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EVALUATION OF PLZT BASED **RAINBOW** CERAMIC SAMPLES DEVELOPED BY Dr. GENE HAERTLING AT CLEMSON UNIVERSITY

by

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Abstract

Dr. Gene Haertling, of Clemson University, USA, has recently shown that the selective reduction of one surface of a high lead containing piezoelectric ceramic wafer produces a stress-biased wafer with a unique domed structure that leads to high electromechanical displacement and enhanced load bearing capability. These ceramics have been called "rainbow" ceramics and their very high displacements make them very promising materials for actuators and some types of transducers. This report presents our characterisation of the dielectric, piezoelectric and hydrostatic properties of a number of lead lanthanum zirconate titanate (PLZT) based rainbow ceramics that were sent to us by Dr. Haertling. The samples had a nominal thickness of 0.48 mm and they were electroded with silver paint; some of them were glued to 1 mm thick brass or stainless steel discs. The samples exhibited a strong piezoelectric effect in the poling direction (d_{33} of the order of 10000 pC/N) under low uniaxial and hydrostatic stress but as the stress was increased there was a marked decrease in the strength of the piezoelectric response which passed through a minimum and then increased to the level of bulk PLZT ceramic. The plated samples showed a reasonable level of thickness mode electromechanical coupling and a hydrostatic voltage coefficient which, at low pressures, was considerably greater than that of PLZT. However, as the hydrostatic pressure was increased, the hydrostatic voltage coefficient decreased towards typical values for bulk PLZT. The rainbow ceramic material shows considerable promise for actuators and for shallow water sonar projectors.

Résumé

Le professeur G. Haertling, de l'Université Clemson aux États Unis, a récemment montré que la réduction sélective d'une surface d'un disque céramique piézoélectrique contenant un surplus de plomb produit un disque avec une tension interne et une structure avec la forme d'un dôme. Ces céramiques, qui ont été nommés céramiques "rainbow", montrent des grandes déformations électromécaniques et le pouvoir de porter des grandes charges. Ils sont ainsi des matériaux qui se révèlent prometteurs pour des actuateurs et pour quelques types de transducteurs. Ce rapport présente notre caractérisation des propriétés diélectriques, piézoélectriques et hydrostatiques d' un certain nombre d'échantillons des céramiques "rainbow", fabriqués du titanate zirconate de plomb et de lanthanum (PLZT), qui ont été fournis par le professeur Haertling. Les spécimens étaient munis d'électrodes d'argent peinturé et ils avaient une épaisseur nominale de 0,48 mm; quelques spécimens étaient joint à des disques de laiton ou de l'acier inoxydable et de 1 mm d'épaisseur. Ces spécimens ont montré un effet piézoélectrique fort dans la direction de polarisation (d_{33} de l'ordre de 10000 pC/N) sous des conditions de faible contrainte uniaxial ou hydrostatique mais l'effet piézoélectrique diminuait lorsque la contrainte augmentait et il est passé par un minimum avant d'augmenter jusq'aux niveau de l'effet piézoélectrique dans le PLZT céramique ordinaire. Les spécimens joint aux disques métalliques ont donnés des valeurs raisonnables de couplage électromécanique (résonance en épaisseur) et des valeurs du coefficient de tension hydrostatique, qui, aux basses pressions, se révélaient bien supérieures à la valeur du coefficient pour le PLZT. Cependant, lorsque la pression hydrostatique augmentait, le coefficient de tension hydrostatique diminuait vers les valeurs typique pour le PLZT. Le matériau céramique "rainbow" se révèle prometteur comme matériau pour les actuateurs et pour les projecteurs de sonar de hauts-fonds.

1. INTRODUCTION

Dr. Gene Haertling and his co-workers at Clemson University have recently produced a new type of ceramic bender which is capable of achieving very high axial displacements and sustaining moderate pressures[1]. This type of ceramic has been called a "rainbow" (**r**educed **an**d **i**nternally **b**iased **o**xide **w**afer) ceramic. Such a ceramic is obtained by the high-temperature chemical reduction of one surface of a high lead containing piezoelectric or electrostrictive ceramic wafer which produces a stress-biased dome like structure. The reduced (concave) side of the wafer can serve as one of the device electrodes. When a voltage is applied to a rainbow ceramic, the dome height varies as a function of the magnitude and polarity of the voltage and this motion is largely a consequence of the lateral contraction produced in the material due to the lateral piezoelectric coefficient d_{31}. Rainbow ceramics have been produced using ceramics such as lead zirconate titanate (PZT), lead lanthanum zirconate titanate (PLZT) and lead magnesium niobate (PMN). Two individual rainbow ceramic elements can be placed together to form a clamshell type device and these devices can be cascaded to form a linear actuator, with the individual devices being connected in series or in parallel. Single elements of rainbow ceramics, 0.2 mm thick, have produced displacements of 1 mm which represents a very high strain of 500%. Rainbow ceramics are also easy and cheap to produce and thus they show considerable promise as materials for actuators and perhaps for sonar activators.

We have obtained several specimens of rainbow ceramics from Dr. Haertling and this report presents the results of our characterisation of these specimens.

2. MEASUREMENTS

2.1 Specimens

The samples were in the form of discs of varying diameter with all the discs having a nominal thickness of 0.48 mm. All except one of the discs were electroded with silver paint. The disc without electrodes was used to find the density and this was determined to be 7575 ± 100 kg/m^3. All the samples had a dome like appearance; thus a disc of diameter 3.14 cm was found to have a radius of curvature of 0.21 ± 0.05 m. Some of the specimens were glued, along their edges, to 1 mm thick, flat, discs of brass or stainless steel. The specimens are described in Table 1. Samples 6 and 8 were damaged and could not be characterised.

2.2 Material Constants from Resonance Measurements

The impedances of the samples were measured as a function of frequency and the results for sample 1 are shown in Figure 1. In addition to radial and thickness mode resonances, the sample shows a bending mode resonance at approximately 30 kHz. The impedance spectra of the samples have been analysed using Smits' method[2] which was outlined in an earlier report[3].

Table 1: Sample Designation

Sample Number	Diameter (cm)/ Plate Diameter	PLZT	Electrode	Plate
1	1.31	1.0%La 53%$PbZrO_3$	Dupont 5504N Silver Epoxy	none
2	3.15	1.0%La 53%$PbZrO_3$	Dupont 5504N Silver Epoxy	none
3	3.14	1.0%La 53%$PbZrO_3$	Dupont 5504N Silver Epoxy	none
4	3.16 plate(3.4)	1.0%La 53%$PbZrO_3$	Dupont 5504N Silver Epoxy	brass
5	1.31 plate(1.32)	1.0%La 53%$PbZrO_3$	Dupont 5504N Silver Epoxy	brass
6	2.27 plate(2.54)	5%La 56%$PbZrO_3$	Dupont 5504N Silver Epoxy	brass
7	3.16	1.0%La 53%$PbZrO_3$	none	none
8	3.17 plate(3.18)	1.0%La 53%$PbZrO_3$	Dupont 5504N Silver Epoxy	steel

An analysis of the thickness resonances gave the material constants shown in Table 2 in which k_t is the electromechanical coupling constant in the thickness mode, c_{33}^{D} is the elastic stiffness at constant dielectric displacement, ε_{33}^{S} is the permittivity in the poling direction at constant strain and h_{33} is a piezoelectric coefficient.

