An Improved Thick-Film Piezoelectric Material by Powder Blending and Enhanced Processing Parameters

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***Abstract*—This paper details improvements of the d_{33} coefficient for thick-film lead zirconate titanate (PZT) layers. In particular, the effect of blending ball and attritor milled powders has been investigated. Mathematical modeling of the film structure has produced initial experimental values for powder combination percentages. A range of paste formulations between 8:1 and 2:1 ball to attritor milled PZT powders by weight have been mixed into a screen-printable paste. Each paste contains 10% by weight of lead borosilicate glass and an appropriate quantity of solvent to formulate a screen printable thixotropic paste. A d_{33} of 63.5 pC/N was obtained with a combination of 4:1 ball milled to attritor milled powder by weight. The improved paste combines the high d_{33} values of ball and the consistency of attritor milled powder. The measured d_{33} coefficient was further improved to 131 pC/N by increasing the furnace firing profile to 1000°C, increasing the poling temperature to 200°C, and using gold cermet and polymer electrodes that avoid silver migration effects and repeated firing of the PZT film.**

I. Introduction

Screen printable piezoelectric materials were first reported in 1987 [1] and have since found use in many applications, including actuators [2] and micromachined silicon devices [3]. The basic formulation of the paste is well-known [4] and involves mixing piezoelectric material in powder form with suitable binders and thick-film vehicles. Both cermet pastes using glass binders [5] and polymer pastes with the active material held within a polymer matrix [6] have been demonstrated.

The most common piezoelectric material used in the paste preparation is lead zirconium titanate ($PbZr_xTi_{1-x}O_3$ or PZT). The piezoelectric, mechanical, and electrical properties of the PZT material will depend upon the composition of the compound. The base material used in this investigation is Morgan Electro Ceramics, Southampton, UK, PZT-5H which exhibits high levels of piezoelectric activity but has a low Curie temperature compared to alternative compositions. A summary of the properties of common bulk PZT materials is given in Table I [7].

TABLE I
Typical Values for Lead Zirconate Titanate Bulk Materials [7].

Characteristic	Material type		
	PZT4D	PZT5A	PZT5H
d_{33} (pC/N)	315	374	593
d_{31} (pC/N)	−135	−171	−274
T_c (°C)	320	365	195
k_p	0.57	0.6	0.65
k_T	0.675	0.71	0.75
N_p (Hz-m)	2180	1960	1965
K_{33}^T ($\times 10^{-15}$)	1300	1700	3400

This paper presents details of a fundamental investigation into the influence of the physical nature of the PZT powder on the piezoelectric properties of the screen-printed film. The physical nature of the powder (i.e., particle size, distribution, and shape) is determined by the processing techniques used in its preparation. Piezoelectric powders are formed by a complex series of processes and are traditionally used in the fabrication of bulk PZT piezoceramics. Powders of the constituent elements are first mixed in the required percentages. This powder mix is calcinated then milled into the final PZT powder form. This milling process defines the physical nature of the powder. Several different milling processes have been evaluated: ball, jet, and attritor.

II. Milling Process

The ball milling process involves the PZT materials being mixed in a slurry and tumbled or shaken with a suitable milling media such as sand, steel, zirconia, or alumina in a horizontally rotating mill. This process is defined as a soft process due to the nature and speed of the mill. Ball milling results in a smooth, rounded particle shape.

Jet milling subjects the powder particles to high-pressure jets of air where they collide and wear against each other in an abrasive process. The mill is constructed in such a way that, when the particles have been sufficiently reduced in size, they will drop out of the mill. This process produces fine particles, exhibiting uneven edges in comparison to the ball milled powder.

Attritor milling is similar to ball milling, except instead of the mill itself rotating, a vertical shaft rotates inside the drum. This process is typically used to further reduce the size of existing powder particles and to obtain a more

