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Gas Gun Impact Testing of PZT 95/5 Part I: Unpoled State

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Gas Gun Impact Testing of PZT 95/5

Part I: Unpoled State

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Abstract

In the present study, 10 impact tests were conducted on unpoled PZT 95/5, with 9% porosity and 2 at% Nb doping. These tests were instrumented to obtain time-resolved loading, unloading and spall signatures. As well, PVDF gauges allowed shock timing to be established explicitly. The ferroelectric/antiferroelectric phases transition was manifested as a ramp to 0.4 GPa. The onset of crushup produced the most visible signature: a clear wave separation at 2.2 GPa followed by a highly dispersive wave. The end states also reflected crushup, and are consistent with earlier data and with related poled experiments. A spall strength value of 0.17 GPa was measured for a shock stress of 0.5 GPa; this decreased to a very small value (no visible pullback signature) for a shock strength of 1.85 GPa.

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Gas Gun Impact Testing of PZT 95/5

Part I: Unpoled State

1.0 Introduction

A knowledge of the electromechanical properties of key ferroelectric materials is important for various pulsed-power applications. Over the past thirty years various studies have been performed to understand the behavior of such materials as barium titanate (BT), lead niobate and lead zirconate titanate (PZT) under shock loading. Early studies such as Linde [1967] and Doran [1968] demonstrated the capability of these materials to generate a large pulse of current and voltage if poled, then abruptly depoled by shock loading. More recent studies [Mock and Holt, 1979; Dick and Vorthman, 1978; Lysne, 1975, 1977; Lysne and Bartel, 1975] have provided considerable experimental and theoretical detail for electromechanical phenomena present in the shock loading of these materials. Most of the recent attention has been given to PZT materials, especially PZT 95/5 (Zr:Ti ratio of 95:5) [Lysne and Percival, 1975; Dick and Vorthman, 1978, Chhabildas, 1984; Chhabildas *et al.*, 1986]. In this particular material, the ferroelectric/antiferroelectric (FE/AFE) boundary is quite close to the room temperature/pressure state, as shown in Figure 1; this attribute facilitates depoling by relatively small amplitude stress waves.

Figure 1.1. (Left) Pressure/temperature phase diagram of PZT 95/5, adapted from Fritz and Keck [1978]. (Right) Temperature composition phase diagram of PZT, adapted from Spears [1978].

For unpoled samples, shock wave properties of interest include phase transition kinetics (between the ferroelectric and antiferroelectric phases), pore crushing, and Hugoniot states. Doran [1968], in a study of PZT of initial density $7.67 - 7.89 \text{ gm/cm}^3$ utilizing explosively-generated waves, noticed two cusps in the Hugoniot. He attributed a pronounced cusp at 4 GPa to yielding, and a weaker cusp at 0.2 GPa to the FE/AFE transition. The starting material was nearly full-density in these experiments. By contrast, a later transmitted-wave study by Chhabildas [1984] with porous PZT 95/5 (density 7.3 gm/cm^3) showed a strong wave separation at 2.8 GPa and a very weak wave separation at 0.2 - 0.5 GPa. The 2.8 GPa break probably corresponded to the onset of pore crushing, and the 0.2 - 0.5 GPa break, to the FE/AFE transition.

Shock recovery experiments [Chhabildas *et al.*, 1986] show reversibility of the FE/AFE transition and clarify the role of pore crushing in porous PZT 95/5. These results appear to corroborate the conclusions reached in the wave profile measurements.

Quasi-static compression studies of this material suggest that the FE/AFE transition may be influenced by resolved shear stress in addition to pressure and temperature (see Figure 2), causing the transition under shock loading to occur over a range of stresses rather than a single stress.