Table 2: Thickness material constants for the Clemson Rainbow PLZT specimens measured at 4 MHz and 20°C

	Sample 1 Electrode Dia. 12.9mm	Sample 2 Electrode Dia. 28.9mm	Sample 3 Electrode Dia. 28.9mm
k_t	0.359(1-0.12i)	0.355(1-0.31i)	0.327(1+0.12i)
c_{33}^{D} (10^{11}N/m²)	1.35(1+0.020i)	1.06(1+0.069i)	1.19(1+0.068i)
ε_{33}^{S} (10^{-9}F/m)	6.39(1-0.33i)	6.87(1-1.1i)	11.3(1-0.35i)
h_{33} (10^{9}V/m)	1.62(1+0.049i)	1.23(1+0.15i)	0.99(1+0.33i)

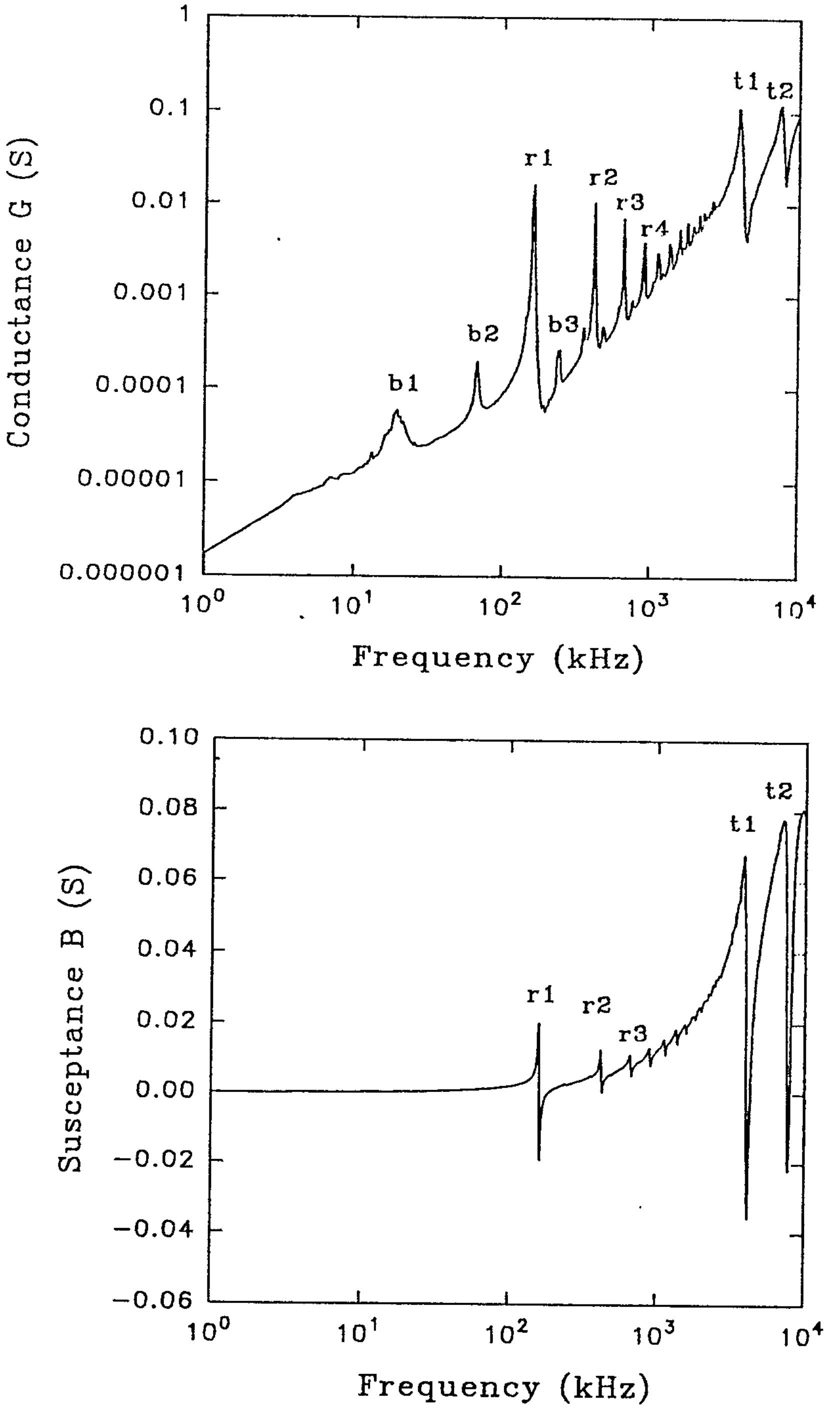

Figure 1. The impedance spectrum for sample 1. The peaks labelled **b**, **r** and **t** correspond to bending, radial and thickness mode resonances.

Figure 2 shows the thickness resonance for sample 1. The fit is acceptable around the fundamental mode but there is significant dispersion in the dielectric and piezoelectric elastic constants in the MHz frequency region. Besides, Table 2 shows that there are large differences in the material constants measured for the various samples. This is likely due to small differences in curvature and aspect ratio between the samples. The curvature is a result of the reduction process and small variations in composition and processing conditions would produce differences in the curvatures of the samples which would very significantly affect the material constants. In Figure 2, the first thickness resonance occurs at around 4 MHz and it follows that a non-dispersive material would have a second resonance at about 12 MHz whereas the figure shows that the second resonance occurs at approximately 7.5 MHz so that the elastic stiffness constant has decreased by a factor of 2 over a 3.5 MHz frequency interval. We note also that the baselines for the data and the fit in Figure 2 differ substantially at frequencies higher than the first resonance frequency and this leads to the conclusion that the permittivity of the material is also