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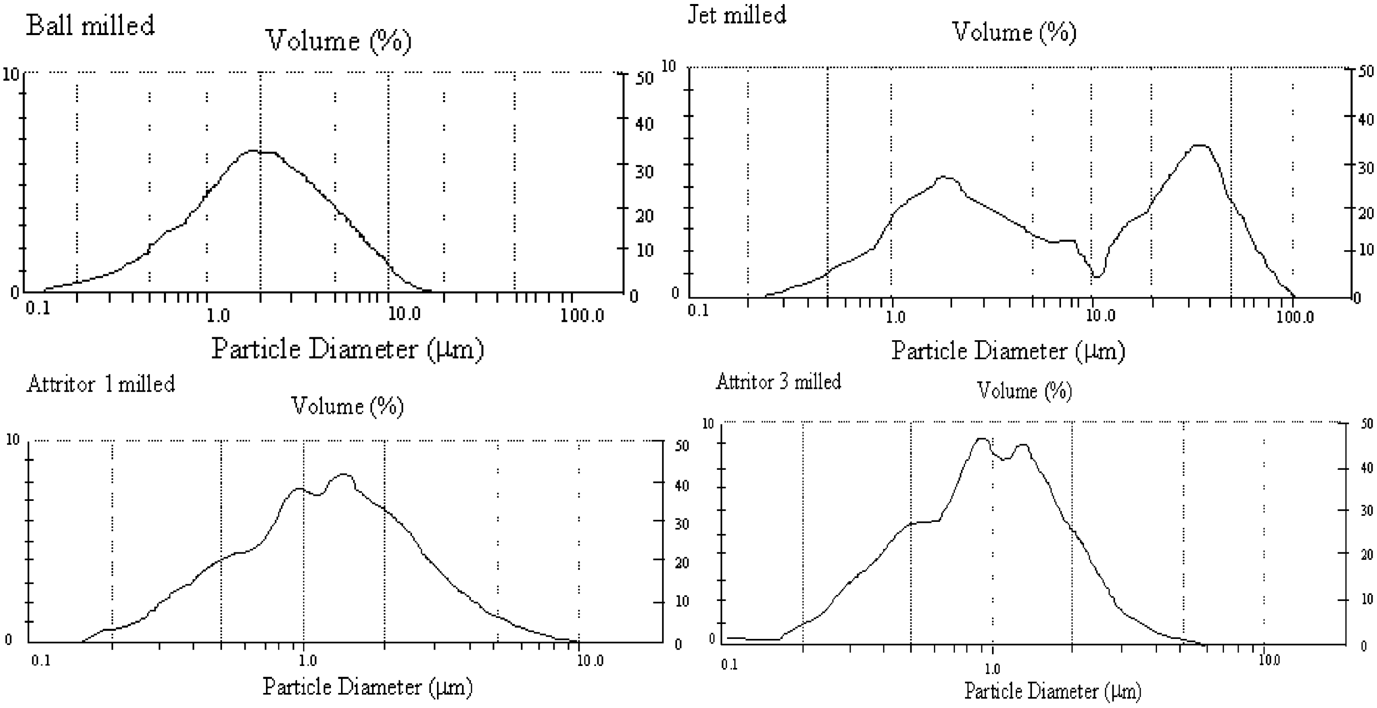

Fig. 1. Particle size distributions for ball, jet, attritor1, and attritor3 milled powders.

uniform size distribution. It is standard practice to feed the powder through the mill a number of times to achieve a tighter distribution. This paper presents details of powders that have been attritor milled once and three times, henceforth denoted attritor1 and attritor3.

The particle size and distribution of powders from each of the above processes were measured using a Malvern Mastersizer X (Malvern Instruments Ltd., Malvern, UK) [8] at Morgan Electro-Ceramics Thornhill plant. The results are shown in Fig. 1. The ball milled particles average 2 μm in diameter, jet 4.3 μm, attritor1 1.2 μm and attritor3 1 μm.

Previous work by Torah *et al.* [9] has shown that the ball milled powder produces the largest d_{33} values with the attritor3 milled powder producing the most consistent d_{33} values. The jet milled powder was considered to be poor in comparison, producing low and inconsistent d_{33} results due to the rough and uneven nature of the milling process. Therefore, it has not been considered in this investigation.

III. Powder Blending

Following the results from previous work [9], the next stage in improving the piezoelectric properties of the thick-film devices considered the combination of ball milled and attritor3 milled powders. The larger particle size associated with the ball milled powder results in increased piezoelectric responses. Therefore, it was important to maintain the ball milled particle as the dominant particle within any powder combination. The attritor3 particles can be used to fill any voids between the ball milled particles, thus resulting in an increased film density and improving its mechanical coupling and piezoelectric properties. Hence, it was anticipated that, by combining these two powders, we would obtain the high d_{33} values associated with ball milled powder and the consistency achieved with attritor3 powders.

