Porous PZT Ceramics for Receiving Transducers

Hudai Kara, Rajamani Ramesh, Ron Stevens, and Chris R. Bowen

Abstract—PZT-air (porous PZT) and PZT-polymer (polymer impregnated porous PZT) piezocomposites with varying porosity/polymer volume fractions have been manufactured. The composites were characterized in terms of hydrostatic charge (d_h) and voltage (g_h) coefficients, permittivity, hydrostatic figure of merit $(d_h.g_h)$, and absolute sensitivity (M). With decreasing PZT ceramic volume, g_h increased, and $d_h.g_h$ had a broad maximum around 80 to 90% porosity/polymer content. The absolute sensitivity was also increased. In each case, PZT-air piezocomposites performed better than PZT-polymer piezocomposites. Hydrophones constructed from piezocomposites showed slightly lower measured receiving sensitivities than calculated values for piezocomposite materials, which was due to the loading effect of the cable and the low permittivity associated with the piezocomposites.

I. Introduction

PIEZOCOMPOSITES with various connectivity patterns [1] such as (0-3), (1-3), and (3-3) have been produced for sensor, actuator, and transducer applications [2]. Of these structures, the 3-3 piezocomposites have been of particular interest for low frequency (0–100 kHz) hydrophone applications. 3-3 piezocomposites, in which the ceramic and polymer phase are interconnected in three directions, are also of interest as materials for acoustic transducers, medical imaging, sonar, and non-destructive evaluation. High hydrostatic sensitivity, low density and stiffness, improved acoustic matching with water or human tissue, high compliance for damping, and mechanical flexibility for machining are some of the potential benefits of 3-3 composites [3].

For hydrophone applications, the important parameters are the hydrostatic charge (d_h) and voltage (g_h) coefficients, the hydrostatic figure of merit $(d_h.g_h)$, and the absolute sensitivity (M). The hydrostatic charge coefficient, which defines the actuating capability of the composite, is related to the piezoelectric charge coefficients, both in the polarization (d_{33}) and transverse directions $(d_{31}$ and $d_{32})$:

$$d_h = d_{33} + (d_{31} + d_{32}) \quad (\text{CN}^{-1} \text{ or } \text{mV}^{-1}). \quad (1)$$

The hydrostatic voltage coefficient, g_h, which defines the

sensitivity of the hydrophone, is related to the value of d_h and the permittivity under constant stress (ε_{33}^T):

$$g_h = d_h/\varepsilon_{33}^T \quad (\text{VM}^{-1} \text{ Pa}^{-1}). \quad (2)$$

The figure of merit (FoM) is the product of (1) and (2) and is termed the "hydrostatic figure of merit," which defines the composite's suitability for underwater sonar applications:

$$\text{FoM} = d_h{}^* g_h \quad (\text{Pa}^{-1}). \quad (3)$$

The receiving sensitivity of a device operating under hydrostatic conditions is related to g_h and the thickness (t) of the piezocomposite element [4]:

$$M = g_h{}^* t \quad (\text{V Pa}^{-1}) \quad (4a)$$

$$M = -20 \log (g_h{}^* t) \quad (\text{dB re 1V } \mu\text{Pa}^{-1}). \quad (4b)$$

3-3 composites are usually produced by manufacturing a porous ceramic structure and subsequently impregnating the porosity with a polymer. The porosity can also be left as air [5]. There are a number of techniques to obtain porous structures: newly developed rapid prototyping [6] and free-form fabrication [7] processes. Using these processes, a desired porous structure is built up layer by layer, mixing ceramic powder with a fugitive binder [3] (BURPS, an acronym for "burned-out plastic spheres") or replicating a porous structure by coating it with a ceramic slurry and burning it out during sintering (lost wax replication of a coral skeleton [8] and polymeric sponge [9] techniques). Of these, the BURPS and polymeric sponge techniques are relatively easy to implement for large-scale production. The polymeric sponge technique is being used successfully to commercially produce high-temperature ceramic filters [10]. The processing technique involves impregnation of polyethylene foams with a ceramic slip and controlled sintering with an intermediate temperature hold for removal of the foam.

The BURPS technique produces a porous structure by mixing ceramic powders with polymer spheres followed by compaction and controlled sintering. A 3-3 BURPS piezocomposite showed that the hydrostatic figure of merit increases with increasing polymer fraction [3]. However, the polymer fraction only varied between 30 and 70% volume because it is not feasible to produce higher pore content ceramics using the BURPS technique. Creedon *et al.* [9] produced PZT/polymer composites with 85–90 vol. % porosity by a polymeric sponge technique; they also found an

increase in hydrostatic sensitivity with increasing polymer fraction. We have produced a range of porous ceramics of PZT-5H matrix ranging from 9 to 92 vol. % porosity using both the BURPS and polymeric sponge techniques. The samples were characterized in terms of d_{33}, d_{31}, ε, d_h, g_h, FoM, and M before and after polymer impregnation. The effect of pore/polymer fraction on the properties is discussed, and the results are compared with corresponding data obtained from hydrophones manufactured for piezocomposite materials.

