Temperature effects on the mechanical behaviour of PZT 95/5

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Abstract. This research is to develop a better understanding of the piezoelectric ceramic lead zirconate titanate (PZT) 95/5 with varying temperatures, porosities and strain rates. Here, unpoled PZT samples of two different porosities were subjected to a range of compression rates, using quasi-static loading equipment, drop-weight towers and Split Hopkinson Pressure Bars (SHPBs). Varying temperatures were achieved using purpose-made environmental chambers. The resulting stress-strain relationships are compared. The samples were square tiles, 7.5 x 7.5 mm and 3 mm thickness. The density of the standard PZT used here was 7.75 g cm\(^{-3}\) (henceforth described as PZT), whilst the density of the higher porosity PZT was 7.38 g cm\(^{-3}\) (henceforth described as PPZT). This research is part of a wider study.

1. Introduction
Induced strain in a poled piezoelectric material results in the generation of electric charge. This can be the result of applied pressure. The amount of charge generated depends on both the applied strain-rate and temperature. Application of a mechanical stress to a piezo-crystal alters its crystal structure, and the ions move with respect to each other, forming a dipole. To develop charge, the crystal structure must be non-centrosymmetric; net polarisation results in the occurrence of the piezoelectric effect.

Many ceramics have some degree of porosity. PZT 95/5 (95% lead zirconate, 5% lead titanate, with no added dopants) is a piezoceramic that is both pyroelectric (affected by temperature), and ferroelectric (having spontaneous polarisation). Bulk PZT can be made porous, by the controlled addition of a poreformer [1].

Previous experiments on piezoelectric ceramics have shown that many of their mechanical properties, such as elastic moduli, strength and toughness, decrease with increasing porosity [2]. Fracture energy appears to have a more gradual decrease with porosity than the fracture toughness [3].

Pores can act both as micro-crack initiation sites and as channels for propagating these micro-cracks. Many pores link together to form the path of least resistance for the crack. The presence of the pores strongly affects the compressive strength of a ceramic material [4]. Parker [5] found that more porous materials exhibited lower fracture toughness at low strain rates. It has been seen for PZT 95/5 doped with 2% niobium (Pb\(_{0.99}\)Nb\(_{0.02}\)(Zr\(_{0.95}\)Ti\(_{0.05}\))\(_{0.98}\)O\(_3\)) that pore collapse begins somewhere between 400 - 800 MPa under strain rates between \(10^{-4}\) - \(10^{-2}\) s\(^{-1}\) [6]. It also appears that both the mechanical and electrical properties of PZT 95/5 are sensitive to the level of porosity, but insensitive to pore morphology [7].
As the temperature of a ceramic decreases towards absolute zero, so does the gradient of a curve of elastic constant as a function of temperature. Conversely, as the temperature increases toward the Debye temperature (the highest temperature that can be achieved due to a single normal vibration), the slope approaches a constant for many ceramics. This constant can be used to estimate a number of mechanical properties of the ceramic, including the value of elastic moduli [8]. It has been seen that the elastic constants of many single piezocrystals generally decrease slowly as the temperature is further increased [9].

A general observation for ceramics between room temperature and the onset of grain boundary softening is that the Young’s modulus and the shear modulus decrease about 1% per hundred kelvin increase [3]. The softening temperature is considerably below the melting temperature of typical crystalline ceramics [10].

Finally, it has been observed that the effective transverse piezoelectric coefficient increases, with a smaller temperature dependence for piezofilms compared to bulk piezoceramics [11].

2. Low strain rate effects
A 250 kN universal testing machine Instron (model 5584) in the Mechanical Engineering Department at Imperial College London was used. The apparatus was a servo-hydraulic machine, and can be used in both compression and tension.

Under compression, the machine was designed and setup to load uniformly and compress a specimen, until a pre-defined limit. Due to the brittle nature of the PZT, these tests were stopped when the material fractured. A compression rate of 0.004 mm s$^{-1}$ was adopted.

