EFFECTS OF DOMAIN STRUCTURE ON ELECTRICALLY EXCITABLE MECHANICAL RESONANCES IN FERROELECTRIC CERAMICS

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Abstract—Recently, I have announced the existence of electrically excitable mechanical resonances in piezoelectric and ferroelectric materials which are not accompanied by any detectable electrical disturbance. These observations are, of course, contrary to conventional understanding, and they are valid for X-cut quartz, Z-cut LiNbO₃, slim loop ferroelectrics, PZT65/35, PLZT7/65/35, BaTiO₃ ceramic, Clevite PZT8 and Channel 5500 ceramic, including virgin and thermally depoled specimens of ferroelectric ceramics. In this paper, the effects of domain structure on the existence of the mechanical resonances in the ferroelectric ceramic PZT65/35 are examined. It is shown that domain structure affects not only the number of these mechanical resonances but also their amplitudes. These observations are suggestive of the notion that the existence of the domains.

1. INTRODUCTION

In two previous papers I have announced the existence of electrically excitable mechanical resonances in piezoelectric and ferroelectric materials which are not accompanied by any detectable electrical disturbance as measured by conventional techniques [1, 2]. These observations are, of course, contrary to conventional understanding. Typically, the onset of a resonance in a piezoelectric or a poled ferroelectric element driven by a cyclic electric field is detected by monitoring the admittance (or impedance) which becomes large (or small) when the resonance occurs. The divergence of the admittance is equivalent to the divergence of the time rate of change of the electric displacement so that the resonance may be viewed as an *electrical* resonance. It is generally believed that the mechanical displacement also diverges during an electrical resonance so that a *mechanical* resonance is said to occur simultaneously.

Experimental determination of the vibrational patterns of resonators is fairly difficult to achieve. Consequently, there are only very limited results in the literature on this subject. In the 1920s and the 1930s there have been some definite attempts of discern the vibrational patterns of quartz crystal resonators, see, e.g. Sections 366-368 of Cady[3]. More recently, Shaw[4], and Shaw and Sujir[5] have examined the vibrational patterns of barium titanate ceramic resonators by employing stroboscopically illuminated multiple beam Fizeau fringes. Of the twelve modes detected in each of the twenty-five cylindrical discs of different diameter to thickness ratios not a single one can be identified as the fundamental thickness resonance and none approaches uniform piston-like motion as postulated in boundary-initial value problems.[†] These authors do not allude to the phenomenon described in this paper.

Recently, the mechanical displacements of a piezoelectric or a ferroelectric element driven by a cyclic electric field are monitored using a displacement laser interferometer system. The resolution of the system is well within 5×10^{-10} m (5Å). With this system mechanical resonances are detected at particular frequencies for many piezoelectric and ferroelectric materials such as X-cut quartz, Z-cut LiNbO₃, slim loop ferroelectrics, PZT65/35, PLZT7/65/35, BaTiO₃ ceramic, Clevite PZT8 and Channel 5500 ceramic. Some of the results are reported in the two previous papers [1, 2]. In particular, the mechanical resonant frequencies of two nearly identical thin cylindrical discs of hot-pressed PZT65/35 in the virgin state are given in one of the papers, together with those of one of the discs after thermal annealing at 400°C, and the mechanical and electrical resonant frequencies of the other disc after being poled [1]. In the second paper, the responses of axially poled cylindrical discs of PZT8 ceramic with different diameter to

^{*}In this regard, the reader may also refer to Thurston[6].

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thickness ratios are examined [2]. The mechanical resonant frequencies of the discs below the lowest detectable electrical resonances are determined.

In this paper, we examine the effects of domain structure on the existence of the mechanical resonances in the ferroelectric ceramic PZT65/35. It is shown that domain structure affects not only the number of these resonances but also their amplitudes. These observations are suggestive of the fact that the existence of these mechanical resonances may be due in part to the coupling of the driving electric field to the unit cell dipoles of the domains.

2. EXPERIMENTAL DETAILS

As we have remarked earlier, the basic instrument which we utilize to determine the mechanical displacements is a displacement laser interferometer system. The essential technical details of the system are given in the paper by Allensworth [7]. In addition, we also determine the electric displacements by monitoring the charge on an integrating capacitor connected in series with the specimens.