II. EXPERIMENTAL PROCEDURE

A. Sample Preparation

Commercially available PZT-5H spray-dried powder (Morgan Electroceramics, Vauxhall Industrial Estate, Wrexham, UK) was compacted and sintered at 1200°C for 2 h to form monolithic PZT ceramics of 4 mm thickness and 40 mm diameter. BURPS and polymeric sponge techniques were used to obtain porous samples with the same dimensions. For the BURPS technique, the PZT powder was mixed with acrylic thermoplastic resin (ATR) and polyethylene oxide (PEO) at various weight ratios to produce a range of pore volume fractions. The mixed powder was then uniaxially compacted at 50 MPa. The compacts were then subjected to a burnout cycle, heating with a ramp rate of 1°C/min (to 600°C for 1 h) to remove the organic phase before final sintering at 1200°C for 2 h. For the polymer sponge technique, a ceramic slip of 80 wt. % PZT, 3 wt. % PVA, 0.75 wt. % dispersing agent, and distilled water was prepared by ball milling for at least 6 h. A number of polyethylene sponges of different cell sizes were cut and impregnated with the PZT slip. The excess ceramic slip was removed by compressed air. The green ceramic foams were then introduced to the same burnout cycle described previously before final sintering at 1200°C for 2 h.

Thin aluminium stickers (150 μm) were used as electrodes for the porous samples (PZT-air composites), and silver paste was used for the dense PZT and PZT-polymer composites. Before polymer impregnation, all samples were individually poled at 110°C and 12.5 kV for 10 min using corona poling. The corona tip height was set to 30 mm. The piezoelectric charge coefficients (d_{33} and d_{31}) and permittivity were measured by a Berlincourt d_{33} Piezo Meter (Take Control, University of Birmingham Research Park, Vincent Drive, Birmingham, UK) and LCR meter (HP 4263B, Hewlett Packard Japan Ltd., Hyogo, Japan) respectively. The hydrostatic charge (d_h) and voltage (g_h) coefficients, hydrostatic figure of merit (FoM), and absolute receiving sensitivity (M) were calculated using (1)–(4).

B. Hydrophone Construction and Testing

To compare the calculated absolute receiving sensitivities of piezocomposites with that of prototype hydrophones assembled from manufactured piezocomposites, hydrophones were constructed from a solid PZT ceramic, 22%-dense PZT-air composite, and 11%-dense PZT-polymer composite. Because of low permittivity values, piezocomposite capacitance values were lower than those of the dense PZT ceramic as shown in Table I. To avoid the loading effect caused by the cables, which has a capacitance of 63 pF for a 1.5-m cable, the device capacitance was increased by attaching two discs of piezocomposites back-to-back using an electrically parallel connection. The configuration is shown in Fig. 1(a). The piezocomposite pieces were attached to a 150-μm thick copper plate electrode with conductive epoxy in opposite poling directions. The remaining two faces were then attached with a single copper plate and soldered to a cable. The center electrode was used as the active connection, and the two outer electrodes were used as screens. Finally, the whole assembly was molded in polyurethane rubber for water isolation as shown in Fig. 1(b). The solid PZT ceramic hydrophone was assembled in the same way for a direct comparison of the relative performance.

The assembled hydrophones were tested in a 1.5- × 1.5- × 2.8-m³ water tank using an impulse technique [11] in the frequency range from 20 to 140 kHz. A truncated parametric array was used as the acoustic source, which enabled measurements to be made over a wide frequency range without interference from surface and wall reflections. The hydrophone sensitivities were measured by comparing the hydrophone outputs with those of a standard Brüel and Kjær 8103 hydrophone that had been calibrated at the National Physical Laboratory, Teddington, UK. The output of both hydrophones were fed in to a high input impedance amplifier. The signals were recorded by a Lecroy digital oscilloscope.

III. RESULTS AND DISCUSSIONS

We first characterize the microstructures of the various PZT-air/polymer composites to verify the connectivity pattern of the composites. Following this, the change in permittivity, hydrostatic charge (d_h) and voltage (g_h) coefficients, hydrostatic figure of merit (FoM), and absolute sensitivity (M) with varying pore/polymer content will be reported; finally, the results from the piezocomposites will be compared. The results will be discussed with respect to the receiving sensitivity measured from the testing of constructed hydrophones.

A. Microstructures

Microstructures of the various PZT-air and PZT-polymer composites are shown in Fig. 2 and 3, respectively. Depending on the PZT ceramic volume, there are two distinct connectivity patterns observable in the composites. The composites with high PZT volume content (>60%) showed 3-0 connectivity (i.e., discrete polymer or pore phases within PZT matrix), while 3-3 connectivity was achieved for the composites containing low PZT vol-

TABLE I
PROPERTIES OF PZT CERAMIC, PZT-AIR AND PZT-POLYMER COMPOSITES.

