

The behavior and calibration of some piezoelectric ceramics used in the STM

by S. Vieira

The high resolution and displacement measurement in the scanning tunneling microscope are dependent upon the behavior of the piezoelectric ceramics used for moving the tip. In this paper certain characteristics and features of piezoelectric ceramics relevant to the desired precision are discussed. These characteristics are the relaxation aftereffects that follow the change of the applied electric field, and the temperature dependence of the piezoelectric response. The above effects have been analyzed in four commercial piezoelectric ceramics by the three-terminal capacitance measurement technique.

Introduction

The scanning tunneling microscope (STM) [1] is a powerful tool for the study of surfaces at atomic resolutions. In the STM the devices which control tip position and motion are

fabricated from commercially available piezoelectric ceramics. To obtain the required resolution and stability in an STM, it is necessary to understand the dependence of the piezoelectric properties of these materials upon temperature, voltage, heat treatment, and other experimental factors. In this paper, we present the results of a study of the behavior of several commercial piezoceramics in experimental environments similar to those of an STM. The experimental quantity measured in these materials was the change in length, Δl , in the direction perpendicular to the polar axis when an electric field was applied along the polar axis. These Δl were measured at different temperatures with varying electric fields. Special attention was paid to the observation of relaxation aftereffects in these piezoceramics. As is well known [2], when an electric field is applied to these materials there is an almost instantaneous strain, α , followed by a much slower one, i.e., the relaxation aftereffect. For the piezoelectric ceramics studied in this work, the aftereffect follows very accurately the logarithmic relation, $\beta \ln t$. Therefore the total strain, $\Delta l/l_0$, can be described by

$$\frac{\Delta l}{l_0} = \alpha + \beta \ln t, \quad (1)$$

where the parameters α and β may be electric-field- and temperature-dependent, and t is the time.

This logarithmic behavior is characteristic of many aftereffect phenomena and is due to the presence of

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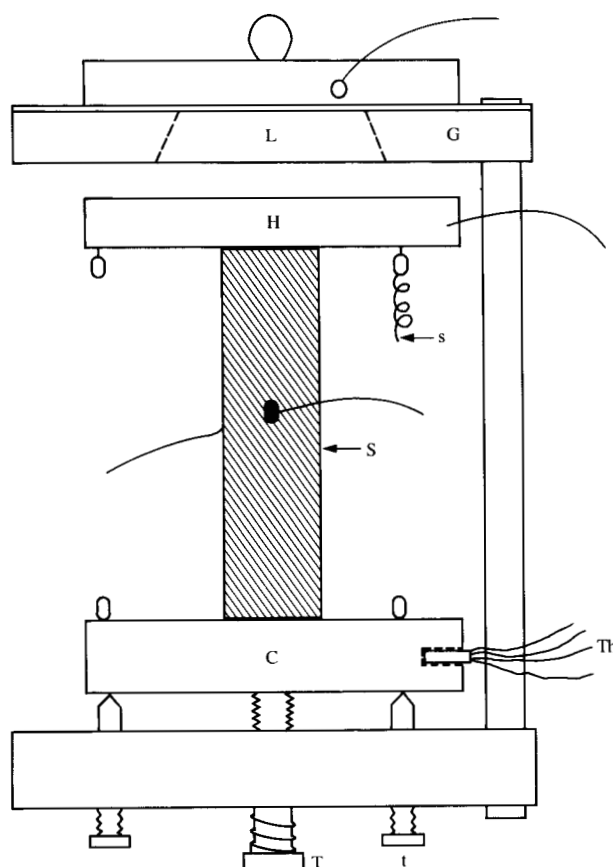


Figure 1

Schematic cross section of the piezoelectric cell: C, copper block; L, low-potential electrode; H, high-potential electrode; G, guard ring; s, spring; S, sample; t, parallelism adjustment screws; T, locking screw; and Th, germanium thermometer.