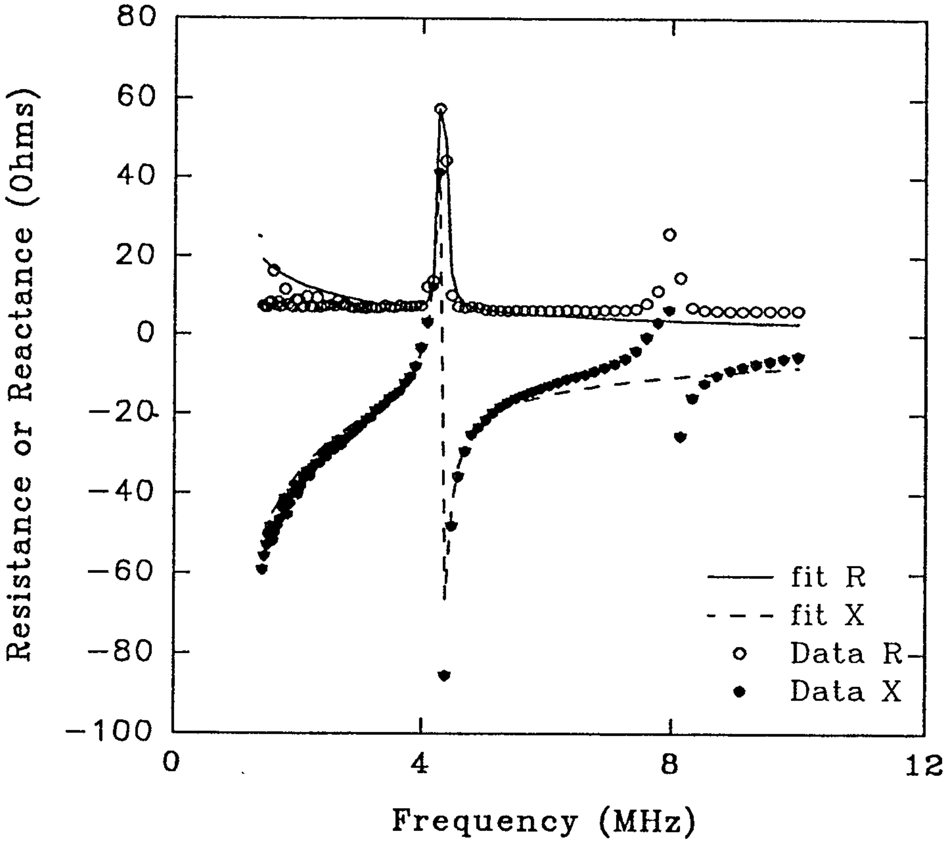

Figure 2. The resistance and reactance versus frequency for sample 1. The fit is reasonable around the fundamental thickness mode. The material constants are dispersive and they are accurate around frequencies in the 3 to 5 MHz interval.

significantly dispersive. The electromechanicalcoupling constant k_t increases as the frequency is increased. Another interesting feature is that the second thickness mode resonances of the larger samples are inductive and yield negative values of permittivity; this is due to the high conductivities of these samples at high frequencies.

The radial modes of resonance of three samples were analysed using the technique that we have developed earlier[4] and the results are shown in Table 3. However it should be stressed that the geometry of these dome shaped samples does not correspond strictly to the geometry assumed in deriving the equations for the radial mode resonance. Once again we find substantial differences between the material constants of the different samples and these are likely to be caused by the different concavities of the discs. Sample 1, which has the smallest diameter of the three samples, was the least concave and it is therefore likely that the values shown for this sample approach the values for the bulk material. Figure 3 shows the data and the fit for the radial mode resonance of sample 1. As in the case of the thickness mode, the fit is very good near resonance. In order to get a reasonable fit to the data throughout the frequency range, (i) the imaginary component of the complex series resonance ratio r_s, and hence Poisson's ratio, was set to zero, (ii) the complex series resonance frequency, f_s, was adjusted slightly and (iii) the measured value of the admittance away from resonance was adjusted to take account of the dispersion in the dielectric properties.

Samples that were bonded to plate electrodes had thickness resonances that saturated the measuring circuit while their radial modes were smaller than for the unbonded samples. This suggests that the electrode plate acts to clamp d_{31} more than d_{33} with a resulting enhancement in k_t..

Table 3: Radial mode material constants for the Clemson Rainbow PLZT specimens measured at 20°C

	Sample 1	Sample 2	Sample 3
s_{11}^{E} x10^{11}m^2/N	1.55(1-0.023i)	1.81(1-0.023i)	2.15(1-0.038i)
s_{12}^{E} x10^{11}m^2/N	-0.517(1-0.023i)	-0.742(1--0.023i)	-1.22(1-0.038i)
d_{31} x10^{12}C/N	-140(1-0.088i)	-123(1-0.085i)	-83(1-0.22i)
ε_{33}^{T} x10^{9}F/m	13.3(1-0.0915i)	11.6(1-0.077i)	7.8(1-0.21i)
σ^{P}	.334	.410	.566
k_p	0.52(1-0.043i)	0.48(1-0.047i)	0.43(1-0.11i)

2.3 Dielectric Measurements

The capacitances of the specimens were measured at a frequency of 1000 Hz at room temperature. The average values for the permittivity, the dielectric constant and the loss tangent of samples 1, 2 and 3 are given in Table 4.

Table 4: Dielectric constants (averaged over samples 1, 2 and 3)

Property	Units	Value
Permittivity ε_{33}^{T}	10^{-9}F/m	14 ± 1
Dielectric Constant K_{33}^{T}		1540 ± 110
Loss tangent $\tan\delta$		0.086 ± 0.012

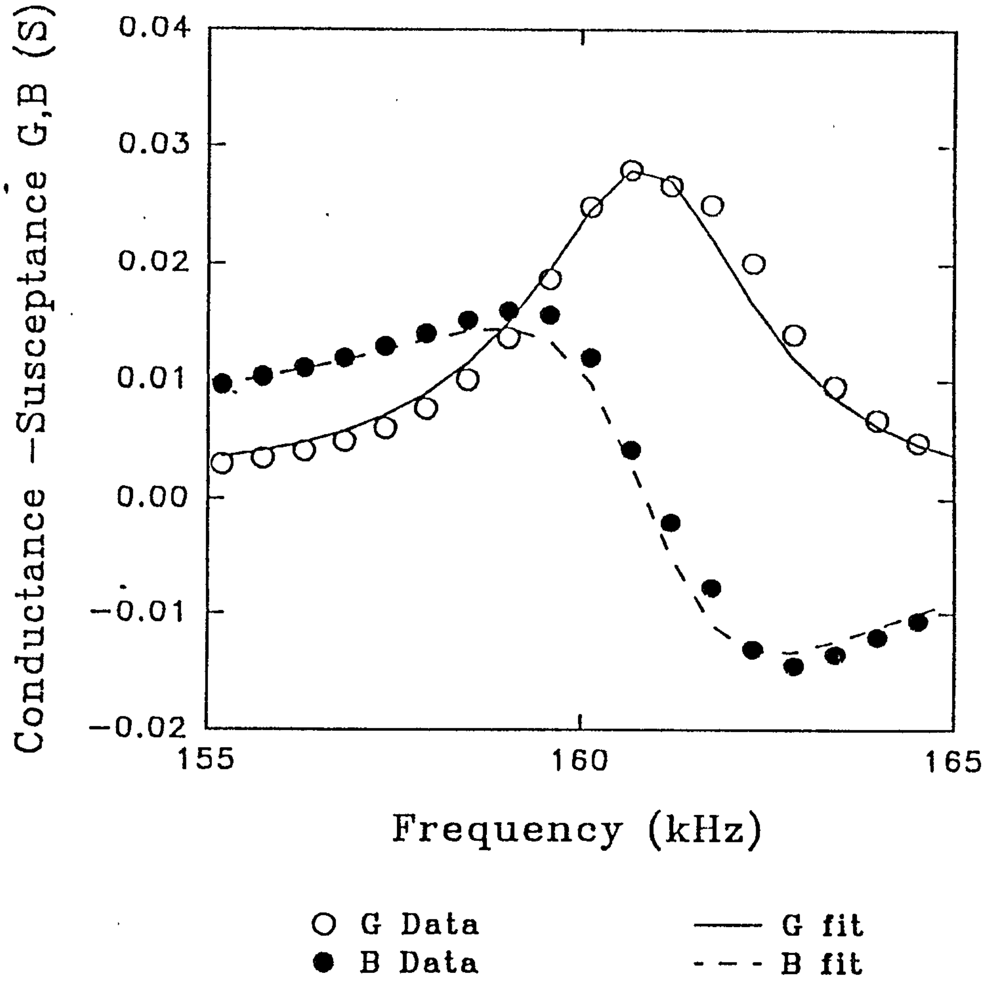

Figure 3. The fundamental radial mode resonance and the fit for sample 1.