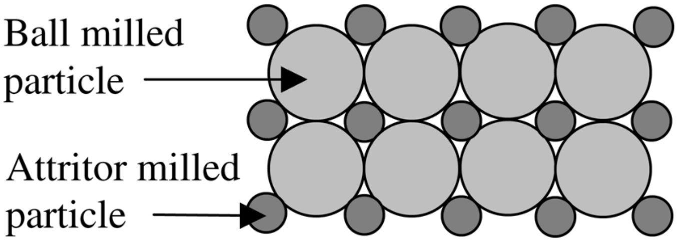

Fig. 2. Ideal 2-D particle distribution for ball and attritor milled PZT.

Therefore, the paste consists of a combination of ball milled, attritor3 milled, and Ferro CF7575 (Ferro Corporation, Cleveland, OH) lead borosilicate glass used as a binding matrix. The analysis of such particle sizes is especially important as the films are fired at about 900°C. This is sufficient to melt the glass; however, minimal sintering occurs between the actual PZT particles.

IV. Initial Powder Percentages

To determine initial values for the ratios of ball and attritor3 milled powders, an ideal analysis of the particle distribution was conducted. It was first necessary to simplify the mathematical model for the PZT layer structure to a two-dimensional (2-D) representation of the particle distribution. A ball milled particle is the central particle in the lattice and the interstice between these are filled with smaller attritor milled PZT particles, shown in Fig. 2. This

TABLE II
EXPERIMENTAL POWDER COMBINATION WEIGHT PERCENTAGES.

Paste	Attritor3 percentage weight	Ball percentage weight	CF7575 percentage weight
8:1	10%	80%	10%
4:1	18%	72%	10%
2.6:1	25%	65%	10%
2:1	30%	60%	10%

model assumes all the glass will melt in the processing and form a perfect bonding matrix surrounding the particles. In this study it was decided to use a fixed percentage of glass, 10% by weight, because this was previously found to optimize the measured piezoelectric properties of the film [9]. The particle sizes available had an average diameter of 2 μm and 1 μm for the ball and attritor milled PZT, respectively. Using this basic model and the average particle size, four powder ratios were calculated to provide initial experimental results. The experimental values are shown in Table II.

V. PROCESSING

Using the values in Table II, the batches were mixed in the University of Southampton Class 100 clean room using a triple roll mill, then printed using a DEK 1200 printer (DEK International Ltd., Poole, Dorset, UK) on an alumina substrate with ESL9633B (ESL Europe, Reading, UK) silver/palladium electrodes. The PZT layer consisted of two prints on an underlying AgPd electrode. The first layer is dried using a DEK 1209 IR drier, then a second layer is printed directly on top and dried. Both PZT layers were then co-fired on a thick-film belt furnace with a peak temperature of 890°C. Once the PZT layer was fired, a single silver/palladium top electrode layer was added using the same process. This printing and firing sequence helps to reduce the effects of silver migration and reduces the risk of a short circuit between the electrodes. Two substrates per batch were printed, with one column from each substrate removed in the clean room for observation using a scanning electron microscope (SEM).

Once fired, it is necessary to induce piezoelectric properties into the devices using the poling process. The substrates were placed on a hot plate at 150°C with an electric field applied for 30 minutes, the substrate then was cooled to room temperature before the field was removed. Due to an increased thickness of the 4:1 samples, the maximum achievable electric poling field with our equipment was 3 MV/m. To maintain consistency, the electric field applied to the other samples was reduced to this value.

In addition to this, one substrate from each of the 4:1 and 8:1 pastes was printed and fired as normal but then an ESL-110S (ESL Europe) silver polymer top electrode was printed instead of the silver/palladium. This silver polymer layer was cured for an hour at 200°C using the DEK 1209 IR drier. The polymer curing cycle negates the requirement for an additional high-temperature firing cycle, which can lead to additional lead loss from the PZT and an associated drop in piezoelectric performance.