Figure 1.2. Role of resolved shear stress on FE/AFE transition [Zeuch and Montgomery, unpublished]

The present study, driven by increasingly stringent model certification requirements, is intended as a set of experiments in both poled and unpoled regimes on comparable samples of PZT 95/5 (see next section for physical properties). This material has been subject to a uniaxial strain environment by planar impact loading. Stresses applied ranged from 0.5 to 5 GPa, with several impact configurations utilized to provide loading, release and spall properties.

The present report describes experiments performed on samples that initially contain randomly oriented ferroelectric domains; i.e. unpoled samples. These are compared with the mechanical response of electrically poled samples, where the poling is either normal to the direction of shock propagation (normally poled) or parallel to it (axially poled), and several electrical circuits are employed to vary electrical fields present during shock depoling. The experiments on poled samples are discussed in more detail in separate reports.

2.0 Sample Descriptions and Physical Properties

The samples employed were lead zirconate titanate with a Zr:Ti ratio of 95:5 (PZT 95/5). The initial porosity is estimated as 9% based on X-ray measurements [Chhabildas et al., 1986] which place the grain density as 8.028 gm/cm³. Grain size is estimated as less than 50 microns. Pores are approximately spherical with typical diameters of 100 microns. These pores, generated during sintering by sacrificial polyethylene pore formers, are occasionally observed to agglomerate into highly porous regions or single pores up to 500 microns in diameter.

Ultrasonic (transducer) measurements were performed on all samples. The results are summarized in Table 2.1.

These samples are very similar to those employed in the Chhabildas [1984] study, although not identical. Since the present study does not include pressure/shear loading, in contrast with the Chhabildas [1984] study, the earlier study presents useful complimentary data.

Table 2.1 Elastic properties of PZT 95/5 samples.

	Present Study	1984 Study
Mean Longitudinal Velocity (km/s)	4.1631	4.1836
Standard Deviation (km/s)	0.0060	0.0085
Mean Shear Velocity (km/s)	2.5178	2.5297
Standard Deviation (km/s)	0.0032	0.0082
Initial Density (gm/cm ³)	7.30	7.30
<i>(Following are based on mean values above)</i>		
Bulk Velocity (km/s)	3.9194	3.9382
Young's Modulus (GPa)	112.1	113.2
Shear Modulus (GPa)	46.3	46.7
Bulk Modulus (GPa)	64.8	65.5
Poisson's Ratio	0.212	0.212
Acoustic impedance $\rho_0 C_l = (\rho_0(K+4/3 G))^{1/2}$	30.4	30.5

3.0 Experiments

The general design of the experiments is shown in Figure 3.1. A compressed gas gun (2.0" bore) is used to accelerate a projectile tipped with fused silica or sapphire to velocities of 0.1 to 0.7 km/s. This projectile then impacts a sample of PZT 95/5 backed by a window of fused silica or Z-cut sapphire. A VISAR interferometer is used to monitor the velocity history of the window surface adjacent to the PZT sample (note the intervening PVDF gauge in tests 2677 and 2690). PVDF gauges provide time-of-arrival information for most shots. Specific experiment parameters are summarized in Table 3.1.

Figure 3.1. Experiment configuration for PZT impact tests. PVDF gauges were not present on all shots. The impactor and window materials varied (see Table 3.1).

Table 3.1. Summary of unpoled experiments

Expt #	FE Thick.	Impactor Thickness ^a	Impact Velocity	Window	Approx. Stress	Anal. '84 Expt.
2677	3.970 mm	12.654 mm	0.494 km/s	Fused Silica	3.65 GPa	FER8
2678	1.994 mm	3.184 mm	0.7016 km/s	Fused Silica	5.05 GPa	FER5
2679	1.994 mm	3.221 mm	0.4496 km/s	Fused Silica	3.45 GPa	FER4
2680	3.988 mm	4.040 mm	0.243 km/s	none	1.87 GPa	FER6
2682	3.988 mm	3.221 mm	0.0659 km/s	none	0.50 GPa	FER7
2683	1.994 mm	3.199 mm	0.5084 km/s	Sapphire	3.84 GPa	FER3
2690	3.972 mm	6.34 mm	0.623 km/s	Sapphire	4.92 GPa	-----
2691	1.99 mm	3.2 mm	0.1095 km/s	Sapphire	0.94 GPa	FER1
2708	3.986 mm	6.342 mm	0.110 km/s	Sapphire	0.96 GPa	
2719	4.003 mm	12.70 mm	0.155 km/s	Sapphire	2.3 GPa	

a. Impactors are fused silica for all tests except for 2719 (sapphire impactor)