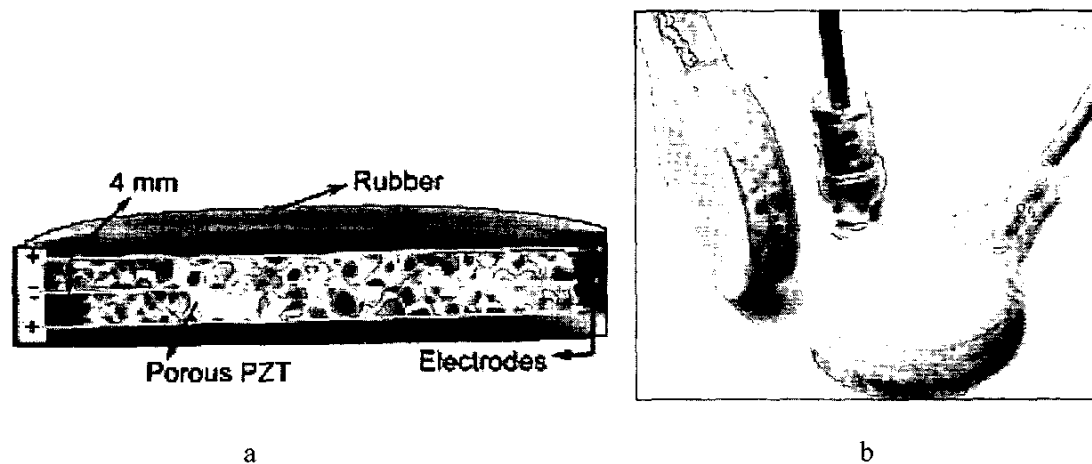|	d_{33} $\left(\mathrm{pCN^{-1}}\right)$	d_{31} $\left(\mathrm{pCN^{-1}}\right)$	$\varepsilon_{\mathrm{rel.}}$	d_h $\left(\mathrm{pCN^{-1}}\right)$	$g_h \times 10^{-3}$ $\left(\mathrm{Vm^{-1}Pa^{-1}}\right)$	$d_h \cdot g_h \times 10^{-15}$ $\left(\mathrm{Pa^{-1}}\right)$	M $\left(\mathrm{dB\ re\ 1\ V\mu Pa^{-1}}\right)$	Device cap. (pF)
PZT-5H	513	220	2859	72.8	2.87	209	-218	5050
PZT-air	210	29	316	152	54.2	8238	-193	800
PZT-polymer	35.7	7	76	21.7	28.3	614	-199	210

a

b

Fig. 1. a) Cross-section view of a piezocomposite hydrophone; b) assembled prototype hydrophones.
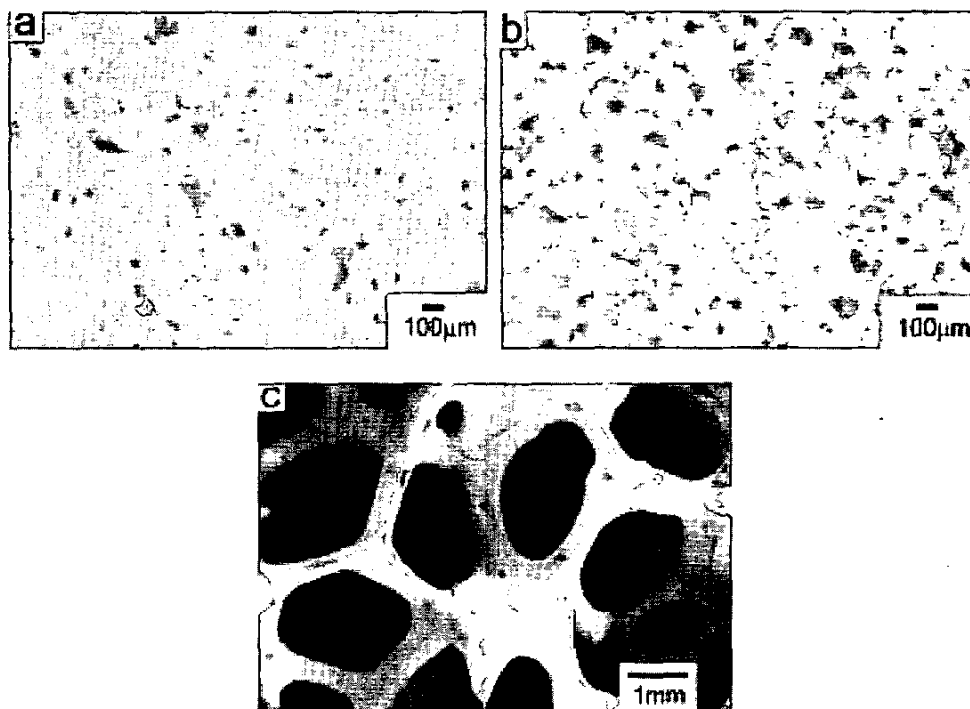