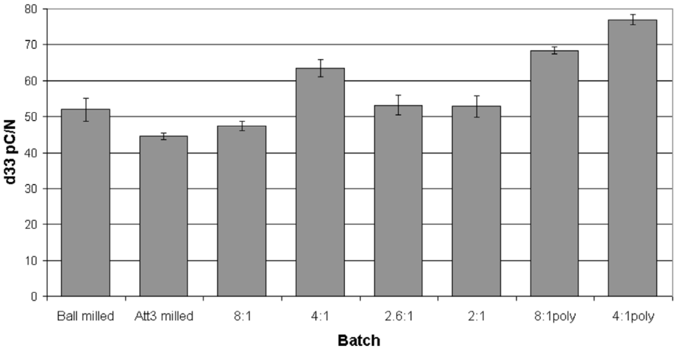

Fig. 3. Average d_{33} results for powder combination experiments.

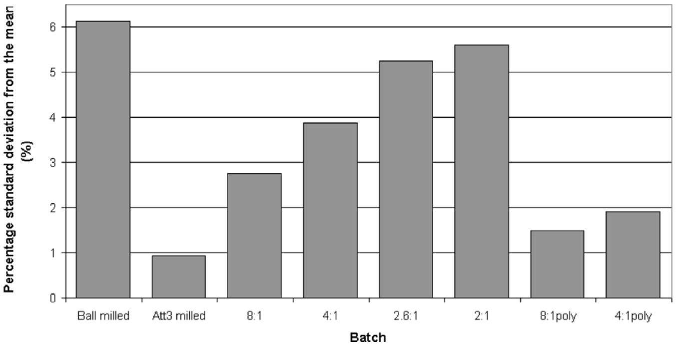

Fig. 4. Percentage standard deviation for each paste formulation.

VI. EXPERIMENTAL RESULTS

After poling, the d_{33} coefficient of each device was measured using a Take-Control PM35 (Take Control, Birmingham, UK) piezometer. Five readings were taken on each device and an average recorded, shown in Fig. 3.

The films with a polymer top electrode are denoted 8:1poly and 4:1poly. Also included for comparative purposes are the average d_{33} results obtained for the 10% ball milled and 5% attritor3 milled films from previous work.

In addition to the d_{33} results, Fig. 4 shows the percentage standard deviation associated with each paste batch to demonstrate the consistency of each batch. Again, the results from previous work for 10% ball and 5% attritor3 films have been included.

Fig. 5 shows an SEM micrograph of the 4:1 ball to attritor PZT layer. It appears that the level of sintering between the PZT particles is low, but that the glass bonding matrix and the combination of powders has produced a relatively dense film with reduced interstices.

VII. PROCESS OPTIMIZATION

Following the results of the powder combination study, the thick-film process parameters were investigated to further improve piezoelectric behavior of the optimum blend.

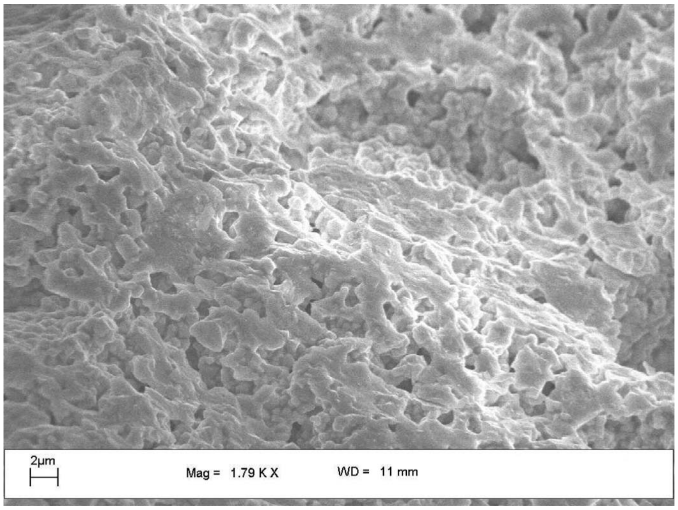

Fig. 5. SEM micrograph cross section of 4:1 ball to attritor PZT layer.