Both elevated and sub-zero temperatures were achieved using a thermostatically-controlled environmental chamber. Liquid nitrogen was used to achieve lower temperatures, and the chamber had an in-built forced convection heating system for higher temperatures. It was necessary to run preliminary experiments to establish the time taken for the sample to reach thermal equilibrium; for these samples 20 minutes was sufficient.

Experiments were carried out both at ambient temperature (+20 °C) and humidity, and at an elevated temperature of +74 °C.

As can be seen from figure 1, the standard, non-porous PZT sample was able to withstand a larger stress than the porous PZT. The difference in the yield stress was significantly larger at room temperature than at the higher temperature. In both cases a higher strain to failure was observed at the elevated temperature, see table 1. Whilst the standard, non-porous PZT shows complete failure at an engineering strain of ~0.32, some initial cracking was observed from an engineering strain of ~0.23. Some cracking was heard from this point, but the standard, non-porous PZT was still able to withstand further stress until complete failure of the specimen at ~550 MPa. What is interesting to note is that the gradient of the engineering stress-strain slopes of both the standard and the porous PZT are almost identical.

3. Drop-weight testing
Drop-weight tests yield strain rates of the order of $10^2$ s$^{-1}$. Here, the spring-loaded Instron (Dynatup) 9250HV machine in the Civil Engineering Department at Imperial College London
Table 1. Numerical results for quasi-static experiments on PZT.

| Sample | Density [g cm$^{-3}$] | Temperature [$^\circ$C] | Compression Rate [mm s$^{-1}$] | Strain Rate [s$^{-1}$] | Strain to Failure | Stress to Failure [MPa] |
|--------|------------------------|--------------------------|-------------------------------|------------------------|---------------------|------------------------|
| PZT    | 7.75                   | 20                       | 0.004                         | 0.0013                 | 0.169               | 640                    |
| PPZT   | 7.38                   | 20                       | 0.004                         | 0.0013                 | 0.139               | 440                    |
| PZT    | 7.75                   | 74                       | 0.004                         | 0.0013                 | 0.321               | 562                    |
| PPZT   | 7.38                   | 74                       | 0.004                         | 0.0013                 | 0.280               | 535                    |

Figure 2. Drop-weight sequence showing the fracture of a porous PZT 95/5 sample; impact mass 7 kg, velocity 5 m s$^{-1}$.

was used. It consisted of a (variable) mass holder, which slid between two external guiding rods, and was fitted with an accelerometer. Arresting brakes were mounted at the base to prevent the mass from impacting the specimen more than once [12].

An impactor mass of 7 kg, and an impact velocity 5 m s$^{-1}$, was adopted. A high-speed camera, a Phantom V12.1, was used to capture the deformation of the specimens.

Both the standard and the porous PZT samples fractured along the axis of impact. Figure 2 shows the drop-weight sequence of a porous PZT 95/5 sample.

From figure 3 it can be seen that, at room temperature, the differences in the yield stress and strain to failure observed between the standard and the porous PZT samples was lower than seen in the results from quasi-static experiments. The strain to failure was itself lower in the drop-weight experiments, but higher dynamic stresses were supported by both PZT samples at these higher strain rates.

This stress-strain curve is not typical for brittle materials such as PZT, where we expect to see a rise and then a sharp decrease in the stress experienced. Future research will investigate the decrease in stress at higher strains, and look to match the mechanical response of the PZT to the known theory.
**Figure 4.** Schematic of a Split Hopkinson Pressure Bar, with the proposed future specimen setup including both electrodes and insulation.

4. **Split Hopkinson pressure bars**

A SHPB (see figure 4) was used to reach strain rates of the order of $10^3 \text{ s}^{-1}$ [13]. The specimen was placed between the input and output bars. A small gas gun was used to launch the striker bar against the input bar. Semiconductor strain gauges in the centre of the input and output bars [14] measured the elastic waves which propagated, and recorded the incident, transmitted and reflected pulses [15]. The resistance change from the strain gauges was recorded and converted to produce stress-strain values [16].

