Pressure Induced Phase Transformation of Controlled Porosity Pb(Zr$_{0.95}$Ti$_{0.05}$)O$_3$ Ceramics

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Chemically prepared Pb(Zr$_{0.95}$Ti$_{0.05}$)O$_3$ (PZT 95/5) ceramics were fabricated with a range of different porosity levels, while grain size was held constant, by systematic additions of added organic pore former (Avicel). Use of Avicel in amounts ranging from 0 to 4.0 weight percent resulted in fired ceramic densities that ranged from 97.3% to 82.3%. Hydrostatic pressure induced ferroelectric (FE) to antiferroelectric (AFE) phase transformations were substantially more diffuse and occurred at lower hydrostatic pressures with increasing porosity. An approximately 12 MPa decrease in hydrostatic transformation pressure per volume percent added porosity was observed. The decrease in transformation pressure with decreasing density was quantitatively consistent with the calculated macroscopic stress required to achieve a specific volumetric macrostrain (0.40%). This strain was equivalent to experimentally measured macrostrain for FE to AFE transformation. The macroscopic stress levels were calculated using measured bulk modulus values that decreased from 84 GPa to 46 GPa as density decreased from 97.3% to 82.3%.

Introduction

A hydrostatic pressure of approximately 300 MPa is required to transform coarse grain size (approximately 15 μm) PZT 95/5 based ceramics from a poled ferroelectric state to the antiferroelectric state as shown by Fritz and Keck.[1] Zeuch and coworkers[2] demonstrated that PZT 95/5 ceramics, of approximately 92% theoretical density, subjected to hydrostatic pressure exhibit roughly linear volumetric strain versus stress behavior until the ferroelectric to antiferroelectric transformation occurs. Volumetric strain levels on the order of 0.40% to 0.45% were measured for mixed oxide ferroelectrics at the FE to AFE phase hydrostatic transformation pressure. Further work[3,4] by these authors showed that the pressure induced transformation behavior of poled and unpoled PZT 95/5 material was to first order quite similar, with the poled material transforming to the AFE state at slightly higher pressures and having a slightly more rapid transformation. Storz and Dungan[5,6] have demonstrated that external pore former additions, resulting in closed porosity within the specimens, help prevent high voltage breakdowns at low temperature during explosive shock wave transformation. However, relatively few, if any, results have been reported concerning the change in hydrostatic pressure induced phase transformation of PZT 95/5 based ceramics as a function of systematic changes in porosity.

Elastic moduli of brittle ceramics have been shown by several workers to be highly dependent on density. Coble and Kingery[7] have shown that for a 20% increase in porosity, the Young's modulus, E, of alumina decreases by roughly 40%. Young's modulus changes with porosity were quantified using the following equation

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$$E = E_o (1 - 1.9 p + 0.9 p^2) \cong E_o \exp(-2.2 p) \qquad (1)$$

where p is the volume fraction porosity and E_o is the Young's modulus for 100% dense alumina. A general, semi-empirical formula for the change in Young's modulus with porosity for brittle ceramics[8] is

$$E = E_o \exp(-bp) \qquad (2)$$

for which the exponent multiplier b is material dependent. Similar relationships will hold for bulk modulus, B, values as long as Poisson's ratio changes very little with porosity, as was the case for our PZT 95/5 ceramics. For the densities of this study, 82% to 97%, the bulk modulus decreases approximately linearly with porosity. If the bulk moduli are constant as a function of stress, then the applied external stress needed to reach a specific volumetric macrostrain level will also decrease in an approximate linear fashion with increasing porosity.

Previous work has shown[9,10] that fine grain PZT materials transform at lower hydrostatic pressures than larger grain samples due to enhanced internal stress. Specifically, this work represented grain sizes that were near the first and second critical grain sizes[11] for 90° domain formation, which are approximately 0.3 μm and 3 μm respectively.. Grain sizes of approximately 10 μm and 15 μm were measured for the two suites of ceramics in this study fired at 1275°C and 1345°C, respectively. Changes in the transformation pressure with grain size in this regime will be shown to be minimal. Thus, the effects of porosity on depoling behavior can to first order be isolated from grain size in this study.