The preceding measurements permit the collection of data which are the Lissajous oscilloscope displays of interference fringe intensity versus driving voltage and charge versus driving voltage. These displays not only furnish information regarding the amplitudes of the various quantities but they also exhibit the phase relationships between fringe intensity versus driving voltage and charge versus driving voltage. The phase relationships are crucial in discerning the onset of "resonances". In order to facilitate the ease of data reduction it is helpful though not necessary to limit the amplitude of the driving voltage so that the total change of the interference fringe intensity is less than that of half a fringe. The resolution of the mechanical displacements is such that each centimeter-division on the oscilloscope screen is equivalent to approx. 5×10^{-10} m (5 Å) depending on the intensity of half an interference fringe.

Given the Lissajous displays, it is a simple matter to discern the onset of resonances". In general, at frequencies below that of a resonance the fringe intensity and the charge are in phase with the driving voltage so that the Lissajous displays are straight lines. As the frequency of the driving voltage is increased, say, towards a mechanical resonance the amplitude of the fringe intensity increases together with a phase shift until the fringe intensity and driving voltage are in quadrature, i.e. they are 90° out of phase. At this point the Lissajous display is in the shape of a circle or an ellipse depending on the scales. Continuing increase of the frequency of the driving voltage is accompanied by a decrease of the fringe intensity and further phase shift. Eventually, the fringe intensity and the driving voltage are 180° out of phase so that the Lissajous display is again a straight line but its slope is the negative of what it was originally. Similar observations are valid for the Lissajous display of charge versus driving voltage during the occurrence of an electrical resonance. The preceding observations are consistent with the solution of a second-order ordinary differential equation with a sinusoidal forcing term, and provide us with the opportunity to identify independently the onset of mechanical and electrical resonances. For our present purpose it suffices to regard the frequencies at which fringe intensity versus driving voltage and charge versus driving voltage achieve quadratures as the mechanical and electrical resonant frequencies, respectively.

After attaining the 180° phase shift two things can, in general, happen with regard to a mechanical resonance. First, the amplitude of the fringe intensity may decrease with increasing driving voltage frequency until it vanishes and then reappears so that the fringe intensity and the driving voltage are in phase. This appears as a rotation of the straight line Lissajous display. Second, the fringe intensity and the driving voltage may remain out of phase at 180° with increasing driving voltage frequency until the onset of a succeeding resonance at 90° phase shift. Eventually, the fringe intensity and the driving voltage are again in phase. After an electrical resonance the amplitude of the charge seems to always decrease with increasing driving voltage frequency until it vanishes and then reappears in the in phase condition. These observations suggest that the physical causes of the mechanical resonances may be very complex.

Equivalently, we may also collect the data displayed on a time base. Given the temporal histories of the fringe intensity, the charge and the driving voltage, it is also a simple matter to ascertain the phase relationships between these quantities and the onset of mechanical and electrical resonances. For a particular driving voltage frequency we usually excite only the fundamental of a mechanical resonance unless the amplitude of the driving voltage is very large.

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Occasionally, the fundamental and second harmonic are excited: However, the mechanical resonance associated with an electrical resonance exhibits multiple frequencies fairly frequently. In these instances it is possible to excite the fundamental alone if the amplitude of the driving voltage is decreased sufficiently.

The two rectangular specimens which we are examining in the current study are PZT65/35 supplied by Channel Industries, henceforth designated as PZT65/35-C1 and PZT65/35-C2. These specimens are initially in the virgin state. Both specimens have width $w = 1.524 \times 10^{-2}$ m and length $l = 5.080 \times 10^{-2}$ m. The thickness of specimen PZT65/35-C1 is $t = 2.235 \times 10^{-3}$ m, and that of PZT65/35-C2 in $t = 2.261 \times 10^{-3}$ m.

In order to attain essentially stress-free conditions the specimens are supported on their lower w - l faces with three ball bearings. The geometry of the spacings of the ball bearings are shown in Fig. 1. The signal beam of the interferometer is directed at the geometric centers of the upper w - l faces of the specimens at which we have glued very small spectral mirrors. This permits the determination of the mechanical displacements perpendicular to the w - l plane. However, it is entirely possible that the geometric centers of the w - l faces of the specimens may be the nodal points of certain resonant vibrational patterns at particular frequencies, and having only small mirrors at the centers need not permit us to determine all the resonant frequencies in some instances. Larger mirrors will undoubtedly affect the responses of the specimens. We are, therefore, exploring the possibility of using highly polished specimens so that the electroded faces are spectral and we may scan these faces.

