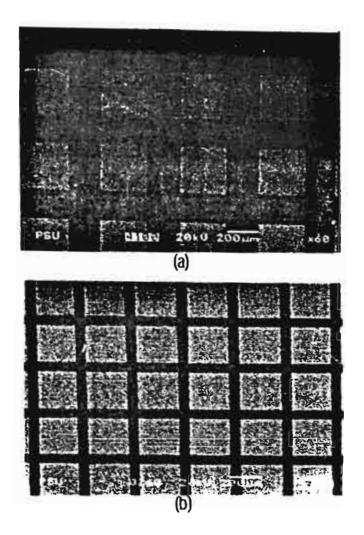
Table 2 Results for the 1-3 PZT/epoxy composites.

Volume fraction	Ceramic width	Epoxy width	
of ceramic	(mm)	(mm)	
0.4	0.325	0.21	
0.6	0.325	0.08	
1	-	-	

osites	Density 10 ³ (kg/m ³)	Dielectric	Heat	d ₃₃	g 33	P	α
		constant	capacity	(10 ⁻¹² m/V)	$(10^{-3} \text{m}^2/\text{C})$	(µC/m² °C)	$(10^{-7} \text{ m}^2/\text{s})$
		(1 kHz)	(J/kg °C)				
роху)	1.1	2	87	-	, -	-	0.9
	3.7	500	214	190	43	44	2.2
	5.0	800	282	188	27	54	2.1
ZT)	7.7	1900	400	360	22	74	4.0

FIGURE CAPTIONS

- 1.SEM micrograph (x60) for 1-3 PZT/epoxy composite with ceramic volume fraction (a) 0.4, (b) 0.6.
- 2. Piezoelectric responses for PZT (PKI 502) observed at a driving signal of 1 kHz.
- 3..Piezoelectric responses at 1 kHz for 1-3 PZT/epoxy with ceramic volume fraction (a) 0.4, (b) 0.6.
- 4. Schematic diagram of the pyroelectric measurement system.
- Typical temperature pattern upon heating and cooling the composites with different currents applied to the Peltier elements.
- 6. Simulation showing the attenuation and phase lag of the thermal waves for the composite with ceramic volume fraction 0.4.
- 7. Variations of (a) phase lag and (b) amplitude of the pyroelectric current measured on the composite with ceramic volume fraction 0.4.



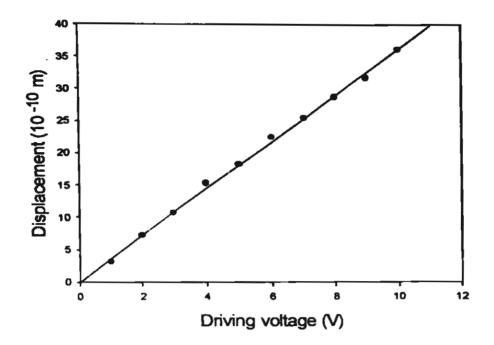


Figure 2

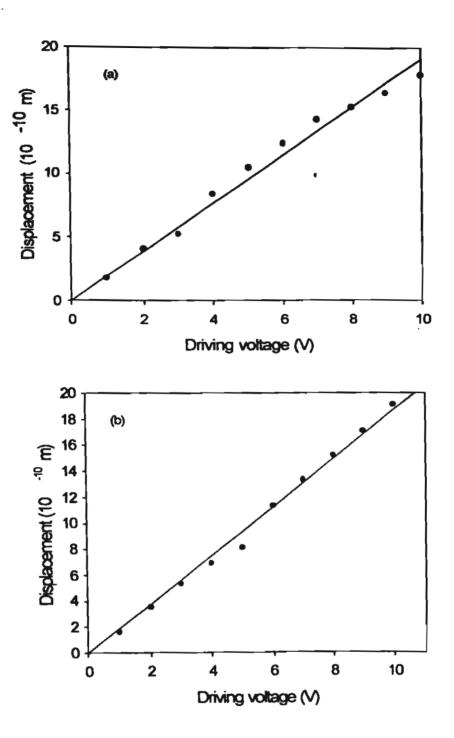


Figure 3 (a) (b)