II. Experimental Procedure

Chemically prepared PZT 95/5 powders were prepared using lead acetate and B-site cation alkoxide - glacial acetic acid solutions. Uniform, homogeneous powders were then precipitated from these solutions using oxalic acid.[12] The specific composition used for this study was $Pb_{0.996}(Zr_{0.953}Ti_{0.047})Nb_{0.018}O_3$ which corresponded to 0.5 mol% PbO in excess of the stoichiometric value. These chemically prepared powders were then calcined at 900°C for 8 hours to develop single phase perovskite material and to coarsen particle size. An appropriate amount of pore former was added to an approximately 400 gram aliquot of dry calcined PZT powder by containerized batch mixing using a tumbling (30 RPM) 1 quart cylindrical container with a high speed (4000 RPM) intensifier bar. Then the appropriate amount of binder solution (3% by weight HA4 acrylic binder (Harshaw Chemical) was sprayed into the tumbling powder to granulate the powder. The total tumbling time for initial tumbling, binder addition, and granulation steps is 9 minutes. The pore formers used to create porosity in the ceramics were cellulose based organic particles that have the commercial name of Avicel (FMC Corporation, Princeton, NJ). Avicel particles used in this study were roughly acicular in shape and of dimensions ranging from 5 μm to 70 μm. Powder compacts were formed by uniaxially pressing

roughly 70 grams of powder at 14 MPa (2 ksi) and then isostatically pressing the compact at 200 MPa (30 ksi). These compacts were then fired at 750°C for 4 hours with a heating rate of 0.85°C/min. This thermal treatment gently pyrolyzed the Avicel, creating pores of greater than 5 µm dimension, without causing significant damage to the rest of the ceramic.

A double crucible technique using chem-prep PZT 95/5 powders as atmosphere control was used to fire the ceramics. Typical weight losses compared to stoichiometric values were less than 0.2%. Firing temperatures of 1345°C and 1275°C with 6 hour hold times were used to densify the 70 gram chem-prep ceramic bodies. The Archimedes technique with deionized water as the suspension fluid was used to measure the densities of the specimens. Geometric densities were also measured.

Acoustic longitudinal and shear velocity measurements were performed using digitized pulse-echo waveforms from which time-of-flight measurements were made through parallel faces of the sample. Ceramic samples of approximately 1 cm X 1 cm X 1 cm dimensions with test faces polished flat and parallel to 0.001 cm were tested in both the poled and unpoled state. After initial measurements in the unpoled state, the samples were poled at 30 kV/cm for 10 seconds at 25°C using sputtered Cr//Au electrodes. The elastic moduli were determined from the elastic constants that were calculated from the acoustic velocities assuming a transverse isotropic material.

Dielectric hysteresis measurements were made with an RTV-6000 ferroelectric tester. All samples that were tested for hydrostatic depoling were encapsulated in urethane such that penetration of the Isobar H transmission fluid into the pores of the ceramics was prohibited. Hydrostatic pressure was increased at a rate of 10.3 MPa (1500 psi) per second and an 8 µF capacitor was used to collect the charge from the depoled ceramic with a capacitance of approximately 250 pF. The samples for the electrical and hydrostatic depoling measurements were sputter deposited with Cr//Au electrodes and were approximately 1 cm X 1cm X 0.1 cm.

III. Results and Discussion

Fired densities for different weight percent additions of pore former for PZT 95/5 ceramics fired at 1275° and 1345°C are shown in Fig. 1. While chem-prep PZT 95/5 ceramics fired at 1345°C exhibited densities ranging from 88.5% to 97.3% theoretical density, ceramics fired at 1275°C exhibited densities that ranged from 82.3% to 95.6% theoretical density. Approximately 1 volume percent porosity was introduced into the specimens for every 0.45 weight per cent of added Avicel for the 1345°C samples. The increase in density change per weight percent pore former for the 1275°C samples suggests that some shrinkage of the Avicel-derived porosity has occurred for the 1345°C samples. Density differences between the 1275°C and 1345°C samples were 1.7% and 6.3% for zero weight percent and four weight percent Avicel additions, respectively. Density measurements were made on 70 gram slugs with machined surfaces to enhance the accuracy of both the geometric and Archimedes techniques.

Acoustic velocity measurements permitted the calculation of both Young's and bulk moduli in both poled and unpoled specimens (shown in Table I). Longitudinal and shear velocities decreased as density decreased and were in close agreement for samples of similar density for the two different firing temperatures. Thus, the change in acoustic velocity and the calculated bulk moduli with grain size from roughly 10 μm to 15 μm was slight. It was not as significant as the effect of density on acoustic velocities and thus bulk moduli for these materials. Specifically for the 1345°C samples of 97.3% and 88.9% density longitudinal velocities in the polar direction of 4.584 mm/μs and 4.018 mm/μs were measured, respectively. Longitudinal velocities transverse to the polar direction of 4.404 mm/μs and 3.853 mm/μs, were measured for the 97.3% and 88.9% dense samples, respectively. These velocity results were mirrored by the decrease in Young's modulus with decreasing density. Specifically E_{33} for the 1275°C samples decreased from 130.2 GPa to 75.2 GPa for a 13% change in density, roughly a 3.2% change in E_{33} per percent change in density. Electrical poling had a smaller, but still significant, effect on mechanical stiffness of the ceramics. The transverse Young's modulus, E_{11}, of the 97.3% dense, 1345°C samples decreased from 132.9 to 128.6 GPa, while E_{33} increased from 132.4 GPa to 138.5 GPa upon poling. Thus, the poled ceramics were stiffer in the poled direction and softer in the direction transverse to poling, as observed for near morphotropic phase boundary PZT materials, although the effect was not as pronounced. For the above, acoustic measurements were made in the open circuit or constant charge density, D, configuration.