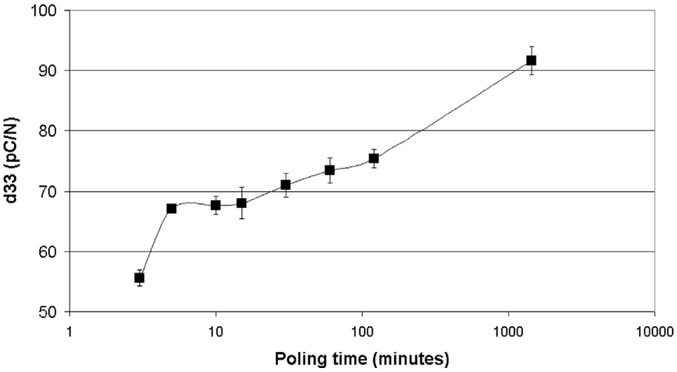

Fig. 6. Poling time study, 250 V applied at 150°C.

Two main process areas were investigated, the poling parameters and the furnace-firing profile.

A. Poling Process Study

The three main parameters of the poling process are the electric poling field, temperature, and time. Maximum piezoelectric properties are obtained with the highest possible poling voltage, temperature, and the longest poling time. Practical considerations such as dielectric breakdown, temperature damage of electrical connections, and throughput place limitations on what can be achieved. This investigation aimed to identify the important parameters and maximize their influence on the piezoelectric behavior.

The parameters used in our initial investigation were 4 MV/m at 150°C for 30 minutes [9]. The first stage investigated poling times. A poling field of 4 MV/m was applied to the substrate at 150°C for poling times between 3 minutes and 24 hours. The results in Fig. 6 show that the poling time increases the d_{33} coefficient linearly in the range investigated. If this poling process was continued for longer time periods, the increase would prove to be exponential as a maximum polarization is achieved [10]. In addition, between 5 and 30 minutes there is only ~6% increase in d_{33}. A typical industrial process uses a poling time of 3 minutes. A poling time of 5 minutes maintains sufficient piezoelectric properties while achieving a realistic throughput suitable for industrial applications. This poling time was used in the remainder of the investigation.

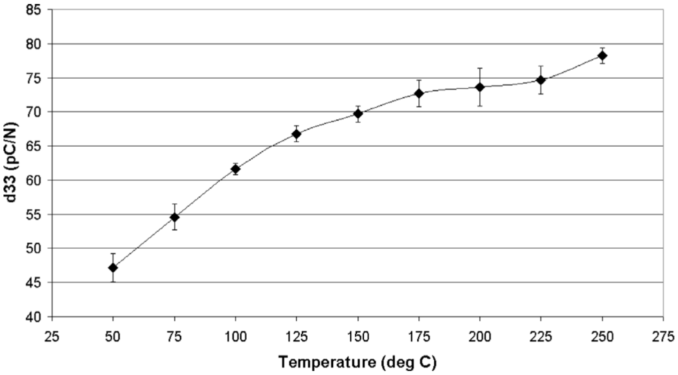

Fig. 7. Poling temperature study, 250 V applied for 5 minutes.

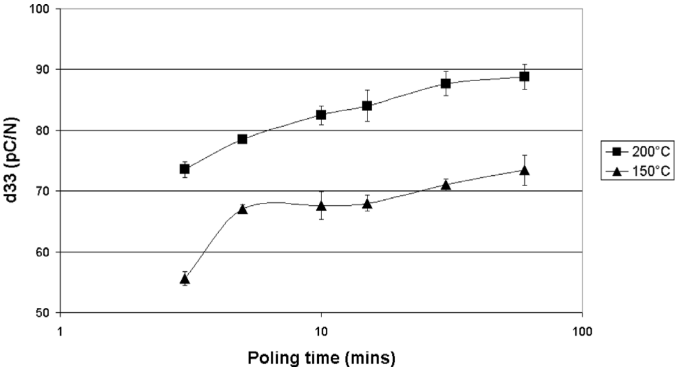

Fig. 8. Optimum combination of poling time and temperature.

Next, a study of the poling temperature was conducted. The poling field was maintained at 4 MV/m for 5 minutes, and temperatures from 50°C to 250°C were evaluated. Fig. 7 shows the increase in temperature provides an exponential increase in d_{33}. A poling temperature of 200°C was found to yield a d_{33} of 74 pC/N. This represents a ~6% improvement over the 70 pC/N obtained at 150°C and negates the ~6% drop that occurs when reducing poling time from 30 minutes to 5 minutes. Beyond 200°C, difficulties were encountered with the electrical connections as the solder melted and the polymer deteriorated. A temperature of 200°C was identified as the optimum poling temperature to be used with our current process.