2.4 Measurement of the Piezoelectric Charge Constant in the 33 direction

The value of the piezoelectric charge constant, d_{33}, for the material was obtained by using a point force head on a Berlincourt type d_{33} meter which was operated at a frequency of 200 Hz. The value of d_{33} was found to vary over the surface of the samples; to find if this was due to coupling to the bending mode of the sample, d_{33} measurements were made at 12 points spaced 1mm apart along a diameter of the domed sample. Our results are shown in Figure 4 where the three curves represent the values obtained (a) when the measurements were made with the sample curvature facing downwards so that the sample formed a cavity with the base plate of the meter with the positive polarity of the terminal towards the point head (indicated as + up data in the figure), (b) when the measurements were made with the sample curvature facing upwards (+ down data in the figure) and (c) the average of the two measurements made in (a) and (b).

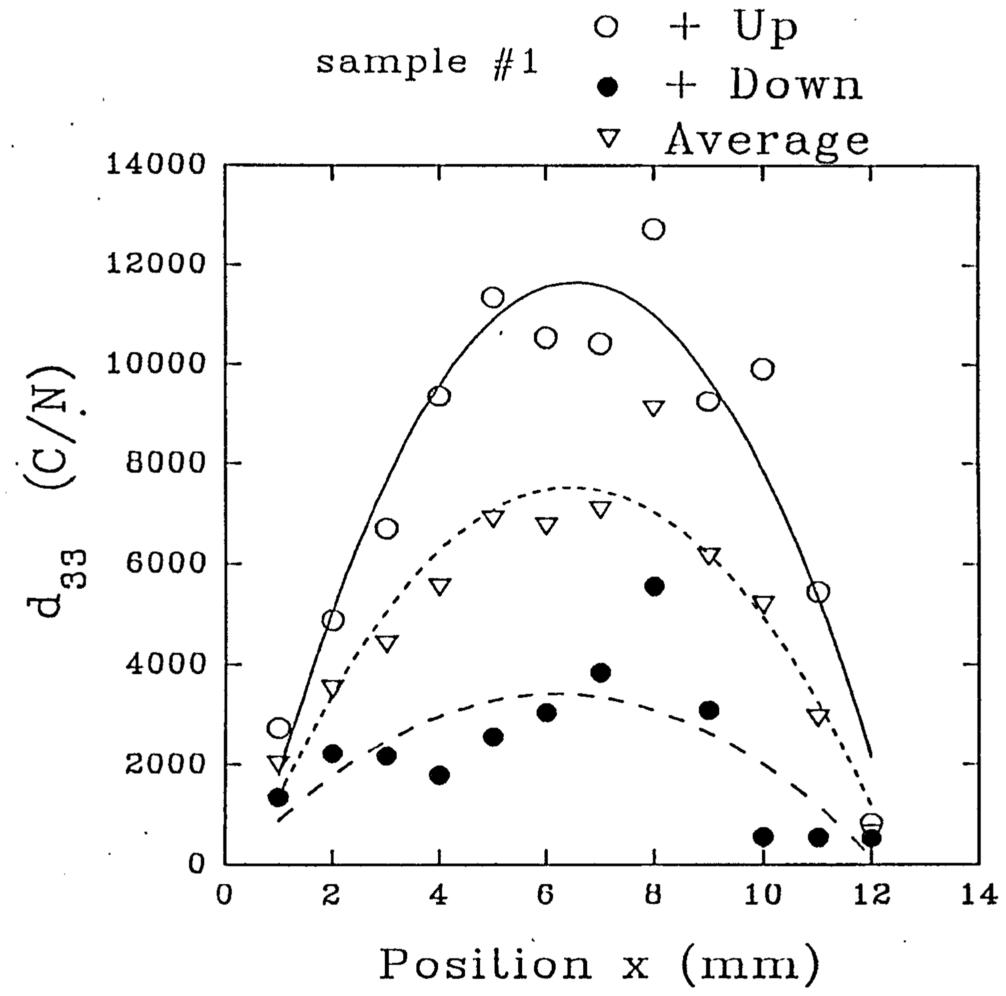

Figure 4. The d_{33} value as a function of the distance along a diameter of sample 1. The significance of the three curves has been explained in the text.

Figure 4 shows that the apparent d_{33} values are quite large and can reach up to 12000 pC/N at the centre of the specimen. It is likely that this large value is due to the sum of the normal uniaxial compression of the ceramic material and the bending modes of the dome shaped sample. In an attempt to elucidate this better, we have studied the effect of stress on this type of rainbow material by determining the d_{33} value as a function of uniaxial compression. This has been done by measuring the piezoelectric voltage generated by a rainbow sample, connected electrically to a standard 1 μF shunt capacitor, as a function of an uniaxial compressive force applied to the specimen. Our results for sample 2 are shown in Figure 5. The d_{33} values can be