The change in bulk modulus with density due to added pore former is shown in Fig. 2 (a) and 2(b) and tabulated in Table I for the 1345°C and 1275°C suites of samples, respectively. Bulk modulus values for our polycrystalline materials were calculated, using both Reuss and Voigt averages, from the acoustically measured elastic stiffness coefficients of our poled ceramics shown in Table II. These elastic coefficients are slightly modified compared to those of the unpoled ceramics due to the piezoelectric effect. For the poled ceramics, c_{33}, c_{13}, and c_{12} are 5% to 15% greater than for the ceramics in the unpoled state. To calculate the average volumetric compression we require the bulk modulus corresponding to uniform pressure in the anisotropic ceramic, which is given by the Reuss average. Bulk modulus values calculated using the Voigt average technique correspond to uniform strain in the anisotropic ceramic. Differences in bulk moduli calculated using the two approximations were less than 1% for all samples in this study. The Reuss average calculated bulk moduli are presented in Table I.

The bulk modulus monotonically decreases as porosity increases. Equivalently, the materials become macroscopically softer with increasing porosity; that is, the macroscopic hydrostatic stress required to obtain a specific volumetric strain decreases with increasing porosity. Specifically, for the poled 1345°C samples, the bulk modulus decreases from 84.0 GPa to 58.2 GPa as density decreases from 97.3% to 88.9%. For the poled 1275°C samples, the bulk modulus decreases from 79.6 GPa to 46.0 GPa as density decreases from 95.6% to 82.6%. Linear approximations show roughly a 3.8% and a 3.0% decrease in bulk modulus for a 1% decrease in density, for the 1345°C and 1275°C suites of ceramics, respectively. The 1275°C samples show less of a linear approximation change, since they extend over a greater range of density. Specifically, as

porosity increases, the change in bulk modulus with porosity becomes less in brittle ceramics as higher order porosity terms have greater impact.[7] The effective bulk modulus values for poled ceramics are greater than those for the unpoled ceramics by amounts that range from 3% to 8%.

Depoling characteristics for three PZT 95/5 ceramics fired at 1275°C with densities of 95.6%, 88.9% and 82.3% are shown in Fig. 3. The transformation characteristics become more diffuse and the transformation pressure decreases with decreasing density. Similar trends were observed for the 1345°C samples. The transformation pressure is defined as the pressure for which half the total charge has been released. The change in FE to AFE transformation pressure with density is substantial. A decrease from 312 MPa (45.4 ksi) to 179 MPa (26 ksi) with a change in density of roughly 13% is measured. This implies that on the microstructural level the mean values of the individual crystallite stress and strain distributions are greater for a given externally applied pressure for the less dense ceramics. This increase in microscopic stress and strain can be attributed to stress enhancements in the vicinity of pores[13] as shown by Carroll and Holt. It is also observed that the beginning transformation pressure (10% charge release level) is lower with respect to the 50% charge release pressure for the lower density samples. This observation indicates a greater distribution of stress due to an increased volume fraction of pores in the low density ceramics. Thus, the depoling characteristics are consistent with both a greater distribution and an increased magnitude of stress and strain at the microscopic level for samples with higher porosities.

The polarization released in the hydrostatic measurements is in good agreement (typically, within 4%) with the remanent polarization values measured for 30 kV/cm, 2 Hz applied fields. Released polarization values of 32.9, 26.1 and 21.2 $\mu C/cm^2$ were obtained for the 95.6%, 88.9% and 82.3% dense ceramics, respectively. The decrease in polarization with density is greater than that expected from considering only the volume fraction of pores, with zero polarization value, within the ceramics. We attribute the greater than linear decrease in polarization with density as being due to the bending of polarization vectors around the added pores creating an effectively larger volume of low polarization material in the ceramic than just the pore volume itself. It is noted that the added pore former derived porosity is much larger than the 1 μm dimension intrinsic porosity, which should enhance the decrease in polarization with density.