The investigation of poling time was repeated with a temperature of 200°C. Poling times from 3 minutes to a practical limit of 1 hour were evaluated. This produced the results shown in Fig. 8. Fig. 8 further confirms the benefit of increasing the poling temperature. Increasing the poling time produces a higher measured d_{33} value, but this is not always practical with an industrial process. It was found that reducing the poling time to 5 minutes to

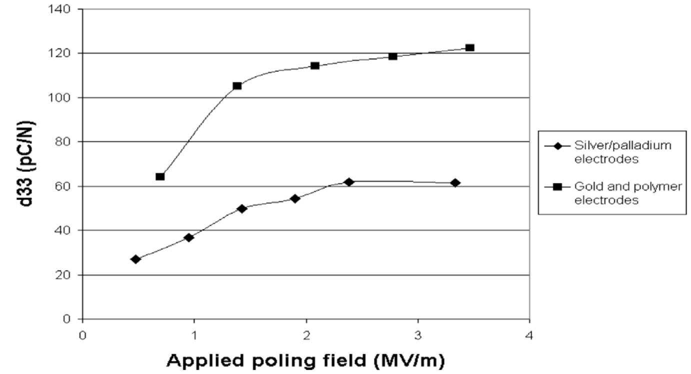

Fig. 9. Poling field study, field applied at 200°C for 5 minutes.

accelerate the poling process while maintaining the higher poling temperature produces a d_{33} of 78 pC/N. However, if sufficient time is available, a poling time of 30 minutes produces a d_{33} of 88 pC/N.

To complete the study, the effects of increasing the poling field was investigated. Previous studies by Dargie *et al.* [11] showed that improved piezoelectric behavior was achieved when the poling voltages were increased. However, the maximum achievable poling field is limited by the electrical breakdown of the dielectric material. Due to variations in device thickness for different processes, it was important to show that any increase in d_{33} was a result of the formulation and process parameters rather than increased poling field strength. Voltages between 50 V and 250 V were applied for 5 minutes at 200°C to two devices. One device was from the original 4:1 batch with an AgPd bottom electrode and one from the latest 4:1 batch with ESL 8836 gold bottom electrodes, both devices had polymer top electrodes. Fig. 9 shows that an increased poling field produces an exponential rise in d_{33}. This exponential increase is a result of improved alignment of the electric dipoles in the material. The polarization level increases until a maximum polarization is reached, and all the dipoles are aligned with the applied field. These results show that the increase in d_{33} between the different devices is not due to a change in applied field and show that 250 V is a sufficient poling voltage in this case. The poling voltage of 250 V equates to an applied poling field of 4 MV/m.

B. Furnace Firing Profile Study

The furnace firing profile affects the piezoelectric properties because of the change in the stoichiometry of the film material and the degree of particle sintering that occurs during the firing process. The change in stoichiometry is a result of the increased lead evaporation with rising firing temperature.

Bulk PZT-5H piezoceramics are typically fired between 1000°C and 1700°C, and the stoichiometry of the powder is balanced to reflect this. A firing profile with a peak of 1000°C (denoted rnt1000), with the same stage increments as the current Dupont60 furnace profile (both shown in Table III) was investigated.

TABLE III

Furnace Firing Profiles for Dupont60 and rnt1000.

Furnace Stage	Furnace firing profile name: Dupont60	rnt1000
1	350°C	393°C
2	600°C	674°C
3	885°C	994°C
4	883°C	992°C
5	890°C	1000°C
6	870°C	978°C

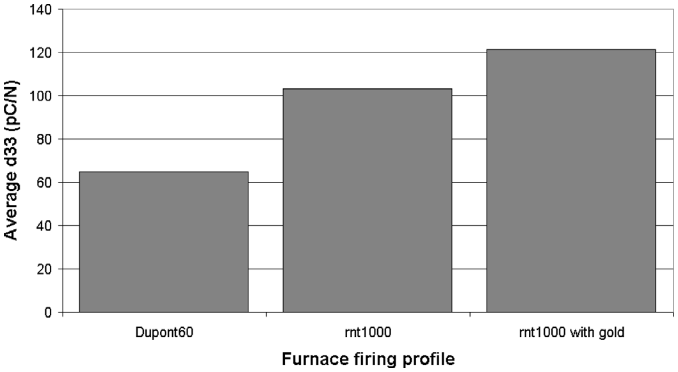

Fig. 10. Piezoelectric responses for alternative furnace profiles.