Fig. 2. SEM micrographs showing PZT-air composites with varying PZT volume fractions: a) 80%, b) 44%, and c) 17%. Note the change in connectivity from 3-0 (a) to complete 3-3 (c). BURPS samples (PEO) had irregular pore morphology (a and b) and reticulated PZT 3-3 composite (c) as fabricated.
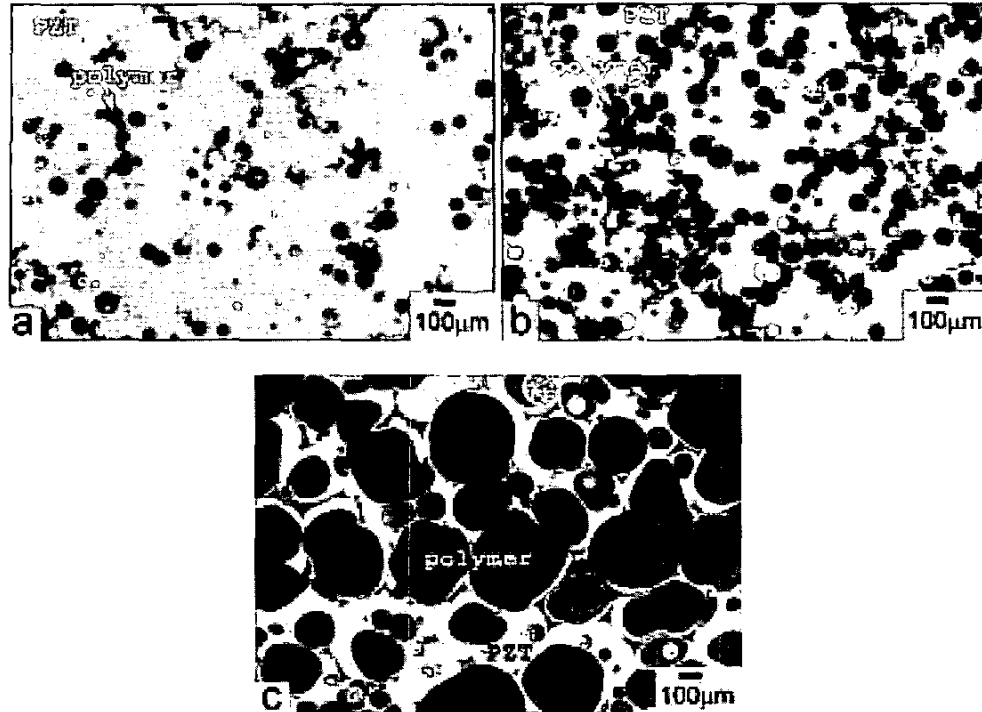
Fig. 3. SEM micrographs showing PZT-polymer composites with varying densities: a) 85%, b) 59%, and c) 21% PZT. Spherical pore morphology in BURPS samples (ATR) (a and b) is due to rounded morphology of ATR. (Polymer is the dark phase).

ume (<40%). The composites falling between these limits (40–60 vol. % PZT) showed a mixture of 3-0 and 3-3 connectivity.

The composites produced via the polymeric sponge technique showed perfect 3-3 connectivity (i.e., both the polymer and PZT were fully interconnected). The composites produced using the BURPS technique showed different pore morphology because of the different morphologies of the polymer phases used to create the pores. The spherical ATR resulted in rounded pores (Fig. 2), whereas irregular PEO resulted in more irregular pore morphology (Fig. 3).

B. Permittivity of Piezocomposites

As the low permittivity polymer replaces the high dielectric PZT ceramic, the overall permittivity decreases with decreasing ceramic content (Fig. 4). There is no significant difference in properties between PZT-air and PZT-polymer composites, which is due to the comparable permittivity of air and polymer phases. It is also interesting to note that at ~60% PZT ceramic volume, where the 3-0 connectivity starts, the permittivity deviates into two separate lines. This result is due to the extensive cracks lying parallel to the surface as revealed by cross-sectional SEM examinations in Fig. 5(a and b). These cracks, which occur after green compaction caused by relaxation of the elastic ATR spheres, prevent connectivity of the ceramic between the electroded surfaces, considerably reducing the permittivity. However, it should also be noted that pore anisotropy can also be a reason for this deviation between
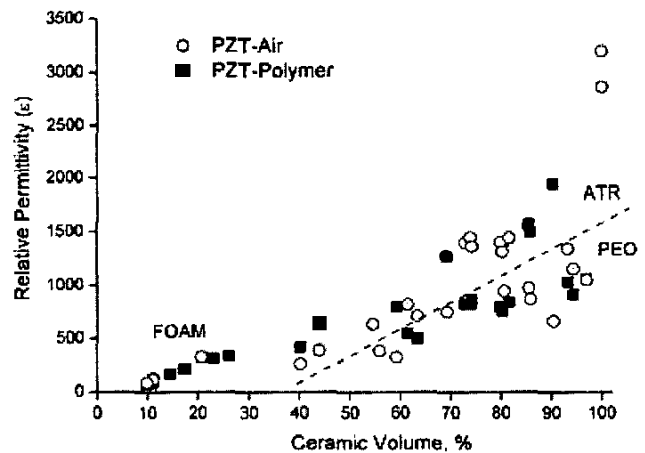

Fig. 4. Variation in permittivity with PZT ceramic volume fraction. Permittivity was reduced with decreasing ceramic volume fraction. Polymer infiltration (PZT-polymer) had no pronounced effect on permittivity when compared with porous PZT (PZT-air) composites. The dashed line separates two different BURPS samples (ATR and PEO).