We propose a straightforward macroscopic concept to permit a rough estimate of the transformation stress required to transform from the poled FE phase to the AFE phase as a function of the density of the ceramic. While this macroscopic treatment addresses the monotonic decrease in the previously defined transformation pressure, it does not address the diffuseness of the transformation. Specifically, it is assumed that with increasing hydrostatic pressure once the ferroelectric ceramic reaches a specific volumetric strain level, then the transformation to the smaller unit cell volume AFE phase will occur. The macroscopic volumetric strain needed for transformation is assumed to be independent of the density of the ceramic. As we have shown previously, as the porosity increases in these ceramics, the bulk modulus decreases and the ceramics

become softer. Thus, high porosity samples will reach the expected strain level for transformation at a lower stress level. For these ceramics, the bulk modulus, B, is given by

$$B = -P \, / \, (\Delta V/V) \qquad\qquad\qquad (3)$$

where P is the isostatic pressure and $(\Delta V/V)$ is the volumetric stain. On a microstructural level, the individual crystallites are subject to more distributed stress and strain with increasing porosity. Our definition of the transformation pressure can be considered as the pressure at which half of the population of crystallites transform to the AFE phase. If the distributions of stress and strain for the individual crystallites are roughly symmetric with pressure, then the "half population" crystallite stress would be similar to the applied macroscopic stress. Thus, the strain generated in the half population crystallite would be directly proportional to the macroscopic bulk modulus.

Fig. 4 is a schematic diagram of the strain versus stress characteristics for our samples fired at 1345°C. The poled bulk modulus values measured in aforementioned acoustic velocity measurements are used for each sample of different porosity level. The volumetric strain at which the FE to AFE transformation occurs is assumed to be 0.4%. This strain level was calculated from the depoling measurements of the 97.3% dense sample fired at 1345°C with a transformation pressure of 326 MPa (47.4 ksi). The strain value is in reasonable agreement with previous work[3] which showed volumetric transformation strains under hydrostatic stress conditions in the range of 4 to 4.5e-3 for mixed oxide PZT 95/5 specimens. Further, it is predicted that the unpoled ceramics would transform from the FE to AFE phase at slightly lower hydrostatic pressure than the poled FE ceramics due to their lower bulk modulus values. Zeuch and coworkers[3] have experimentally measured slightly lower transformation pressures for unpoled PZT 95/5.

It is assumed that the bulk modulus is constant with applied stress, that is, the strain versus stress characteristics are linear. The linear range extends from 0 MPa to the FE to AFE transformation pressure, which can be as high as 400 MPa depending on the sample. Our assumption of linear behavior over this pressure range is in good agreement with experiment as shown by the stress - strain characteristics of Zeuch in hydrostatic and triaxial stress measurements[3] for mixed oxide PZT 95/5 ceramics. The stresses corresponding to a volumetric strain of 4.0e-3 are calculated and shown in Fig. 4 as a function of density. These values are the calculated FE to AFE transformation pressures.

Measured and calculated FE to AFE transformation pressures are plotted versus density for ceramics of both firing temperatures in Fig. 5. The measured values are depicted by solid symbols connected by solid lines. Both calculated and measured data predict a monotonic decrease in transformation pressure with added porosity. The largest deviation between calculated and measured transformation pressures for the ten data points is 5.6% with most differences less than 3%. An approximate linear dependence for the measured transformation pressures on density is observed. Linear fits to the measured data result in slopes of 12.2 MPa and 9.9 MPa change in transformation pressure per percent change in density for the 1345°C and 1275°C samples, respectively.

Correlation of the calculated values of hydrostatic stress for a constant strain level to the transformation pressures measured in the depoling experiments is within the experimental errors expected from our depoling, acoustic velocity and density measurements. There is a slight increase in the measured transformation pressure for the 1275°C samples compared to the larger grain size 1345°C samples of the same density. The differences range from 7% to 11%, or roughly 25 MPa (3.6 ksi), suggesting that grain size changes in the 10 μm to 15 μm regime also have a slight influence on transformation pressure.

IV. Summary

We have shown that the hydrostatic FE to AFE transformation stress decreased with increasing porosity for PZT 95/5 ceramics. Further, quantitative agreement between measured hydrostatic transformation stresses and those calculated using a straightforward, first order macroscopic strain concept was demonstrated. Specifically, it was assumed that at a given macroscopic volumetric strain level (0.40%) poled FE ceramics transform to the AFE phase independent of density. The decrease in calculated transformation stress was due to the decrease in bulk modulus, which decreased by approximately 45% for a 15% decrease in density. It is acknowledged that this concept is a rough estimate of transformation behavior and it does not provide detailed analyses of microscopic stress distributions of individual crystallites. However, the agreement between measured and calculated FE to AFE transformation stress values using our macroscopic strain concept was shown to be very good for two suites of PZT 95/5 ceramics fired at 1275°C and 1345°C, respectively.