PVDF gauges were used to monitor the impact face of the PZT for all tests in this series except for 2680 and 2682, which were spall experiments. As well, they were used to monitor the sample/window interface for tests 2677 and 2690. All of these gauges (approx. 40 microns thick) were separated from the PZT samples by 40 microns of Teflon[®] to reduce local effects of shock heterogeneities. The wave propagation time across these PVDF/Teflon combinations was estimated as 25 ns.

Due to local electrical interference from the PZT, the quantitative pressure data from the PVDF gauges was not reliable and the gauges were used as time-of-arrival indications only.

The VISAR instrumentation used included two different sensitivities for each shot (dual delay method); velocity-per-fringe sensitivities for free surfaces were 0.15127 km/s for a leg using glass etalon elements to provide the delay, and 0.03724 km/s for a leg using a telescope system (air or lens delay). In all cases the data from the air delay leg were used for the final velocity profile after comparison with the data from the etalon delay leg as a check for missed fringe jumps.

Results - Velocity Profiles The first three tests were conducted with fused silica impactors and windows to provide loading/release properties. Velocity profiles are shown in Figure 3.2. Velocity profiles from the next set of two experiments, designed to yield spall data, are shown in Figure 3.3. No windows were used for these tests. The remaining five tests all utilized sapphire windows, and are grouped according to sample thickness 4 mm vs. 2 mm) in Figures 3.4 and 3.5, respectively.

Figure 3.2. Interface velocity profiles for tests 2677 - 2679.

Figure 3.3. Free surface velocity profiles for spall tests 2680 - 2682.
The tungsten foil provided a VISAR reflecting film.

Figure 3.4. Interface velocity profiles for tests 2690, 2708 and 2719
(4 mm samples, sapphire windows)

Figure 3.5. Interface velocity profiles for tests 2691 and 2683
(2 mm samples, sapphire windows)

The velocity profiles shown in Figures 3.2, 3.4 and 3.5 were timed utilizing the PVDF gauge data, and corrected by subtracting out the transit time through the gauges and Teflon layers. The profiles shown in Figure 3.3 (no PVDF gauges) were timed by assuming that the toe (first motion) propagated at the average longitudinal sound speed $C_l = 4.163$ km/s. Generally the timing determined using the PVDF signal was within 10 ns of the timing determined using toe arrival for shots where PVDF data were acquired.

These profiles may be compared with the analogous profiles from Chhabildas [1984]. The appropriate wave profiles are juxtaposed in Figure 3.6 (fused silica windows) and 3.7 (sapphire windows). For ease of comparison transit times are normalized for 1 mm thick samples.

The loading portions of the profiles appear very similar, although FER5 and FER6 release much sooner than do 2677 - 2679 due to relatively thinner flyer plates (e.g. 1 mm fused silica flyer plate and 2 mm sample on FER5 versus 3 mm fused silica flyer plate and 2 mm sample on 2678 and 2679).

Figure 3.6. Wave profiles for unpoled samples with fused silica windows, from the present data set as well as from Chhabildas (1984). Times normalized to sample thickness of 1 mm for ready juxtaposition.

Figure 3.7. Wave profiles for unpoled samples with sapphire windows, from the present data set as well as from Chhabildas (1984). Times normalized to sample thickness of 1 mm for ready juxtaposition.