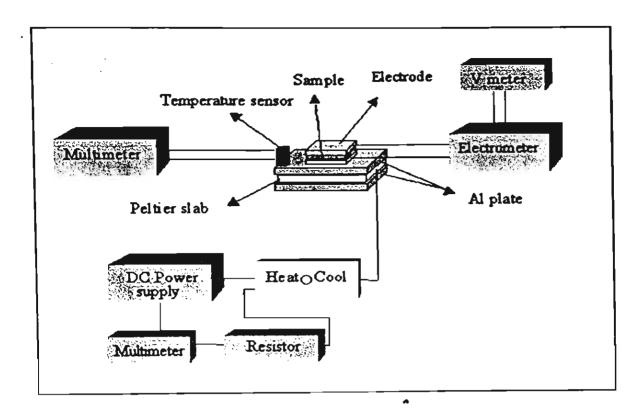


Figure 4.

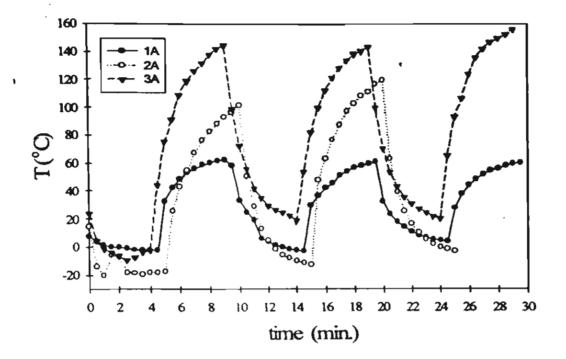


Figure 5

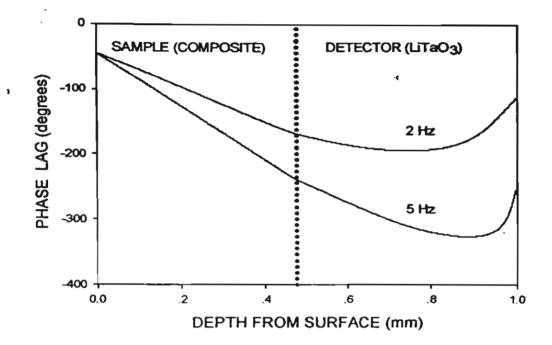


Figure 6

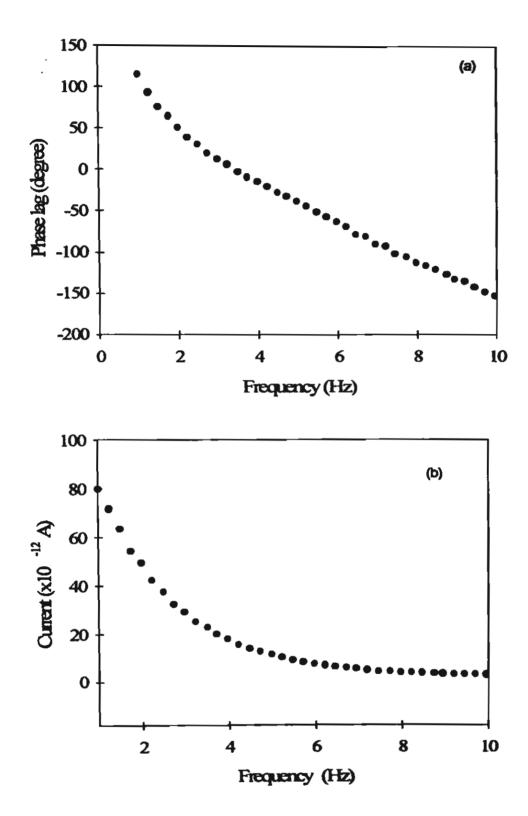


Figure 7 (a), (b):

Electrothermal Effects of PZT/P(VDF-TrFE) Composite with 0-3 connectivity

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ABSTRACT Owing to a growing interest in using the composites in the pyroelectric sensor applications, composites of lead zirconate titanate (PZT) powder dispersed in a polyvinylidene fluoride/trifluoroethylene (PVDF/TrFE) copolymer matrix with 0-3 connectivity and 0.3 ceramic volume fraction were fabricated and characterized the electrothermal properties. The first use of pyroelectric technique for determining the thermal material parameters of the composite was described. From the measurements, the thermal diffusivity of 2.0 x 10⁻⁸ m²s⁻¹ was found for the composite.

KEYWORDS: lead zirconate titanate, polyvinylidene fluoride/trifluoroethylene, composite, electrothermal.

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