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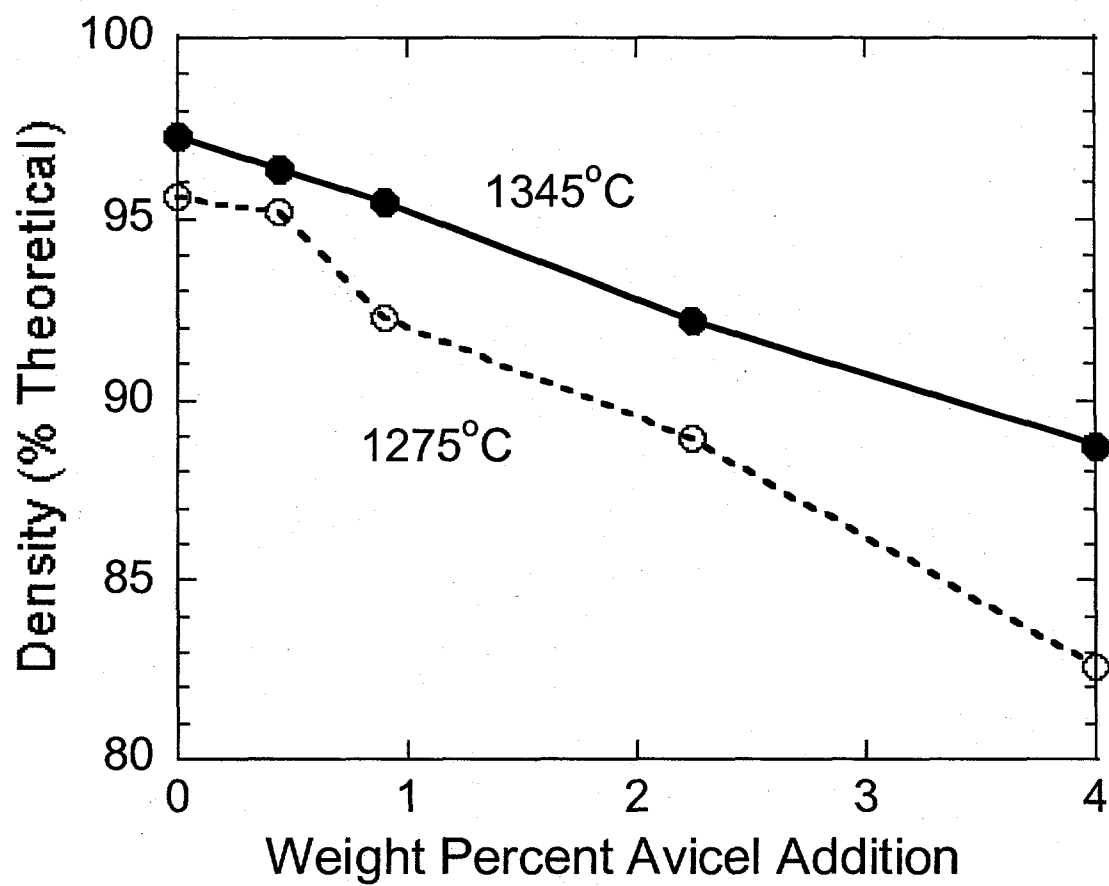
Fig. 1. Density of PZT 95/5 ceramics as a function of Avicel content.

Fig. 2. Bulk Moduli for PZT 95/5 Ceramics of different density for firing temperatures of: (a) 1275°C and (b) 1345°C.

Fig. 3. Hydrostatic depoling characteristics for PZT 95/5 ceramics fired at 1275°C of 95.6%, 88.9% and 82.6% density.

Fig. 4. Schematic diagram of macrostrain versus stress for PZT 95/5 ceramics of different densities.

Fig. 5. Measured and calculated values of FE to AFE transformation pressure for PZT 95/5 ceramics of systematically modified density fired at 1275°C and 1345°C.