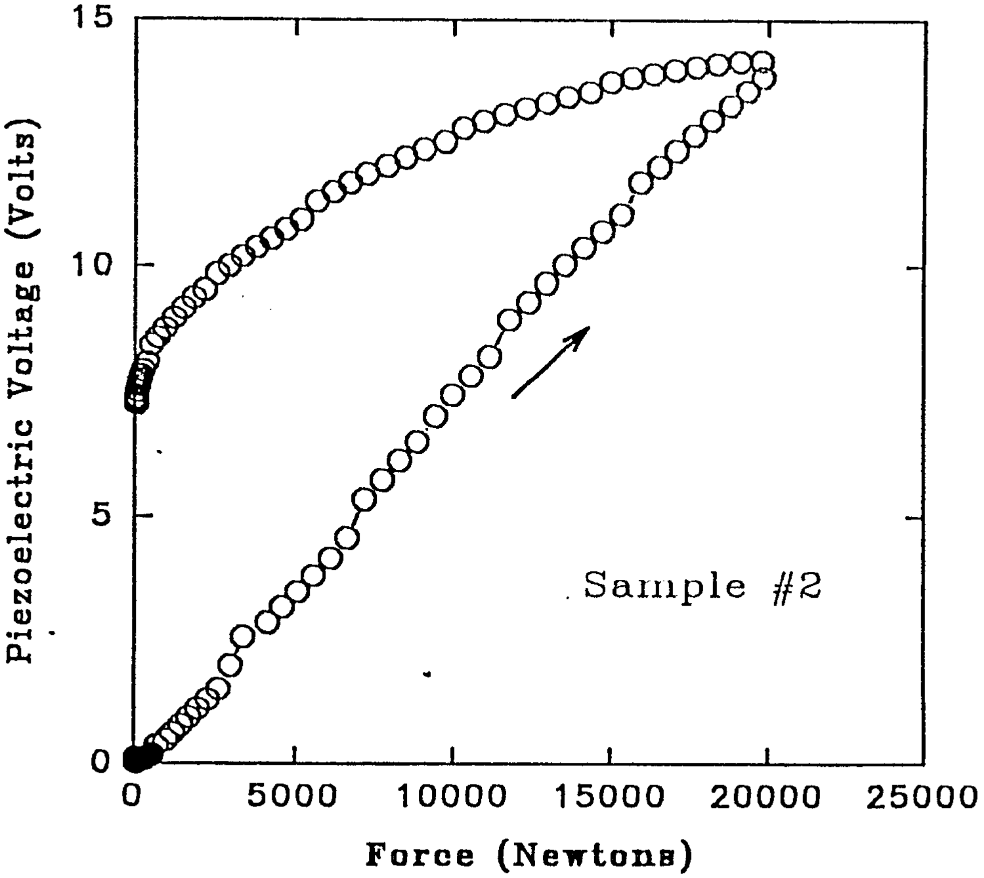

Figure 5. The piezoelectric voltage as a function of the compressional force applied to sample 2.

calculated from the voltage curve shown in Figure 5 by using the expression

$$d_{33} = C^T\left(\frac{dV}{dF}\right)$$

where *(dV/dF)* is the derivative of the voltage - force curve in Figure 5, and $C^T = C^{Sample} + C^{Shunt}$ is the sum of the sample and shunt capacitances. Figure 6 shows the values of d_{33} as a function of the applied uniaxial stress calculated from the part of the curve in Figure 5 which corresponds to increasing force. It can be seen that d_{33} has a value of about 10000 pC/N at low force, it then decreases rapidly as the dome shaped sample is flattened out as a result of increasing force and it passes through a minimum at a force of about 200 N from which point it rises up to typical ceramic values as the sample undergoes compression. We can therefore conclude that the large d_{33} values are indeed caused by the bending of the dome shaped sample when a stress is applied; after the rainbow material has become flat, it begins to act like a plain bulk ceramic such as PZT.

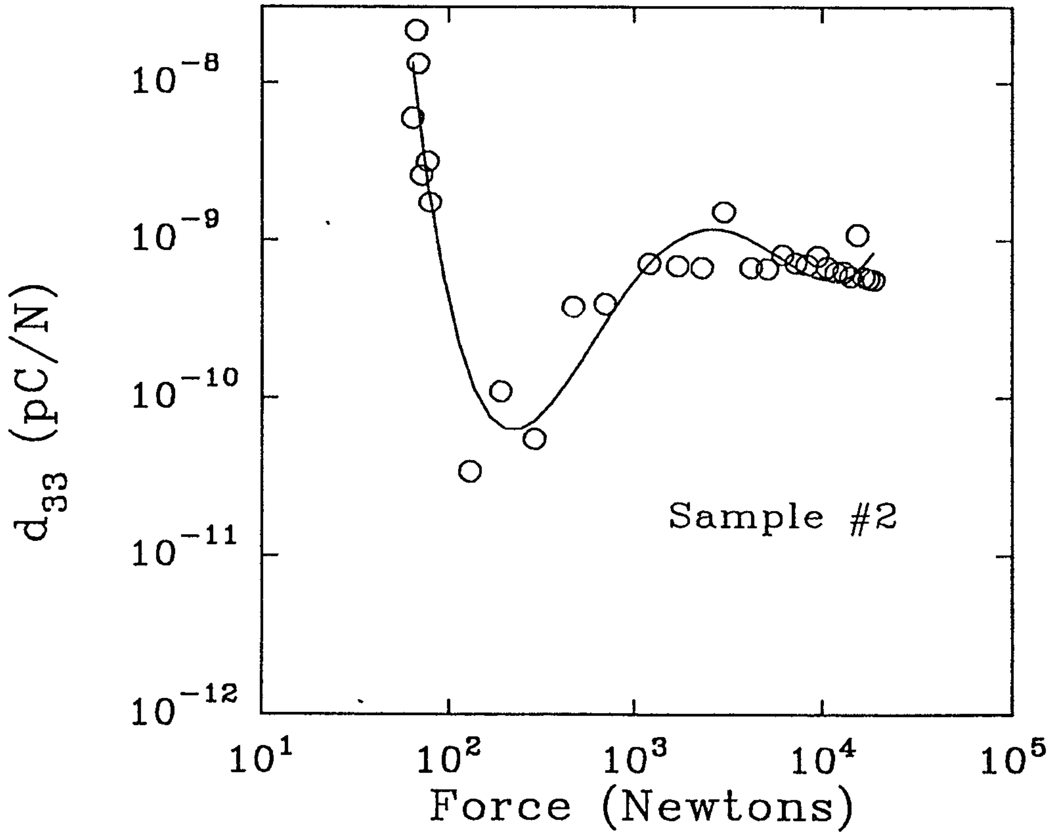

Figure 6. d_{33} as a function of the compressional force applied to sample 2.

The hysteresis in the voltage-force curve of Figure 5 is due to the time dependence of the response of the ceramic to stress. Figure 7 shows the time-dependent piezoelectric response of a rainbow ceramic to a 20 kN force step: the piezoelectric voltage shows a corresponding step and then increases logarithmically with time until the R-C time constant of the electrical circuit begins to dominate. This behaviour is very similar to the time-dependent behaviour of bulk PZT[5].