ATR and PEO samples because higher permittivity is obtained when pores align along the thickness direction compared with those normal to the thickness direction [12]. All of the reticulated foam-based composites with low ceramic volume (3-3) followed a similar reduction in permittivity with decreasing ceramic volume fraction, although cracks are apparent within the struts [Fig. 5(c)] and at the polymer/ceramic interface [Fig. 5(d)].
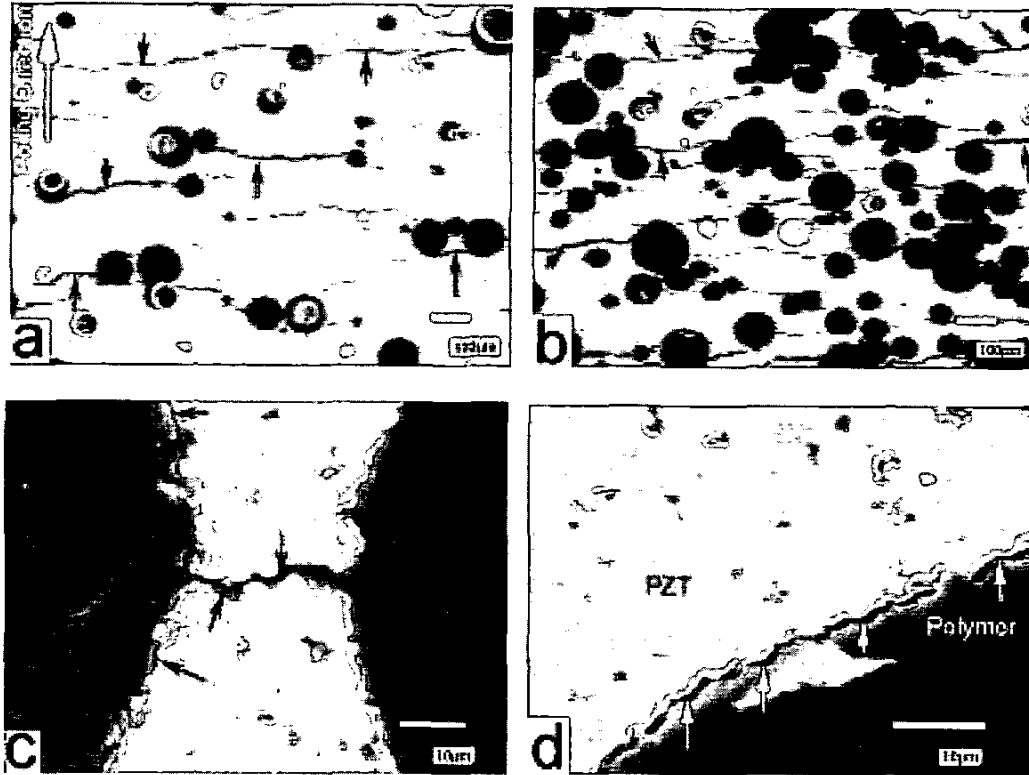
Fig. 5. Cross sections of a) 85%, b) 59% PZT (BURPS, ATR), and c) 12% PZT (foam) PZTpolymer composites. The cracks parallel to the surface and normal to the poling direction in (a) and (b) reduce the permittivity and electrical connectivity of the composite. Cracking in struts (c) and at the PZT/polymer interface (d) is also apparent.