Table I. Bulk and Young's Moduli of PZT 95/5 Ceramics of Different Density

Sample Description (Density, Firing Temperature)	E_{11} Unpoled (GPa)	E_{11} Poled (GPa)	E_{33} UnPoled (GPa)	E_{33} Poled (GPa)	Bulk Modulus Unpoled (GPa)	Bulk Modulus Poled (GPa)
97.3%, 1345°C	132.9	128.6	132.4	138.5	77.5	84.0
96.3%, 1345°C	123.8	120.4	127.1	128.0	72.8	76.8
95.5%, 1345°C	122.9	119.3	117.7	121.3	70.5	74.5
92.3%, 1345°C	110.6	107.2	111.4	112.8	63.4	66.2
88.9%, 1345°C	94.3	91.1	96.8	97.0	53.6	58.2
95.6%, 1275°C	127.6	123.2	125.1	130.2	75.2	79.6
95.2%, 1275°C	123.1	119.9	121.4	121.1	71.0	74.6
92.3%, 1275°C	112.5	109.4	112.4	113.7	64.0	69.2
88.9%, 1275°C	100.0	96.0	100.0	98.4	55.5	57.9
82.6%, 1275°C	79.0	77.1	74.7	75.2	44.9	46.0

Table II. Poled PZT 95/5 Ceramic Elastic Stiffness Coefficients

Sample Description (Density, Firing Temperature)	C_{11} (GPa)	C_{33} (GPa)	C_{44} (GPa)	C_{12} (GPa)	C_{13} (GPa)
97.3%, 1345°C	150.9	163.5	53.7	46.4	49.7
96.3%, 1345°C	140.3	149.5	50.0	42.5	44.3
95.5%, 1345°C	138.5	141.3	48.7	41.2	42.4
92.3%, 1345°C	123.4	129.9	44.7	35.9	37.0
88.9%, 1345°C	105.6	114.8	38.7	30.0	34.8
95.6%, 1275°C	145.6	155.6	51.4	43.7	47.7
95.2%, 1275°C	140.2	143.4	49.1	40.2	43.6
92.3%, 1275°C	126.7	132.6	45.1	37.7	39.9
88.9%, 1275°C	109.1	112.5	39.6	30.3	31.8
82.6%, 1275°C	87.9	86.3	31.1	24.6	25.3

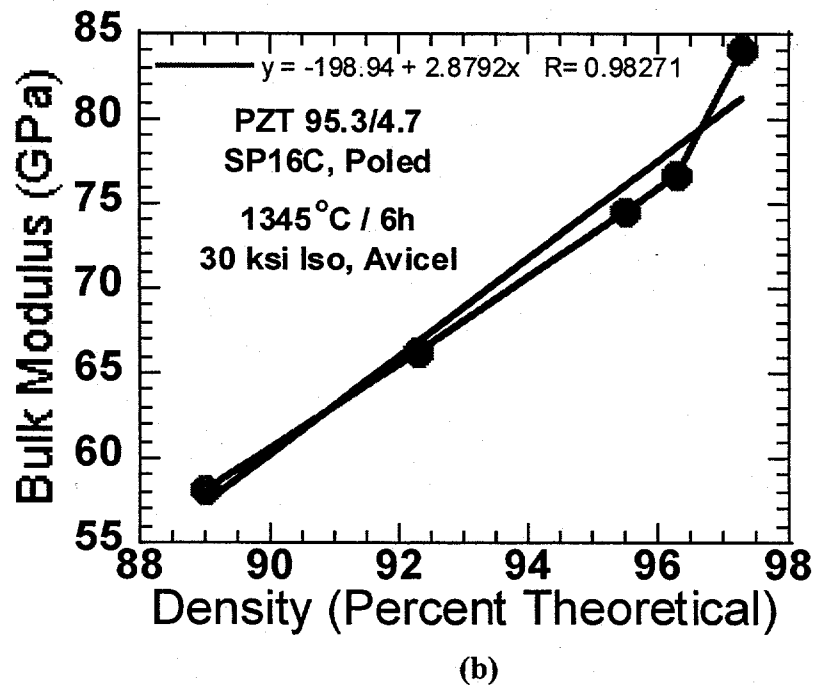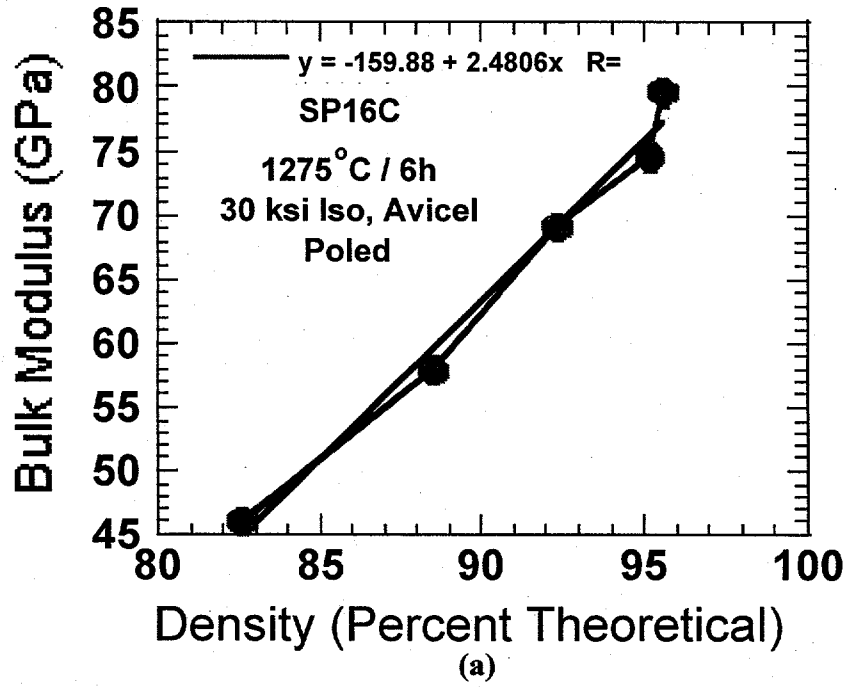Fig. 1. Density of PZT 95/5 ceramics as a function of Avicel content