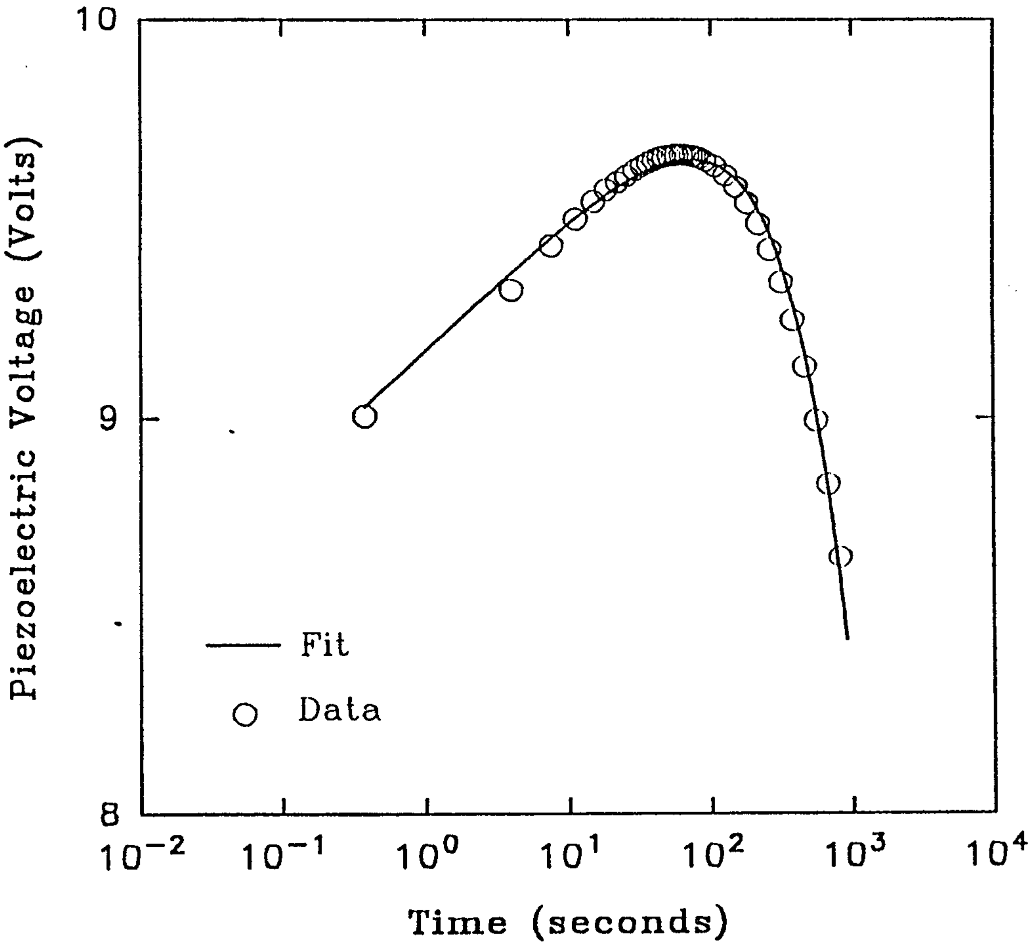

Figure 7. The time-dependent voltage response of the rainbow ceramic to a stress step. The response is similar to that of PZT indicating that, once flattened, the rainbow material behaves like bulk PZT.

2.5 Hydrostatic Properties

We have measured the hydrostatic voltage constant, g_h, of the rainbow material at 400 Hz using the method outlined in an earlier report[3] and our results for samples 1, 3 and 5 are shown in Figure 8. Two series of measurements were carried out on sample 1: the results indicated by "+ up" correspond to the dome shaped rainbow ceramic being placed with its curvature facing down and forming a small cavity with the base plate of the apparatus while the results indicated by "+ down" correspond to the rainbow ceramic sitting on the base plate with its curvature facing up. The hydrostatic voltage response is the sum of the contributions arising from the bending of the dome shaped rainbow and the compression of the ceramic itself. In the case of sample 1 the bending effects will be small since the edge of the rainbow ceramic can move laterally and so the g_h value is close to that of standard bulk PZT ceramic. The small difference between the two series of measurements on sample 1 is probably due to different contributions from the bending of the specimens. Samples 1 and 3 are similar in that both were not bonded to a base plate so that the static hydrostatic pressures would be the same on both faces and the

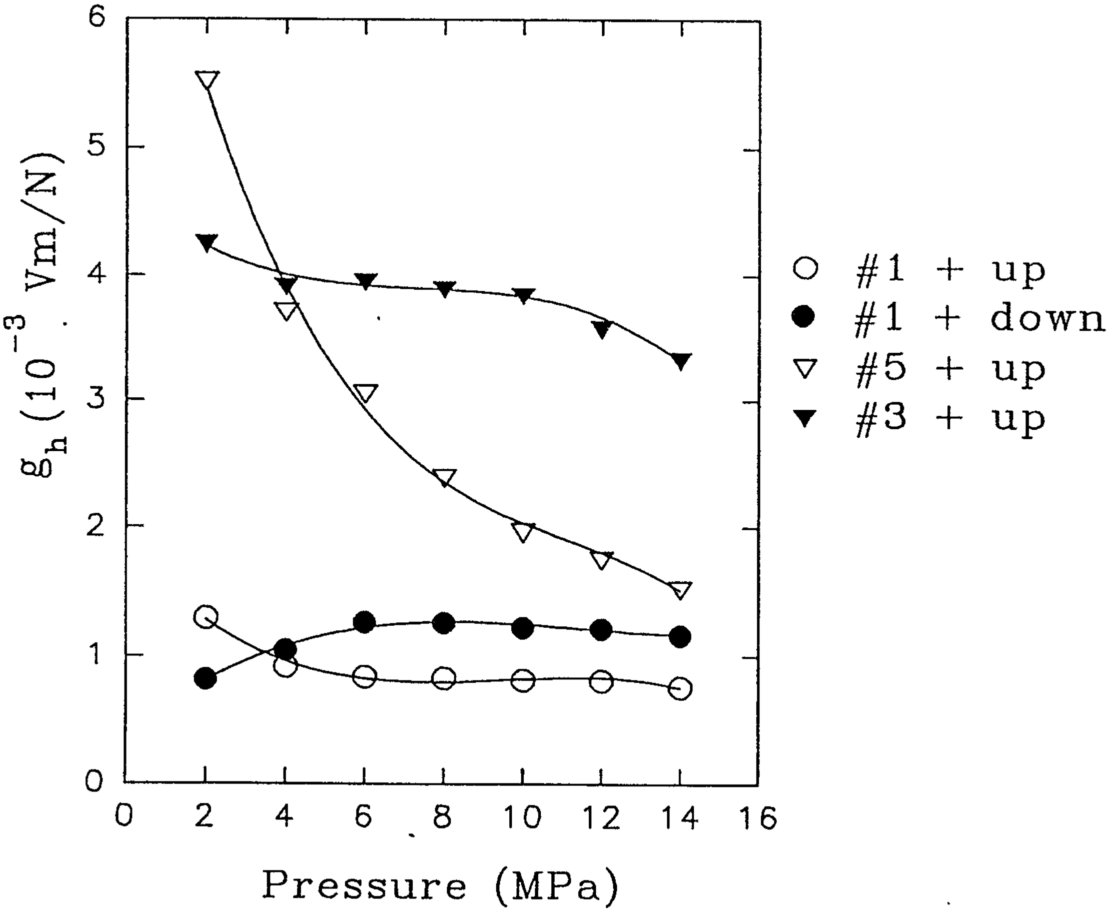

Figure 8. The hydrostatic voltage coefficient, g_h, as a function of the hydrostatic pressure for different samples and orientations.

dome shaped structure does not undergo any flattening. Sample 3, which has the bigger radius, has a larger value of g_h and this is perhaps due to the larger bending deflections which are possible in this case. Sample 5 is a rainbow ceramic of the same radius as sample 1 but it is bonded to a brass plate about 1 mm thick so that the static hydrostatic pressure is not now transmitted to the inner surface of the rainbow and the rainbow ceramic will gradually flatten as the external static pressure is increased. At low pressures the flattening is negligible but since the rim of the rainbow is bonded the bending response to the signal is considerably greater than in the case where the rainbow is not bonded (as in sample1) and hence the much larger value of g_h. As the pressure increases, the rainbow gradually flattens out, the bending contributions decrease and the g_h value approaches that of a normal bulk PZT ceramic.