To ensure any change in d_{33} was due to the firing profile, an ESL 1110-S silver polymer top electrode again was used. This removed the requirement for the PZT layer to endure further firing cycles. In addition to these, one substrate was printed with ESL 8836 gold cermet bottom electrode to observe the effects of removing any silver migration into the film during firing; this was only fired using the rnt1000 profile. These devices were poled at 4 MV/m for 5 minutes at 200°C.

Fig. 10 shows the average d_{33} coefficients measured from the six devices fired with each profile. The results show that the higher firing temperature produces an increase from 65 pC/n to 103 pC/N. In addition, the introduction of a gold bottom electrode, thus removing all silver from the firing process, provides a further increase to ~121 pC/N. Poling a gold device with an applied field of 4 MV/m for 30 minutes produces a d_{33} of 131 pC/N.

VIII. Discussion of Results

The results presented in this paper show that blending the ball milled powder with the attritor3 milled powder does produce an increase in the piezoelectric activity of the film. Fig. 3 suggests that the number of smaller particles added to the paste has a saturating effect beyond the peak ratio of 4:1. This saturation is believed to be a result of the interstice gaps within the matrix already being filled and a reduced dominance of the ball milled particle. The original 10% ball milled PZT paste demonstrated a d_{33} of 52 pC/N. The highest measured d_{33} value, 63.5 pC/N, was obtained with the 4:1 by weight combination ball to attritor3 milled powder. This was further increased to 77 pC/N when a polymer top electrode was used. The polymer top electrode removes the requirement of additional firing of the piezoelectric film. The additional firing cycles increase the volume of lead lost due to evaporation, and hence a change in the stoichiometric balance of the PZT film and a change in the piezoelectric behavior. A previous study by Torah *et al.* [12] has shown the substrate has a significant effect on reducing the measured d_{33} value of a thick-film piezoelectric layer. It is believed that the reduced Poisson's ratio of the polymer electrode reduces the clamping effect on the piezoelectric properties of the film. In addition, it is proposed that the reduced clamping of the polymer allows for a reduced amount of dipole realignment when the poling field is removed, thus allowing an increased level of polarization in the film for the same poling field.

These developments represent an increase of approximately 20% and 30%, respectively. The percentage standard deviation of the 4:1 devices was 4%. The attritor3 devices from the previous study exhibited a 1% deviation. However, this is still an improvement compared to the original ball milled devices that exhibited a standard deviation of 6% in measured d_{33} value between devices.

Having identified the optimum paste formulation, the investigation of poling time, temperature, and firing profile yielded further improvements in piezoelectric behavior. Combining the poling time of 5 minutes and poling temperature of 200°C generates an increase in d_{33} of 10% to 78 pC/N compared with the original poling process. Increasing poling time to 30 minutes produces a d_{33} of 88 pC/N, an improvement of 20%. The increased poling temperature produces an increase in piezoelectric activity because the additional softening of the PZT material facilitates the permanent dimensional change that occurs with polarization. Hence, improved dipole alignment in the direction of the poling field is possible. The increase in poling time allows alignment of a greater proportion of dipoles [10].

Increasing the peak firing temperature produces an increase in measured d_{33} value from 65 pC/N to 103 pC/N obtained using the Dupont60 and rnt1000 firing profiles, respectively. This increase in measured d_{33} is because the rnt1000 profile is closer to the industrial process used for bulk PZT-5H powder. Therefore, the final stoichiometric balance of the powder and the level of sintering in the film is closer to that of the optimal bulk equivalent. The raised firing temperature increases both the sintering between the PZT particles themselves and the binding matrix of the lead borosilicate. This improves the density and mechanical quality of the film that provides an increase in the measured d_{33}. This increase could be attributed, in part, to an improved electromechanical coupling coefficient. The use of a higher firing temperature is well suited for alumina substrates but will reduce compatibility with silicon substrates. The higher temperature will exacerbate the reaction that occurs between the PZT and silicon first reported by Beeby *et al.* [4]. This problem could be reduced with the use of a suitable barrier layer on the silicon or the use of an additional sintering stage on the PZT powder before it is mixed into a screen-printable paste.