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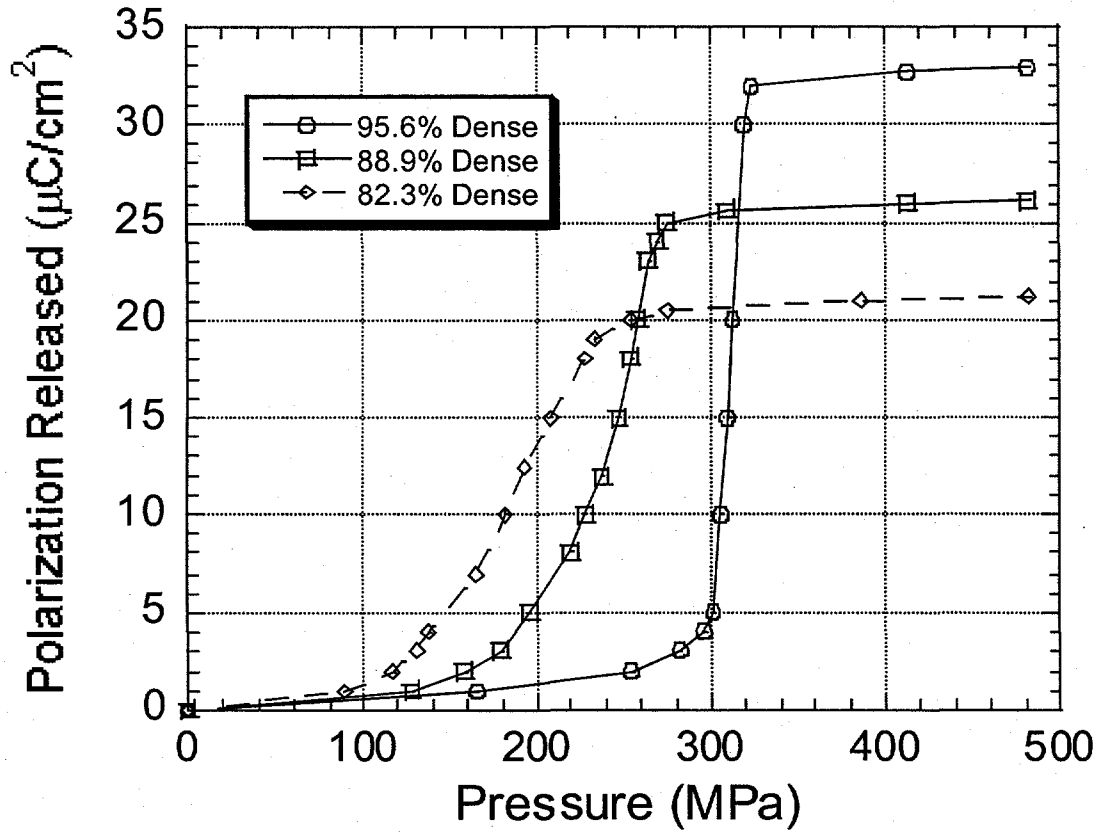
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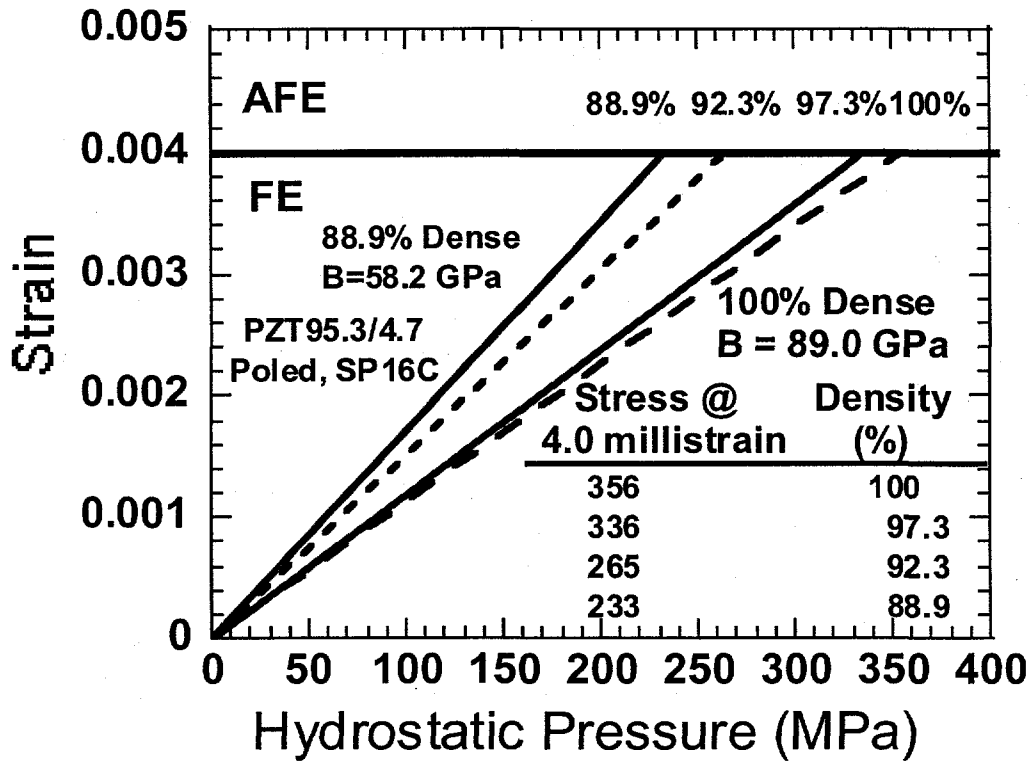