Figure 9 shows the hydrostatic figure of merit, $g_h d_h$, of samples 1, 3 and 5. We note that the small rainbow ceramic (sample 1) has a figure of merit that is substantially lower than that of PZT while the larger rainbow ceramic (sample 3) has a figure of merit which is comparable to that of PZT.

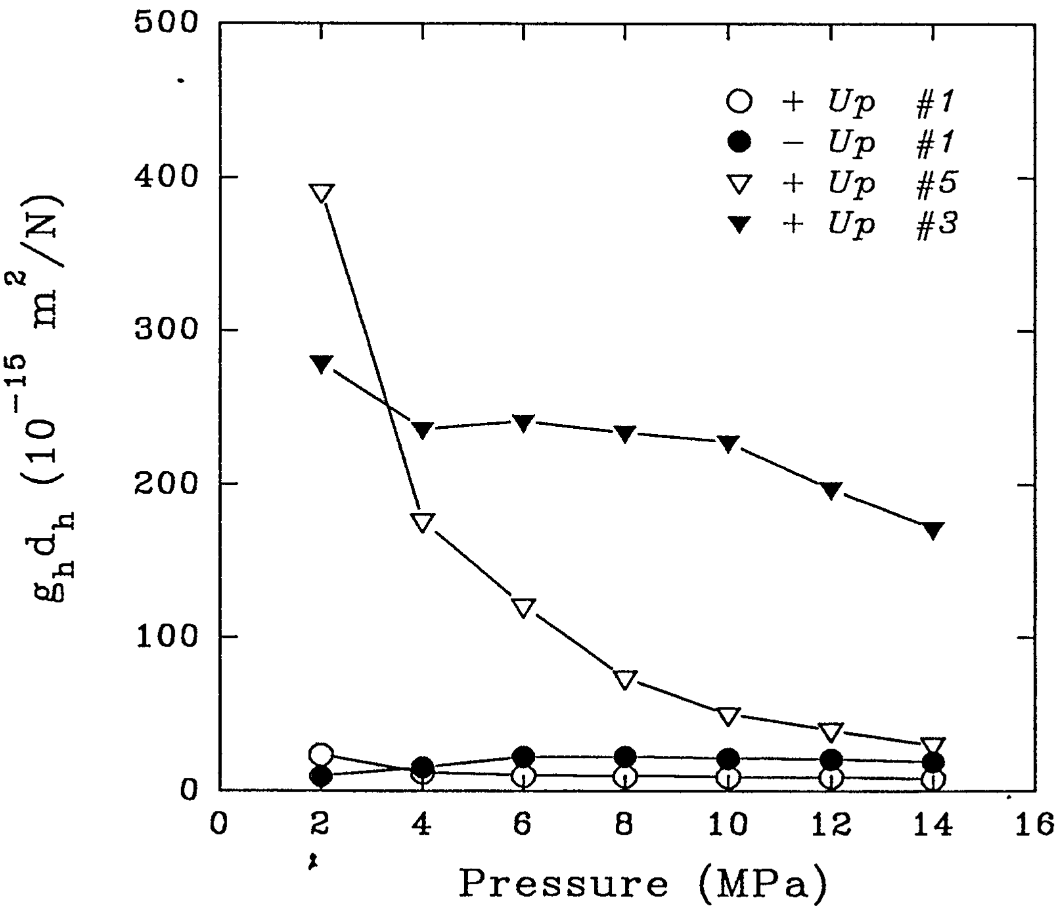

Figure 9. The hydrostatic figure of merit, $g_h d_h$, of samples 1, 3 and 5.

Finally, the g_h and $g_h d_h$ values for sample 4 are shown as a function of pressure in Figures 10 and 11. This sample is a large rainbow ceramic, 3.16 cm in diameter, bonded to a 1mm thick brass plate and its behaviour is qualitatively similar to that of sample 5: at high pressures the values are slightly lower than the nominal values for normal bulk PZT but they rise dramatically at low pressures.

It should be pointed out that the two contributions to charge generation (bending and compression) are not independent and this explains the minima in d_{33} and in g_h which are visible in Figures 6 and 10.

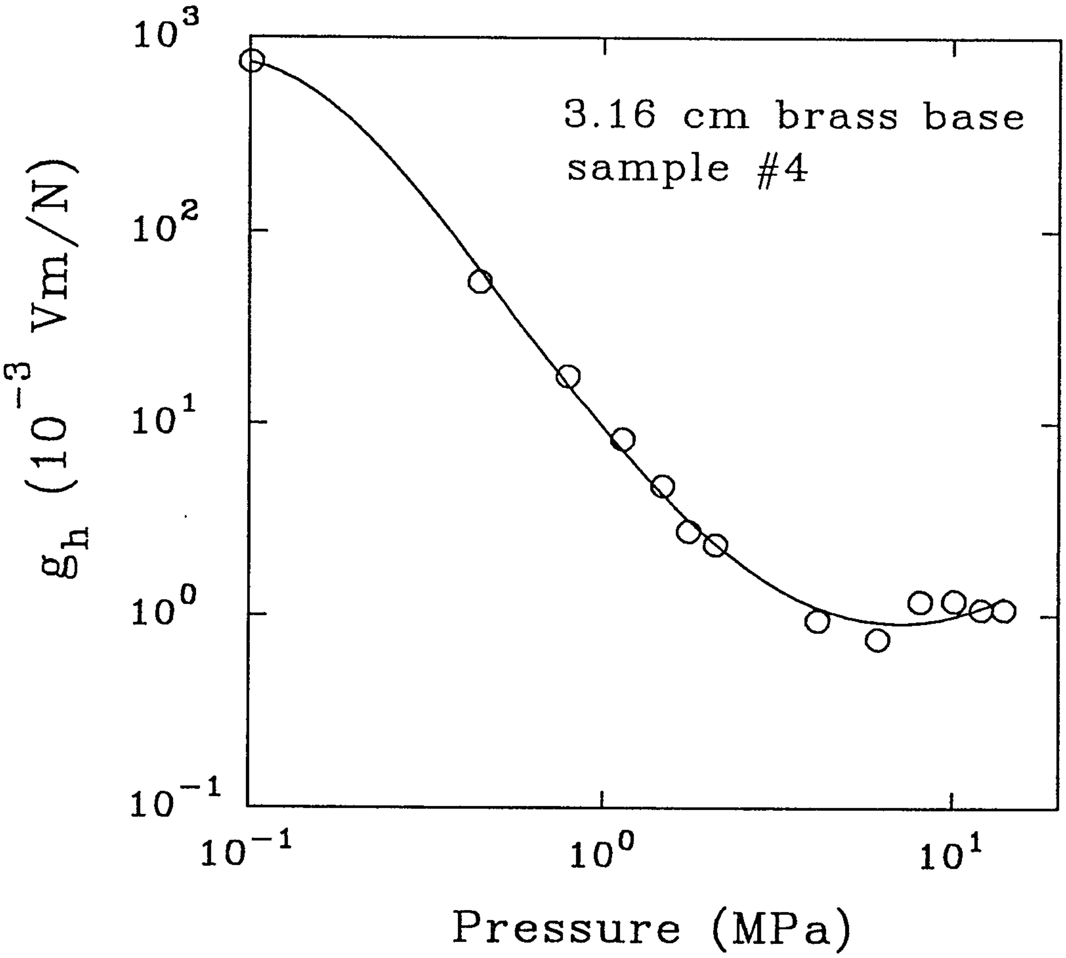

Figure 10. The hydrostatic voltage coefficient, g_h, as a function of pressure for sample 4.