Specimen PZT65/35-Cl is electroded on its w - l faces with vapor deposited aluminum of thickness 3.0×10^{-7} m (3000 Å). A sequence of experiments is conducted with this specimen by arranging its domain structure in different configurations. Specimen PZT65/35-C2 is initially electroded on its t - l faces. After poling the specimen perpendicular to the t - l plane with a linearly increasing electric field having a maximum magnitude of 2.1×10^6 V/m at 25 s (remanent polarization being 0.3657 C/m²), the specimen is returned to an electrically neutral state exhibiting no remanent polarization by slowly varying the switching field. In this state there are more dipoles essentially parallel to the w - l plane than the other two planes. The electrodes on the t - l faces are then removed and the w - l faces are electroded with vapor deposited aluminum of thickness 3.0×10^{-7} m (3000 Å).

Another specimen which we are examining is a piece of initially virgin hot-pressed PZT65/35 in the shape of a cylindrical disc prepared at our Laboratories, henceforth designated as PZT65/35-S2. It has a diameter of 3.607×10^{-2} m and a thickness of 8.128×10^{-4} m. The center circular region of the faces of the disc with diameter 1.793×10^{-2} m are electroded with vapor deposited aluminum of thickness 3.1×10^{-7} m (3100 Å). The disc is symmetrically supported on three ball bearings equally spaced at 1.270×10^{-2} m. Here, it is held in contact with the ball bearings by three small springs directly over the locations of the ball bearings and exerting a total force of less than 6.675×10^{-2} N. A very small spectral mirror is glued at the center of the top face. The axial mechanical displacements of the disc are determined using the laser interferometer system.

3. EXPERIMENTAL RESULTS

Temperature fluctuations and residues of fluids used to clean the specimens affect not only the resonant frequencies but also the magnitudes of the mechanical displacements as deter-



Fig. 1. Geometry of support conditions of specimens PZT65/35-Cl and PZT65/35-C2.

mined by the laser interferometer. Accordingly, the specimens are usually allowed to stabilize for at least 12 hr after being placed in their test configurations, and the entire system including the laser interferometer is enclosed. The temperature within the enclosure is maintained at a constant 25.6°C. This procedure ensures that the results obtained are as repeatable as possible. The magnitudes of the mechanical displacements and to a much lesser degree the resonant frequencies may also be affected by the spacings of the support ball bearings. The reason for this is quite obvious because the measured values of the mechanical displacements depend on the vibrational patterns whose manifestations in turn depend on the support conditions. It is, nevertheless, meaningful and useful to compare results between different specimens for the same support conditions and between different resonant frequencies for the same specimen.

We have also noticed that the resonant frequencies and the peak mechanical displacements of the mechanical resonances are quite repeatable before the specimens are allowed to resonate electrically. Immediately after a specimen has resonated electrically there are appreciable changes in the frequencies and displacements of its mechanical resonances. However, if the specimen is allowed to stabilize for sufficiently long time, e.g. 12 hr, its former results are again obtained. The reasons for this are not understood at this time—one reason may be that the specimen is heated due to power dissipation during an electrical resonance.

The results reported in this paper are taken after the specimens have been allowed to stabilize. However, in instances for which we give the mechanical resonances between electrical resonances the data are taken sequentially with increasing driving voltage frequencies. This is because it is not convenient to allow the specimens to stabilize in order to determine the characteristics of the mechanical resonances which occur between electrical resonances.