Results - Lagrangian Analysis and Impedance Match: The wave profiles for experiments with windows (sapphire or fused silica) were analyzed by a Lagrangian calculation utilizing the input and output wave profiles for the sample. The incremental form of the release is calculated using $d\sigma = \rho_0 C du$ and $d\epsilon = du/C$, where C is the Lagrangian wave velocity for the corresponding particle velocity increment/decrement du at particle velocity u . Here, the input wave profile is taken as a 10 ns wide ramp at a time determined by the PVDF gauges. The output waveform is that measured by VISAR.

Primary goals of this analysis are as follows:

- Assess loading profiles to determine features corresponding to phase transitions or yield points (slope breaks in the stress/strain curve, *i.e.* cusps)
- Assess release profiles to determine reversal of phase transition, and
- Assess Hugoniot states.

The loading profiles, analyzed in this way and plotted in stress vs. sound speed space, are shown in Figure 3.8.

Figure 3.8. Loading profiles for present tests, plotted as stress vs. sound speed.

General features observed are as follows. A ramp loading beginning with the toe loads to a stress of approximately 0.4 GPa, followed by a sharp loading to 2.2 GPa propagating at about 3.7 km/s. Further loading is effected by a much more slowly propagating (2 - 2.5 km/s), dispersed wave. This is relatively independent of window material, although the tests with fused silica windows do not show the 2.2 GPa breakoff as cleanly as do the sapphire window tests. Physically, the difference may be due to the impedance mismatch. Fused silica has a substantially lower shock impedance than does the PZT, and sends a release back into the PZT beginning with the toe arrival. By contrast, the sapphire windows have a higher impedance than the PZT, so send a reshock back into the sample.

The loading profiles represented in strain vs. wave speed space are shown in Figure 3.9. The strain associated with the FE/AFE transition is quite small, about 0.005 or less. Following an elastic loading to a strain of 0.02, the slower dispersive final loading wave produces strains of order 0.06 - 0.10. This is consistent with a crushup phenomenon.

Figure 3.9. Loading profiles for present tests, plotted as strain vs. sound speed.

The release paths may also be calculated with an explicit Lagrangian calculation, with the caveat that the forward-propagating release wave is propagating through some amount of sample which has been either partially released (fused silica windows or free surface) or reshocked (sapphire windows). The results are shown in Figure 3.10, and discussed further below.

Figure 3.10 also shows Hugoniot points and inflection points (onset of crushup or of FE./AFE transition). The Hugoniot points were calculated by the explicit Lagrangian method, while cusp points (peak stress levels and “kinks” points) were calculated by standard impedance match methods. Since the impedance match methods require the assumption of steady wave propagation, these may not be used for final loading states at stresses above ~2.2 GPa (the crushup wave does not appear to be a steady wave). On the other hand, the cusp points may be calculated readily in this manner, and the results generally fall on the trajectories calculated by the explicit Lagrangian method in stress/strain space.

Table 3.2 shows the cusp and Hugoniot points thus calculated.

For reference, analogous points and paths for poled experiments are included in Figure 3.10 and Table 3.2 as well as the points for the unpoled experiments and for the experiments described by Chhabildas [1984]. Note that the unpoled experiments appear to be less stiff than the normally poled experiments at low stresses, and comparable to the axially poled experiments.

Figure 3.10. (Top) Hugoniot (squares) and inflection points (*+) for present experiments and for related experiments, together with stress-strain loops calculated by explicit Lagrangian analyses. (Bottom) Detail of low stress/low strain region of top plot (Lagrangian loops only shown; only lower stress experiments shown).

Releases are calculated using two alternative assumptions for the time at which the release entered the sample. The first is based on known stress/strain properties of sapphire and fused silica (the impactor material), while the second is based on the release arrival shown in the PVDF gauge records (which generally indicated an arrival ~20 ns later than the first method). Both results are shown in Figure 3.10 where available.