C. Piezoelectric Coefficients of Piezocomposites

1. Hydrostatic Charge Coefficient: The hydrostatic charge coefficient (d_h) increases with decreasing PZT ceramic volume, reaching a broad peak around 50 to 60 vol. % PZT for PZT-air composites, as shown in Fig. 6. This is in good agreement with analytical models that indicate a broad maximum around 40 to 50% PZT for d_h [13]. It is difficult to draw a similar conclusion from PZT-polymer composites because of the large scatter of the results. Decoupling of d_{33} and d_{31} coefficients by the polymer and ceramic struts along the direction transverse to the poling direction resulted in a large reduction in d_{31} compared with d_{33} as shown in Fig. 7, which in turn increases d_h. For PZT-5H, the d_{33}, charge per unit force produced in the direction of polarization, is almost the same in magnitude (513 pCN^{-1}) as the combination of d_{31} and the d_{32}, the charge on the transverse directions, but opposite in sign (-220 pCN^{-1}). Therefore, if a hydrostatic force is applied to monolithic PZT-5H, the charges cancel each other out according to (1). As seen in Fig. 7, an increase in the d_{33}/d_{31} ratio with decreasing ceramic volume fraction indicates strong decoupling. Air provides better decoupling than the polymer. The d_{33}/d_{31} ratio does not tail down as in d_h in Fig. 6 because of the low values of the d_{33} and d_{31} charge coefficients at low ceramic fraction.
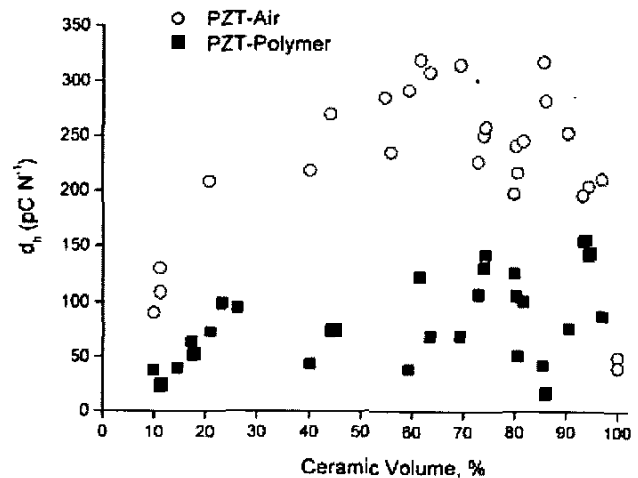

Fig. 6. Variation in d_h with PZT ceramic volume for PZT-air and PZT-polymer composites. Polymer infiltration reduced the hydrostatic response of the composite.

Polymer infiltration of the poled porous ceramics reduced the hydrostatic response of the composites considerably when compared with the PZT-air composites, although there was still improvement over dense PZT ceramic for most of the PZT-polymer composites. The re-
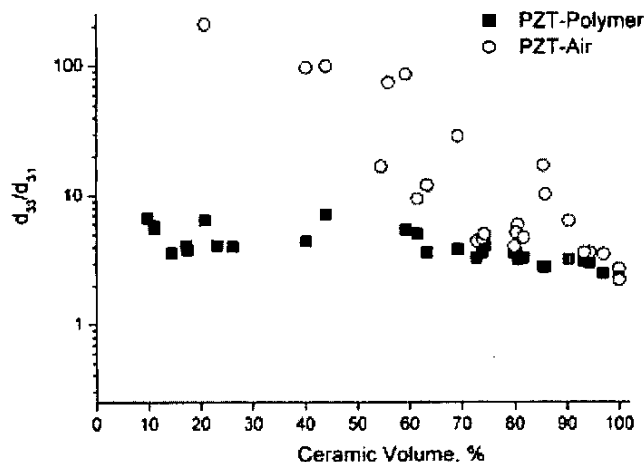
Fig. 7. Variation in d_{33} and d_{31} with PZT ceramic volume fraction for PZT-air and PZT-polymer composites. Air provides better decoupling than polymer phase.
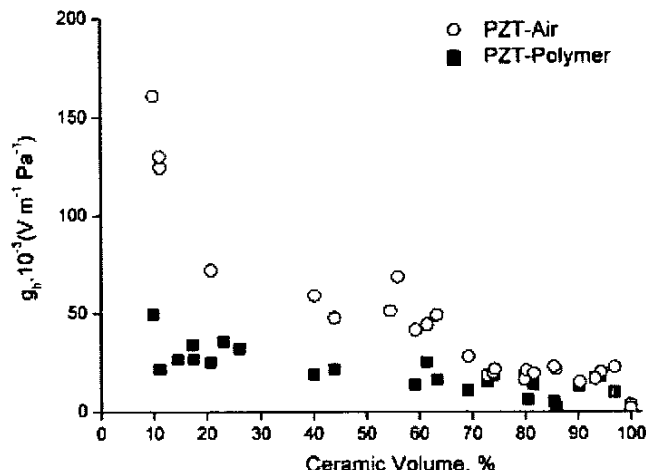

Fig. 8. Variation in hydrostatic voltage coefficient, g_h, for PZT-air and PZT-polymer composites. Hydrostatic sensitivity of the composite increases with decreasing ceramic volume fraction.

duction in d_h by polymer infiltration is due to reduction in stress born by the ceramic phase, which is not the case for PZT-air composites, where stress is born by the ceramic phase only. As explained previously, extensive cracking in BURPS (ATR) samples hinder the stress or charge transfer between the electroded faces, resulting in a dramatic reduction in d_h compared with BURPS (PEO) samples.

The hydrostatic charge coefficient was shown to be a function of the Young's modulus of the polymer [13], increasing with decreasing stiffness because of an increase in stress transfer into the PZT ceramic phase. The epoxy resin used in this work had the Young's modulus of 2.8 GPa determined by tensile testing. It has been reported that the softer epoxy yields lower acoustic impedance and higher electromechanical coupling [14]. The type of epoxy used will also influence the electromechanical properties. Weak bonding of the polymer to the ceramic might also contribute to a reduction in d_h because of reduction in stress transfer efficiency.