As we have remarked earlier the domain structure of specimen PZT65/35-Cl is arranged in various different configurations and a sequence of experiments is conducted during the process. From the virgin state, the specimen is subjected to three and a half cycles of a linearly varying cyclic electric field with amplitude 1.566×10^6 V/m and period 100 s. The corresponding remanent polarization is $+0.3617 \text{ C/m}^2$. The electrically neutral state is then established by slowly varying the switching field. However, we did not attain true electrical neutrality in this instance and the residual polarization is -6.5×10^{-3} C/m² which is about 1.8% of the remanent polarization. From this electrically neutral state the specimen is thermally annealed at 400°C for 1 hr. By poling the specimen with a linearly increasing electric field having a maximum magnitude of 1.566×10^6 V/m in 25 s, a remanent polarization of + 0.2777 C/m² is attained indicative of the fact that the switching characteristics of the domains have altered. Again, the specimen is annealed at 400°C for 1 hr and poled with a linearly increasing electric field having a maximum magnitude of 1.544×10^6 V/m in 25 s. This time the remanent polarization is 0.3061 C/m^2 . Notice that besides the virgin state and the electrically neutral state, we have two thermally depoled states and three poled states. No mechanical resonances are detected for this specimen in the virgin state at the geometric center of its upper w - l face. In Tables 1 and 2, we compare the results for the two thermally depoled states and the three poled states, respectively. The results corresponding to the electrically neutral state are compared to those corresponding to the laterally electrically neutral state of specimen PZT65/35-C2 in Table 3.

Specimen PZT65/35-S2 is first poled with a linearly increasing electric field with maximum magnitude 2.1×10^6 V/m in 25 s (remanent polarization being 0.3365 C/m²), arranged in the electrically neutral state, and poled again with the same electric field. Since the results corresponding to the virgin state and the first poled state are reported in a previous paper[1], they will not be repeated here. Hence, we shall record the results corresponding to the electrically neutral state and the second poled state, and they are given in Table 4.

4. DISCUSSIONS

Table 1 shows that the frequencies of the mechanical resonances exhibited by the specimen PZT65/35-Cl in the two thermally depoled states are quite consistent while the peak mechanical displacements of the resonances associated with the first thermally depoled state are generally larger than those associated with the second thermally depoled state. We know that the domain structures of the two states are different because their domain switching characteristics from

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PZT65/35-C1					
First Thermally	Depoled State	Second Thermally Depoled State			
Resonant Frequencies	Peak Mech. Displ.	Resonant Frequencies	Reak Mech. Displ.		
1.372 kHz	2.0x10 ⁻¹⁰ m	1.468 kHz	0.6x10 ⁻¹⁰ m		
1.969 kHz	1.11x10 ⁻⁹ m	2.010 kHz	0.6x10 ⁻¹⁰ m		
3.429 kHz	4.59x10 ⁻⁹ m	3.464 kHz	0.6x10 ⁻¹⁰ m		
17 `.049 kHz	6.3x10 ⁻¹⁰ m	17.050 kHz	1.7x10 ⁻¹⁰ m		
35.110 kHz	4.7x10 ⁻¹⁰ m	35.239 kHz	0.3x10 ⁻¹⁰ m		
40.110 kHz	6.7x10 ⁻¹⁰ m	40.184 kHz	1.7x10 ⁻¹⁰ m		
43.072 kHz	1.5x10 ⁻¹⁰ m				
59.470 kHz	0.5x10 ⁻¹⁰ m	59.619 kHz	0.6x10 ⁻¹⁰ m		
71.448 kHz	0.5x10 ⁻¹⁰ m	71.422 kHz	(No data taken)		
83.166 kHz	0.5x10 ⁻¹⁰ m	83.068 kHz	(No data taken)		
107.168 kHz	0.5x10 ⁻¹⁰ m	(No additional			
123.601 kHz	0.5x10 ⁻¹⁰ m	data taken)			

Table	1.	Mechanical	resonant,	frequencies	and	peak	mechanical	displacements of	thermally	depoled
					PZ	ZT65/3	5-CI			

Amplitude of driving voltage: 5V RMS.

	PZT65/35-C1						
First Poled State, (0.3617 C/m ²)		Second Po (0.277	led State ¹ , 7 C/m ²)	Third Roled State ² (0.3061 C/m ²)			
Resonant Frequencies	Peak Mech. Displacements	Resonant Prequencies	Peak Mech. Displacements	Resonant Prequencies	Peak Mech. Displacements		
2.000 kHz	8.37x10 ⁻⁹ m	2.017 kHz	7.35x10 ⁻⁹ m	2.111 kHz	6.49x10 ⁻⁹ m		
3.555 kHz	9.16x10 ⁻⁹ m	3.487 kHz	1.53x10 ⁻⁸ m	3.529 kHz	2.03x10 ⁻⁸ m		
17.566 kHz	9.6x10-10m	17.330 kHz	1.84x10-9m	17.350 KHz	1.01x10 ⁻⁹ m		
E35.782 kHz ³		E35.724 kHz ³		E35.673 kHz ³			
41.425 kHz	2.55x10 ⁻⁹ m	40.709 kHz	1.99x10 ⁻⁹ m	40.995 kHz	1.26x10 ⁻⁹ m		
		43.973 kHz	9.4x10-10m	44.246 kHz	8.0x10 ⁻¹⁰ m		
E123.319 kHz	9.65x10 ⁻⁹ m	E123.584 KHz	7.68x10 ⁻⁹ m	E123.325 kHz	7.40x10 ⁻⁹ m		