Fig. 4. Schematic diagram of macrostrain versus stress for PZT 95/5 ceramics of different densities.
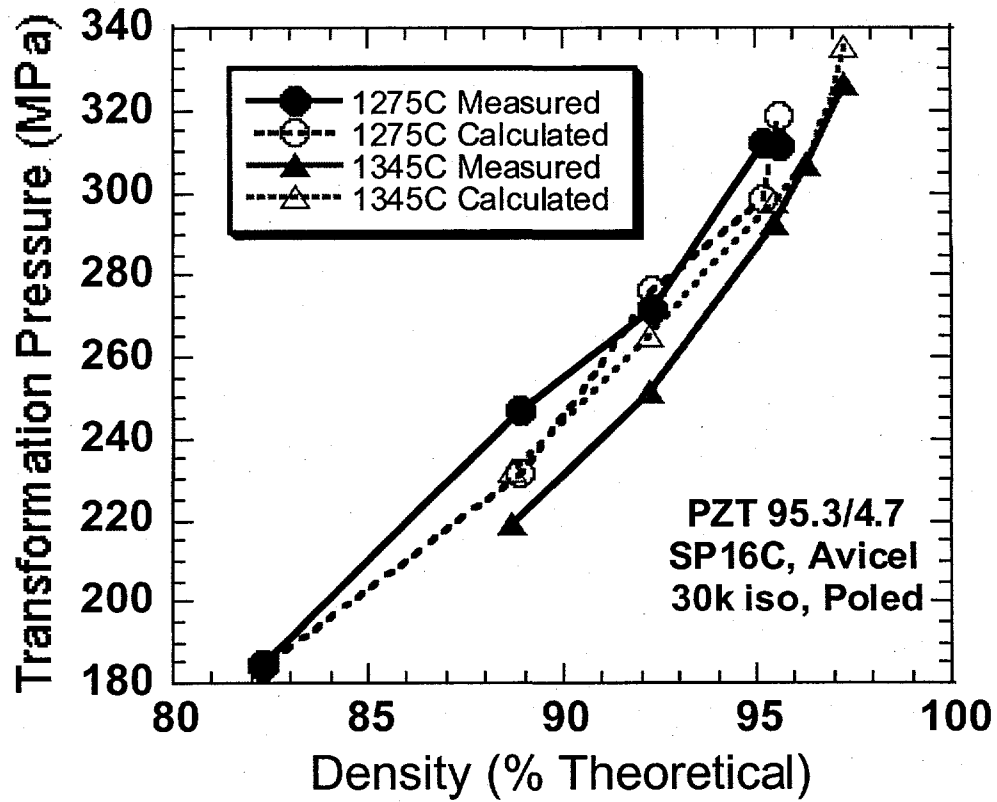
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