2. Hydrostatic Voltage Coefficient: The hydrostatic voltage coefficient, g_h, increases with decreasing PZT ceramic volume as shown in Fig. 8. The increase in g_h is more pronounced for PZT-air composites because of the higher d_h values. As low permittivity air or polymer replaces the high value dielectric PZT phase, an increase in g_h is expected according to (2) because g_h is inversely related to the permittivity (ε). In a 3-3 composite, the improved d_h and the reduction in permittivity will increase g_h. Furthermore, a decrease in the PZT ceramic volume fraction will lead to the PZT ceramic component bearing more stress, resulting in an increase in electric field per unit of hydrostatic stress. The highest hydrostatic voltage coefficients measured for the PZT-air and PZT-polymer composites are 49.4×10^{-3} and 161×10^{-3} $Vm^{-1}Pa^{-1}$, respectively. These values are considerably higher than those for dense PZT ceramic (3.7×10^{-3} $Vm^{-1}Pa^{-1}$). The Young's modulus of the polymer is also thought to influence g_h [13].

As the compliance of the polymer increases, stress transfer from the polymer to the PZT ceramic phase will also increase, thus increasing g_h.

In reality, continuing increases in g_h with ever-decreasing ceramic volume are impractical. As permittivity decreases, the capacitance of the piezocomposite will decline to a level where its use as an active element for a hydrophone becomes unacceptable. In addition, there will be a breakdown limit at which the PZT ceramic phase cannot bear the hydrostatic stress (mechanical breakdown) or the high level of stress in the active PZT ceramic phase will result in depoling.

3. Hydrophone Figure of Merit: The hydrophone figure of merit (FoM) defines the hydrophones' sensing (g_h product) and actuating capability (d_h product). The FoM is a product of d_h and g_h, expresses the optimum value of an active element for hydrophone applications, and is also a FoM for the noise level [15]. Fig. 9 shows optimum values around 20% PZT ceramic volume. Although both PZT-air and PZT-polymer composites follow a similar trend, the PZT-polymer composites have smaller values than the PZT-air composites because of their low d_h values; reasons have been explained in the previous section. Nevertheless, there is a significant improvement from a dense PZT (368×10^{-15} Pa^{-1}) compared with a 20% PZT-polymer composite (1821×10^{-15} Pa^{-1}). This improvement is more pronounced in the case of the 20% PZT-air composite, which has a value of $15\,095 \times 10^{-15}$ Pa^{-1}.

D. Receiving Sensitivity of Piezocomposites

The receiving sensitivities (M) of piezocomposites calculated from (4) are shown in Fig. 10. Sensitivity increases as PZT ceramic volume decreases. PZT-air piezocomposites had better sensitivity than PZT-polymer piezocomposites. The difference is more pronounced for <40% ceramic volume fraction, where complete 3-3 connectivity
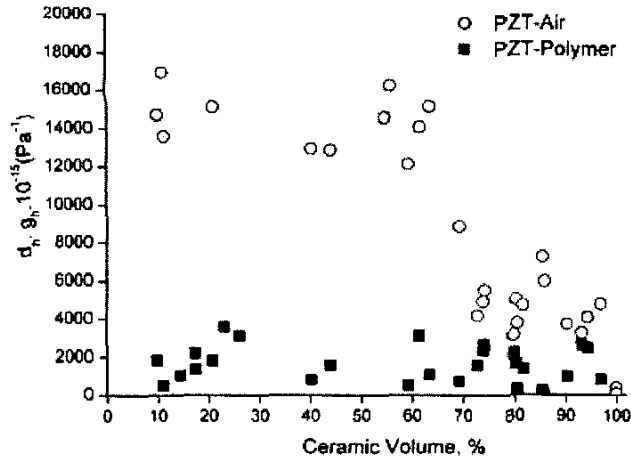
Fig. 9. Variation in hydrostatic figure of merit (FoM), $d_h \cdot g_h$, for PZT-air and PZT-polymer composites. Hydrostatic FoM had a clear maximum around 10 to 20% PZT for PZT-air composites.
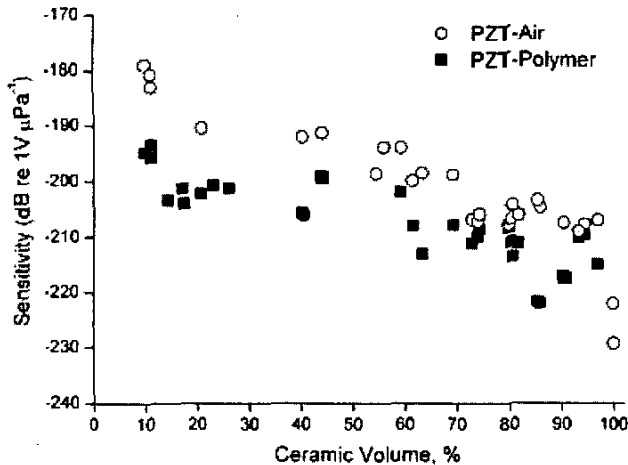

Fig. 10. Receiving sensitivity of PZT-air and PZT-polymer composites calculated from (4a).