The SHPB system at the Cavendish Laboratory, Cambridge, was used to dynamically load the samples. The bars used were made of Inconel 718 and tungsten. Their properties are listed in table 2. While it is usual to use bars with similar impedances, often accomplished by using the same material for the striker, input and output bars, the lengths of the bars can vary. The tungsten striker bar was 15.1 cm long and the Inconel 718 bar was 18.2 cm long.

**Table 2.** Properties of the Inconel 718 and tungsten input and output bars.

| Property             | Inconel 718 | Tungsten       |
|----------------------|-------------|----------------|
| Density $\rho_b$ [kg m$^{-3}$] | 8269        | 16900          |
| Wave Speed $c_0$ [m s$^{-1}$]     | 4980        | 4406           |
| Impedance $Z$ [kg m$^{-2}$ s$^{-1}$] | 41.3x10$^6$ | 75.3x10$^6$   |
| Elastic Modulus $E_b$ [GPa]       | 205         | 411            |
| Length $L_b$ [mm]            | 500         | 500            |
| Diameter $D_b$ [mm]          | 12.7        | 12.7           |

A number of preliminary experiments were carried out, all at ambient temperature and humidity. The firing pressure used was 3 bar and, due to the different masses of the bars, the impact velocities for the Inconel 718 and tungsten bars were $\sim 8.7 \text{ m s}^{-1}$ and $\sim 6.8 \text{ m s}^{-1}$ respectively.
Figure 5 shows the engineering stress versus engineering strain data obtained for the porous and non-porous PZT 95/5. The porous PZT is denoted by ‘PPZT’, whilst ‘W’ represents data obtained using the tungsten bars, and ‘Incl’ the Inconel 718 bars.

From this graph, it can be seen that higher stresses were developed using the tungsten bars. The porous PZT experienced a lower strain to failure than the standard, more dense, PZT. However, no significant differences between the peak stresses generated in either the standard PZT or the more porous PZT were discernible for the two bar types.

Table 3 shows the quantitative difference between strain rates at room temperature. The failure stress is quite variable, sample to sample, as often found with brittle materials.

| Sample | Density [g cm\(^{-3}\)] | Bar Material | Bar Velocity [m s\(^{-1}\)] | Strain Rate [s\(^{-1}\)] | Strain to Failure | Stress to Failure [MPa] |
|--------|-------------------------|--------------|-----------------------------|---------------------------|-----------------|-----------------------|
| PZT    | 7.75                    | Inconel 718  | 8.697                       | 1600                      | 0.015           | 1550                  |
| PPZT   | 7.38                    | Inconel 718  | 8.600                       | 1080                      | 0.011           | 1550                  |
| PZT    | 7.75                    | Tungsten     | 6.822                       | 900                       | 0.012           | 2520                  |
| PPZT   | 7.38                    | Tungsten     | 6.884                       | 800                       | 0.009           | 2540                  |

5. Conclusions and future research
We present here preliminary results from low to mid-strain rate experiments conducted on PZT with varying porosities and temperatures. Overall, the mechanical behaviour of PZT appears to be that of a typical ceramic, having significant temperature and strain rate dependence. However, since failure in ceramics varies widely, and is often dominated by initiation, a great deal of further work still needs to be carried out in order to have higher statistical data and therefore be able to draw better conclusions from this research.

Future samples will be machined into circular discs in order to minimise the effects of stress concentration in the corners of square-tile samples. These samples will also be poled and the electrical properties measured. Future research will be carried out on the poled PZT material at strain rates between \(10^{-4}\) - \(10^{4}\) s\(^{-1}\), at temperatures between −80 - +80°C, and with additional diagnostics to measure the piezoelectric charge output.

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