Table 2. Resonant frequencies and peak mechanical displacements of poled PZT65/35-CI

Amplitude of driving voltage: 5V RMS.

Electrical resonances are prefixed by the letter E.

¹Phase shift between fringe intensity and driving voltage detected at 1.393 kHz.

²Phase shift between fringe intensity and driving voltage detected at 1.645 kHz.

³Associated mechanical resonances exhibit multiple frequencies.

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PZT65/35 Electrically Ne	-Cl Sutral State ¹	PZT65/35-C2 Laterally Electrically Neutral State		
Resonant Frequencies	Reak Mech. Displ.	Resonant Frequencies	Peak Mech. Displ.	
1.339 kHz	4.7x10 ⁻¹⁰ m	1.437 kHz	4.1x10-10m	
1.854 kHz	1.43x10 ⁻⁹ m	1.979 kHz	2.6x10 ⁻¹⁰ m	
3.458 kHz	1.07x10 ⁸ m	3.559 kHz	3.3x10-10 _m	
		6.415 kHz ²		
		9.437 kHz	5.0x10 ⁻¹⁰ m	
		13.112 kHz	9.6x10-10m	
17.394 kHz	3.66x10 ⁻⁹ m	17.576 kHz	2.52x10 ⁻⁹ m	
		20.508 kHz ³		
		28.413 kHz	3.7x10 ⁻¹⁰ m	
		30.242 kHz ⁴	3.7x10 ⁻¹⁰ m	
35.842 kHz	5.4x10 ⁻¹⁰ m	34.689 kHz	9.2x10 ⁻¹⁰ m	
41.201 kHz	6.9x10 ⁻¹⁰ m	40.614 kHz	7.78x10 ⁻⁹ m	
		59.901 kHz	3.59x10 ⁻⁹ m	
		71.339 kHz	5.5x10 ⁻¹⁰ m	
		73.437 kHz	3.8x10-10m	
		84.118 kHz	2.30x10 ⁻⁹ m	
		97.933 kHz	5.0x10 ⁻¹⁰ m	
		112.989 kHz	1.43x10 ⁻⁹ m	
125.615 kHz	6.5x10 ⁻¹⁰ m	128.339 kHz	3.5x10-10 _m	

Table 3. Resonant frequencies and peak mechanical displacements of electrically neutral PZT65/35-Cl and laterally electrically neutral RZT65/35-C2

Amplitude of driving voltage: 5V RMS.

¹Phase shifts between charge and driving voltage detected at 17.866 kHz, 59.544 kHz and 90.040 kHz. Phase shift between fringe intensity and driving voltage detected at 1.382 kHz.

²Excitation of the second harmonic.

³Resonance exhibits multiple frequencies.

⁴Accompanied by phase shift between charge and driving voltage.

these states are different yielding different states of remanent polarization for essentially the same applied electric field.

The mechanical and electrical resonant frequencies exhibited by the specimen PZT65/35-Cl (Table 2) in the three poled states are again quite consistent even though they have different remanent polarizations. There does not seem to be any corelation between the values of the remanent polarizations and the peak mechanical displacements. Notice that electrical resonances now occur at 35 kHz and 123 kHz which show up as mechanical resonances in the thermally depoled states.