Table 3.2. Inflection and Hugoniot points for PZT 95/5 in the present study.

Shot #	First wave				Final Wave		
	Particle Vel. Vel. (km/s)	Stress (GPa)	Shock Vel. (km/s)	Strain	Stress (GPa)	Strain	Particle Vel. (km/s)
<i>Unpoled</i>							
2677	0.074	1.99	3.66	0.0204	3.67	0.0637	0.172
2678	0.076	2.11	3.79	0.0205	5.04	0.1027	0.253
2679	0.078	2.10	3.69	0.0217	3.46	0.0564	0.156
2680	----	----	----	----	1.85	0.0201	0.071
2682	----	----	----	----	0.51	0.0063	0.021
2683	0.092	2.48	3.70	0.0248	3.87	0.0791	0.191
2690	0.091	2.45	3.68	0.0248	4.81	0.1068	0.253
2691	0.018	0.50	3.83	0.0047	0.93	0.0156	0.043
2708	0.018	0.50	3.85	0.0046	0.90	0.0121	0.038
2719	0.086	2.19	3.49	0.0247	2.28	0.0271	0.092
<i>Normally Poled</i>							
2706	0.028	0.80	3.88	0.0072	4.55	0.0849	0.222
2710	0.023	0.66	3.88	0.0060	0.94	0.0117	0.038
2717	0.021	0.60	3.89	0.0054	2.26	0.0240	0.086
2720	0.021	0.61	3.97	0.0053	2.25	0.0238	0.085
2721	0.027	0.76	3.90	0.0068	0.95	0.0109	0.037
2724	0.021	0.55	3.59	0.0059	0.66	0.0083	0.027
2725	0.023	0.67	3.93	0.0059	2.27	0.0234	0.085
2726	0.021	0.61	3.86	0.0056	2.24	0.0239	0.085
2727	0.026	0.73	3.79	0.0070	2.27	0.0256	0.089
<i>Axially Poled</i>							
2707	0.0267	0.78	4.01	0.0066	3.72	0.0644	0.173
2711	0.026	0.73	3.87	0.0067	3.37	0.0544	0.153
2712	0.019	0.51	3.79	0.0049	0.86	0.0119	0.037
2718	0.026	0.77	4.02	0.0065	2.34	0.0244	0.088
2729	0.018	0.52	3.92	0.0046	0.99	0.0143	0.043
2730	0.026	0.76	3.94	0.0067	2.21	0.0234	0.084
2731	0.024	0.67	3.94	0.0061	1.73	0.0202	0.069

Only six tests were available for release path calculation; the other four either included flyer plates that were so thick that edge effects obscured release waves (2677 and 2719) or did not exhibit release waves at all (the spall experiments, 2680 and 2682). Of those that were available, those with sapphire windows exhibited qualitatively different release paths (straight-line in this space) than did the tests with fused silica windows (which showed a distinct change in release slope at lower pressures). This difference, as mentioned above, probably is due to the tendency of the sapphire windows to reshock the sample while the fused silica windows partially release the sample (producing a region of the sample "contaminated" by a different stress regime than the bulk of the sample).

Figure 3.11. Hugoniot states observed for PZT 95/5.

The Hugoniot points alone for various PZT 95/5 experiments are shown in Figure 3.11. The break in slope at 2.2.- 2.5 GPa is the most obvious feature of these plots. There does not appear to be a systematic difference between the Hugoniot of the unpoled PZT 95/5 and the poled ($30 \mu\text{C}/\text{cm}^2$) samples.

The FE/AFE transition is not reflected well in the Hugoniot. However, it has an observable effect on the wave profiles, as shown in Figure 3.12. The suggestion is that the FE/AFE transition occurs in the unpoled material, is enhanced in the axially poled material (20 m/s break), and is suppressed in the normally poled material. This is also consistent with the dependence of the FE/AFE transition pressure on poling inferred from static experiments (Figure 1.2), wherein the axially poled samples undergo the transition at lower mean stress (pressure) than do the normally poled samples (200 MPa vs. 300 MPa).