Fig. 11. Receiving sensitivity of dense PZT, PZT-air, and PZT-polymer hydrophones along with a standard hydrophone.

starts to occur in the microstructure. This pronounced difference is due to high g_h values as shown in Fig. 8. This is a good example of how connectivity influences the receiving sensitivity of piezocomposites. The sensitivity values in Fig. 10 are shown in logarithmic scale (dB re 1V μ Pa^{-1}) so the difference between PZT-air and PZT-polymer piezocomposites appears to be small compared with g_h values in Fig. 8. The sensitivity of 20% PZT-air and PZT-polymer piezocomposites is -190 and -202 dB re 1V μPa^{-1}, respectively. In the 3-3 connectivity region, impregnating the porous PZT ceramic, reduced the sensitivity to $\sim$10 dB re 1V μPa^{-1}.

E. Receiving Sensitivity of Hydrophones

The measured receiving sensitivity of hydrophones, constructed from piezocomposites as in Table I, is shown in Fig. 11. It is apparent that radial resonances are sup-
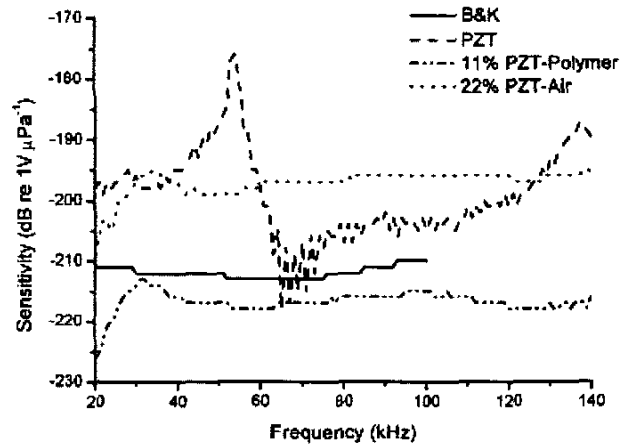
pressed in the composites when compared with the sharp resonance peak obtained for dense PZT. Suppression of the radial resonances gives a flatter, more uniform response to the hydrophone. In this case, the piezocomposite hydrophones showed a flat response up to 140 kHz. This set of results clearly demonstrates that reticulated piezocomposites can be beneficial for broadband receiving applications. The PZT-air hydrophone is more sensitive than the PZT-polymer hydrophone reaching a value of -198 dB re 1V μPa^{-1} at $\sim$100 kHz. The depth sensitivity of this type of device will be the subject of further work; PZT-air composites may be of use in shallow waters ($<$100 m). For deeper waters, PZT-polymer composite hydrophones would provide high mechanical strength and stability with a small compromise in receiving sensitivity. The sensitivity of both types of hydrophones might be further improved by optimized engineering design and the integration of a preamplifier.

As shown in Table I, the calculated sensitivities of PZT-air and PZT-polymer hydrophones are -193 and -199 dB re 1V μPa^{-1}, respectively, which are -5 and -16 dB re 1V μPa^{-1} lower than the measured values shown in Fig. 11. This can be attributed to the loading effect of the connecting cables because the capacitance of both composite hydrophones is low compared with the dense PZT hydrophone, and the difference is accordingly reflected in sensitivity. For the PZT hydrophone, where the measured and calculated sensitivities are -205 and -218 dB re 1V μPa^{-1}, the effect of cable loading was not significant on the measured sensitivity because of the high device capacitance (5050 pF), but the low piezoelectric voltage coefficient (g_h) resulted in a low calculated receiving sensitivity.

IV. Conclusions

PZT-air and PZT-polymer composites for hydrophone applications were successfully produced and characterized. Impregnation of a polymer phase into porous PZT ma-

trix had a detrimental effect on the measured properties, which has been attributed to the composite microstructure, connectivity, and the properties of the polymer filler. Nevertheless, both PZT-air and PZT-polymer composites showed improved values of g_h, FoM, and M compared with monolithic PZT.

Hydrophones showed similar behavior to piezocomposites in terms of receiving sensitivity. Hydrophones made from PZT-air composite had better sensitivity than those of PZT-polymer, with a value of -198 dB re 1V μPa^{-1}. The piezocomposite hydrophones also displayed a relatively flat broadband response up to 140 kHz because of the damping of radial resonances. The lower measured sensitivities of piezocomposite hydrophones were attributed to cable loading. Cost-effective manufacturing of reticulated 3-3 piezocomposites could make them a material of choice for shallow water receiving applications, and they are possibly low cost alternatives to all piezocomposites (such as 1-3s) for receiving applications.

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