The introduction of a gold bottom electrode produced a d_{33} of 121 pC/N and an increased poling time of 30 minutes further improved this to 131 pC/N. This is the maximum d_{33} we have ever measured on the Take Control PM35 piezometer. This equipment has been found to produce consistent and repeatable results compared with other measurement techniques [5]. From our experience, the measurement of d_{33} of a film deposited on a substrate is not straightforward. Any slight bending of the substrate, for instance, will artificially amplify the measured d_{33}. Therefore, care was taken to adjust the PM35 to apply a minimum amount of clamping force on the device being measured to reduce the possibility of introducing additional d_{31} effects. The introduction of a gold bottom layer negates the effects of silver migration during the PZT firing cycle. The migration of silver into the PZT layer reduces the level of electric field that can be applied before breakdown occurs. Therefore, the amount of dipole alignment, and consequently the piezoelectric properties of the film, is reduced. The addition of a polymer top electrode removes subsequent firing cycles, thereby maintaining the stoichiometry of the film at a preferred level.

It should be noted that all measurements of d_{33} were taken within 24 hours of poling. The piezoelectric behavior of ball milled samples previously was shown to decay by approximately 25% over 3 months [13]. The optimum 4:1 PZT sample with the gold bottom electrode, silver polymer top electrode that was poled for 5 minutes deteriorated from 121 pC/N to 110 pC/N, 10%, over a 3-month period.

IX. Conclusions

This investigation has shown that the combination of 4:1 by weight of ball milled to attritor milled powder (i.e., 18% attritor3, 72% ball, and 10% CF7575 powders) produces the highest d_{33} coefficient with an average measured value of 63.5 pC/N. The results indicate that the combination of small and large particles does increase the d_{33} of the film and justifies the investigation. It also has shown that the consistency of the results can be increased in comparison to the ball milled devices, although they are not as consistent as the attritor3 devices.

Further improvements in the piezoelectric response of the material were achieved by increasing the poling time and temperature. An optimum poling temperature of 200°C and a poling time of 30 minutes produced a d_{33} of 88 pC/N. The increased firing temperature of 1000°C peak gives a further improvement of 131 pC/N when used in combination with a gold bottom electrode and polymer top electrode.

The improvement of d_{33} from the original ball milled device with a d_{33} of 52 pC/N, to a maximum achieved d_{33} of 131 pC/N represents a significant improvement of 152% in the piezoelectric activity of a PZT-5H thick-film device.

Further work will consider the PZT formulation as part of a multilayer structure investigation to combine the piezoelectric effects of a number of thick-film layers to obtain further increases in d_{33}. The effects of cofiring the devices with a gold electrode also will be investigated so that a more robust top electrode material can be used without the need for the PZT layer to experience further firing cycles.

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He has since been awarded a prestigious Engineering and Physical Sciences Reseach Council (EPSRC) Advanced Research fellowship to continue his research into combining thick-film printed active materials with micromachined devices. His other research interests include energy harvesting for remote wireless sensor networks, and he is the principal investigator at Southampton University on an EU funded Specific Targeted Research Projects (STREP) project entitled, "Vibration Energy Scavenging (VIBES)." His other research interests include smart materials, novel thick-film material development, micromachining process development, and human biometric systems.

He has over 85 publications in the field including in learned journals and presented at conferences and colloquia. He is co-author of a forthcoming book entitled, *MEMS Mechanical Sensors*, published by Artech House and is a Chartered Engineer and Chartered Physicist.

Neil M. White holds a personal chair in the School of Electronics and Computer Science, University of Southampton, Southampton, Hampshire, UK. He has been active in sensor development since 1985. In 1988 he was awarded a Ph.D. degree from the University of Southampton.

He has considerable experience in the design and fabrication of a wide variety of sensors, formulation of novel thick-film sensing materials, and intelligent sensor systems. In 1994 he co-authored a book entitled *Intelligent Sensor Systems*, and he is the co-author of *MEMS Mechanical Sensors*, published by Artech House in 2004.

Dr. White was the Chairman of the Instrument Science and Technology (ISAT) group of the Institute of Physics from 1997–1999. He has over 120 publications in the area of instrumentation and advanced sensor technology. His professional qualifications include Chartered Engineer, Fellow of the IEE, Fellow of the IOP, Chartered Physicist, and Senior Member of the IEEE.