Figure 3.12. Waveform detail observed for PZT 95/5, unpoled vs. poled. A step structure in the waveform corresponds to a segmented path in stress/volume space, here inferred to mean a FE/AFE phase transition).

Results - Spall Strength: The wave profile for Test 2682 (Figure 3.3) shows a classic pull-back signature of a finite spall strength. Recall that this test was conducted without a window, and with a foam behind the fused silica flyer, to promote spall.

Per Romonachenko and Stepanov [1980], the spall strength may be estimated as $S = S_m + \Delta S$, where:

$$S_m = \frac{1}{2} \rho_o c_l \Delta u_{pb} \quad \text{and} \quad \Delta S = \frac{h}{2} \left(\frac{dp}{dt} \right) \left(\frac{1}{c_b} - \frac{1}{c_l} \right)$$

Here, S_m is the estimate of the tensile wave strength at the free surface and ΔS is the magnitude of the stress attenuation in the tensile wave as it traverses a distance h from the spall plane to the free surface. For this problem, the initial density is $\rho_o = 7.3 \text{ gm/cm}^3$; the longitudinal and bulk sound velocities are, respectively, $c_l = 4.163 \text{ km/s}$ and $c_b = 3.919 \text{ km/s}$;

and the observed pullback amplitude is $u_{pb} = 0.010$ km/s (representing a stress drop of 0.14 ± 0.01 GPa). This stress drop occurs over a time of $0.16 \mu\text{s}$, giving $dp/dt = 0.9$ GPa/ μs . Finally, the distance h from the spall plane to the free surface may be calculated using a wave speed of 4 km/s and a rarefaction input time for the sample from the fused silica of $1.17 \mu\text{s}$; the result is 2.33 mm. Incidentally, these values give a pullback arrival time at the surface of $2.16 \mu\text{s}$, which is comparable to the observed value of $\sim 2.1 \mu\text{s}$.

This yields $S_m = 0.152$ GPa and $\Delta S = 0.016$ GPa, giving a spall strength is 0.17 GPa.

4.0 Summary

Lead zirconate titanate (PZT) with a Zr:Ti ratio of 95:5 (PZT 95/5), is initially in the ferroelectric phase. It is of interest in pulsed-power applications because it is very close to the FE/AFE transition, which corresponds to a point of shock depoling. Hence shock waves of moderate amplitude in poled PZT 95/5 are able to produce short-term pulses of high voltage and/or current.

In the present study, 10 impact tests were conducted on unpoled PZT 95/5, with 9% porosity and 2 at% Nb doping. These tests were instrumented to obtain time-resolved loading, unloading and spall signatures. As well, PVDF gauges allowed shock timing to be established explicitly.

Several common features were observed in the time-resolved wave profiles. An initial ramp loading to approximately 0.4 GPa may correspond to the FE/AFE phase transition. Related experiments conducted on poled samples in the present study show different signatures for this transition (i.e. a sharp step structure for axially poled samples, and an immediate rise without evidence of a dispersive phenomenon for normally poled samples). The implication is that the FE/AFE transition occurs under lower stress conditions in the unpoled samples than in either normally poled or axially poled samples. Similarly, based on release wave signatures, the back transformation appears to progress more rapidly in unpoled samples than in either normally poled or axially poled samples.

A clear wave separation at 2.2 GPa followed by a highly dispersive wave may correspond to the onset of plastic deformation including a large component of pore crushing. The end states, which are consistent with data from earlier studies and from related experiments conducted on poled samples in the present study, also appear to reflect crushup. This is based on the strains consistent with crushup as well as on the sample release paths which suggest a large permanent strain.

A spall strength value of 0.17 GPa is measured for a shock stress of 0.5 GPa; this decreases to a very small value for a shock strength of 1.85 GPa.

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