All the mechanical resonant frequencies exhibited by the specimen in the thermally depoled states also occur as mechanical or electrical resonant frequencies in the poled states except at 59, 71, 83 and 107 kHz. Similar mechanical resonances at 59, 71, 84 and 112 kHz and others also occur in specimen PZT65/35-C2 whose domains are arranged in the laterally electrically neutral state, but not in specimen PZT65/35-C1 when its domains are arranged in the electrically neutral

PZT65/35-62 Electrically Neutral State ¹		PZT65/35-62 Second Poled State ²		
Resonant Prequencies	Peak Mech. Displ.	Resonant Frequencies	Peak Mech. Displ.	
		1.493 kHz	8.6x10 ⁻¹⁰ m	
2.179 kHz	8.6x10-10m	2.173 kHz	7.85x10-9m	
		2.607 kHz ³		
5.458 kHz	9.27x10 ⁻⁹ m	5.439 kHz	1.41x10 ⁻⁸ m	
		5.838 kHz	1.6x10 ⁻¹⁰ m	
6,181 kHz	5.96x10 ⁻⁹ m	6.103 kHz	4.19x10 ^{-9m}	
17.592 kHz	1.09x10 ⁻⁸ m	17.301 kHz)4	3.9x10 ⁻¹⁰ m	
		17.953 kHz	2.52x10-9m	
		27.747 kHz	1.03x10 ⁻⁹ m	
		41.037 kHz	1.86x10 ⁻⁹ m	
68.833 kHz	6.4x10-10m	69.928 kHz	2.10x10 ⁸ m	
		E72.078 kHz	5.82x10-9m	
105.042 kHz	4.6x10-10m			
146.676 kHz	4.0x10-10m	148.839 kHz	4.3x10-10m	
		187.492 kHz	2.54x10 ⁻⁹ m	
190.316 kHz	1.51x10-9m	E189.561 kHz	8.01x10 ⁻⁹ m	
		194.138 kHz	2.52x10-9m	

Table 4. Resonant frequencies and peak mechanical displacements of electrically neutral and poled PZT65/35-S2

Amplitude of driving voltage: 5V RMS.

Electrical resonances are prefixed by the letter E.

¹ Thase shifts between fringe intensity and driving voltage detected at 1.397 kHz,

124.853 kHz. Possible mechanical resonances at 28.281 kHz, 39.234 kHz and 39.867 kHz - barely detectable.

²Phase shifts between fringe intensity and driving voltage detected at 1.354 kHz, 9.826 kHz and 53.655 kHz.

³Mechanical resonance consists of the fundamental and the second harmonic.

⁴Fringe intensity and driving voltage achieve quadrature successively without achieving 180° phase shift between these frequencies.

state (Table 3). In the laterally electrically neutral state there are more dipoles essentially parallel to the w - l plane (perpendicular to the driving field) than the other two planes. This is suggestive of the notion that these mechanical resonances can be due to the coupling of the driving voltage to the perpendicular dipoles. This observation is substantiated by two facts. First, in the electrically neutral state, there are more dipoles parallel to the driving field than the other two directions and these mechanical resonances are not detected. Second, the peak mechanical displacements associated with these mechanical resonances are much larger than those in the thermally depoled states.

The mechanical resonant frequencies of specimen PZT 65/35-Cl in the electrically neutral state are consistent with the mechanical and electrical resonant frequencies of specimen PZT65/35-Cl in the poled states. In these instances there are more dipoles parallel to the driving field than the other two directions, and these resonances can be due to the coupling of the driving voltage to the parallel dipoles which may be easier to excite. Notice that the peak mechanical displacement of the mechanical resonance at 3.4-3.5 kHz remains large, independent of whether specimen PZT65/35-Cl is poled or is in the electrically neutral state.

The mechanical and electrical resonant frequencies of specimen PZT65/35-S2 in the second poled state (Table 4) are consistent with those reported in a previous paper for the first poled state[1]. The domain structures of the two states are, of course, different. Therefore, differences in the peak mechanical displacements are detected, but they are of comparable magnitudes. The interesting results of the specimen PZT65/35-S2 occur in the electrically SS Vol. 20, No. 2---C

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neutral state. First, a mechanical resonance is detected at 190 kHz which shows up as an electrical resonance at 189 kHz in the second poled state, but nothing analogous occurs at 72 kHz. Second, the peak mechanical displacement of the mechanical resonance at 17 kHz is quite large when specimen PZT65/35-S2 is in the electrically neutral state. Curiously, the same is true for the 17 kHz mechanical resonance of specimens PZT65/35-Cl and PZT65/35-C2 in either of the electrically neutral states.

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