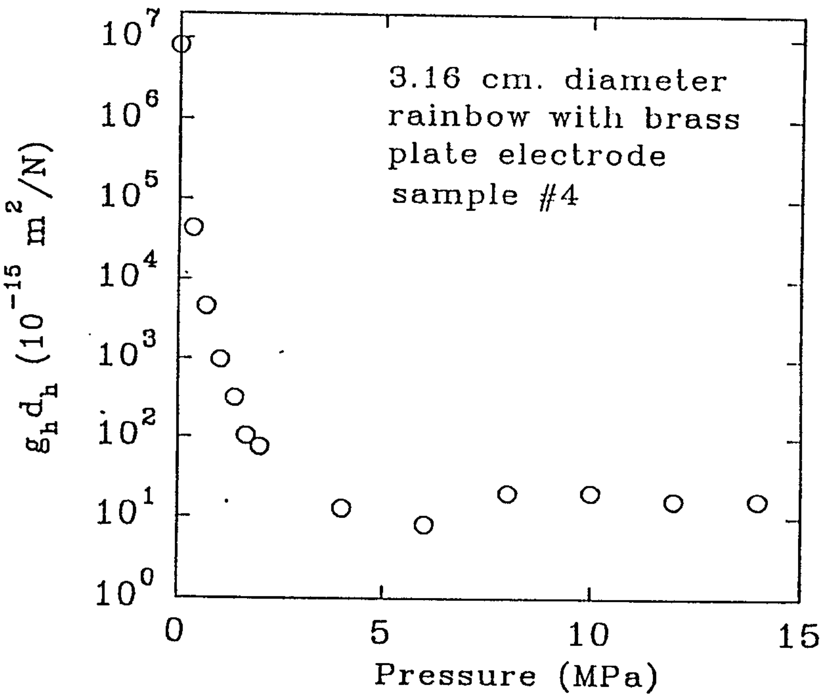

Figure 11. The hydrostatic figure of merit, $g_h d_h$, as a function of pressure for sample 4.

3. CONCLUSIONS

A set of lead lanthanum zirconate titanate (PLZT) “rainbow” ceramic samples have been analysed. These rainbow ceramics have been processed to contain pre-stresses and they have a geometry of slightly dome-shaped discs. The samples were made by Dr. Gene Haertling and his co-workers at Clemson University, USA, who have patented the manufacturing process. The samples exhibit a strong piezoelectric effect in the poling direction under low planar and hydrostatic pressures. As the pressure is increased there is a marked decrease in the strength of the piezoelectric response. The larger piezoelectric response at low planar stresses is thought to be due to a bending mode corresponding to flattening of the dome shaped samples and a consequent release of charge. At larger stresses, with the samples already flattened, the sample only suffers compression and the charge released is proportional to the d_{33} of of the ordinary bulk PLZT. There is a minimum in the charge generated as a function of pressure which suggests that the bending and compression modes are coupled with the strain being relieved by the bending action of the monomorph. When the rainbow ceramic is completely flattened at high pressures, compression becomes the dominant mode of charge production.

The resonance curves for the material were somewhat distorted due to their dome shape and the consequent presence of additional bending modes. Samples which were not bonded to any base plate were used to determine the material constants for the radial and thickness modes of operation and these constants were found to exhibit geometric effects and dispersion.

The rainbow samples that were not bonded to a base plate had hydrostatic properties in the same range as ordinary bulk PLZT with some variation depending on the orientation of the sample in the g_h apparatus. Typical values for g_h were in the range 1 to 4 x 10^{-3} Vm/N. However, the rainbow samples that were bonded to electrode plates showed substantially larger values of g_h and $g_h d_h$ at low pressures. But when the static pressure increased, their g_h and $g_h d_h$ values decreased to values similar to those of bulk PLZT.

The dielectric properties of the rainbow ceramic samples were similar to those of bulk PLZT.

In conclusion, the rainbow ceramic material shows considerable promise as an actuator material where large displacements are required (solid state speakers, pumps, switches, positioners, etc..) and possibly for shallow water sonar projectors. The large pressure dependences exhibited by the material reduce its applicability in deep water sonar. However, with proper design, it might be possible to maintain a pressure-independent sensitivity that will be somewhat greater than that of PZT, the current standard in sonar transducer materials.

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Dr. Gene Haertling, of Clemson University, USA, has recently shown that the selective reduction of one surface of a high lead containing piezoelectric ceramic wafer produces a stress-biased wafer with a unique domed structure that leads to high electromechanical displacement and enhanced load bearing capability. These ceramics have been called "rainbow" ceramics and their very high displacements make them very promising materials for actuators and some types of transducers. This report presents our characterisation of the dielectric, piezoelectric and hydrostatic properties of a number of lead lanthanum zirconate titanate (PLZT) based rainbow ceramics that were sent to us by Dr. Haertling. The samples had a nominal thickness of 0.48 mm and they were electroded with silver paint; some of them were glued to 1 mm thick brass or stainless steel discs. The samples exhibited a strong piezoelectric effect in the poling direction (d33 of the order of 10000 pC/N) under low uniaxial and hydrostatic stress but as the stress was increased there was a marked decrease in the strength of the piezoelectric response which passed through a minimum and then increased to the level of bulk PLZT. The plated samples showed a very high level of thickness mode electromechanical coupling and a hydrostatic voltage coefficient which, at low pressures, was considerably greater than that of PLZT. However, as the hydrostatic pressure was increased, the hydrostatic voltage coefficient decreased towards typical values for bulk PLZT. The rainbow ceramic material shows considerable promise for actuators and for shallow water sonar projectors.

14. KEYWORDS, DESCRIPTORS or IDENTIFIERS (technically meaningful terms or short phrases that characterize a document and could be helpful in cataloguing the document. They should be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location may also be included. If possible keywords should be selected from a published thesaurus, e.g. Thesaurus of Engineering and Scientific Terms (TEST) and that thesaurus-identified. If it not possible to select indexing terms which are Unclassified, the classification of each should be indicated as with the title).

Piezoelectric ceramics
Actuator applications
Hydrostatic properties
Dielectric properties
Figure of merit

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