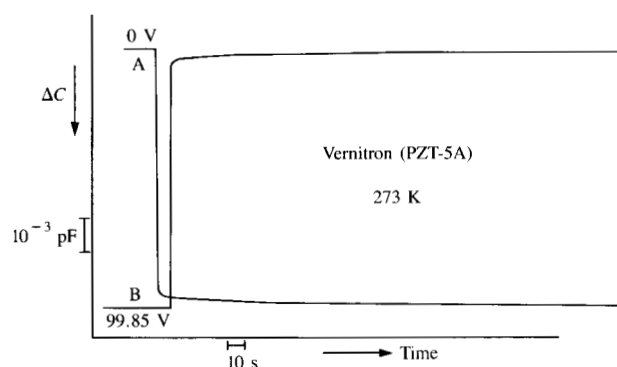


Figure 2

Typical response showing the relaxation phenomena for Vernitron PZT-5A. These curves are taken directly from the recorded data and represent the capacitance change ΔC vs. time (s) for two voltage changes: Curve A, from 0 to 99.85 V and Curve B, from 99.85 to 0 V.

thermally activated processes with widely varying activation energies. As indicative examples we cite Plessner [3], who reported this behavior for the permittivity and power-factor of ceramic barium-titanate; Street and Woolley [4] in connection with the phenomena of magnetic viscosity; and Davis and Thompson [5] on creep in a precipitation-hardened alloy. In these examples the change in an external parameter (electric field, magnetic field, or mechanical stress) causes an aftereffect which follows a logarithmic dependence upon time. The phenomenon is important because of its influence on the stability and reproducibility of the STM measurements.

Experiment

The measurement technique used involves a modification of the low-temperature thermal expansion cell developed in our laboratory [6] and is based upon the precise determination of very small changes in electrical capacitance. **Figure 1** is a schematic diagram of the piezoelectric cell. The sample (S) is fixed between the high-potential electrode (H) of the parallel plate capacitor and the copper block (C). In most of the measurements the specimen was fastened to the sample holder with epoxy resin; however, in some cases its position was fixed by means of the three springs (s). (No difference in experimental results was observed between the two mounting methods.) The capacitor is formed by the high-potential electrode (H) and the low-potential electrode (L), which is surrounded by the guard ring (G). The gap between the electrode plates is adjusted so that the capacitance is about ten picofarads. The parallelism between the plates is obtained by introducing a plastic sheet successively into the gap in three different positions. The three screws (t) are adjusted until the same variation in capacitance is observed upon insertion of the plastic sheet, thereby indicating that the electrode plates are parallel. Finally the block (C) is locked with screw (T), which ensures the mechanical stability of the systems. The cell hangs from the top of the cryostat inside a vacuum container, which is surrounded by an outer vacuum container that can be immersed in liquid helium for the low-temperature measurements. The capacitance changes are measured with a GR1621 bridge system (General Radio, Concord, MA), the output of which is observed on an *xy* recorder. The resolution of the capacitance system, 10^{-7} pF, corresponds to a change in sample length of about 10^{-2} Å. For $T < 100$ K, the temperature was measured with a germanium thermometer calibrated between 1.5 K and 100 K.

The samples analyzed in this work were the following:

1. A prism of Vibrit 200, $25 \times 3.25 \times 3$ mm (Siemens, Erlangen, Federal Republic of Germany). This sample was previously used in the scanning tunneling microscope of Baro et al. [7].
2. A prism of PZT-5A from Vernitron; $28 \times 7 \times 2.65$ mm (Vernitron Piezoelectric Division, Bedford, OH).

3. A tube of PZT-5A from EBL Crystal Products; 39.3 mm long, with a 6.35-mm outer diameter and a wall thickness of 0.5 mm (EBL Crystal Products, East Hartford, CT).
4. A prism of Philips PXE-41; $27.15 \times 9.9 \times 0.3$ mm (Philips, Eindhoven, the Netherlands).

Results

• For $T > 77$ K

Figure 2 shows the typical response, as shown by the capacitance change ΔC , of Sample 2 at 273 K to the application of an electric field and the behavior when the field is switched off. In each case, after the initial rapid change in length, the length change attributed to the aftereffect is clearly observable. For each sample about ten measurements were taken at three different temperatures: 77 K, 273 K, and 293 K. The experimental curves for all samples were fitted to Equation (1), and the mean values of the parameters α and β thus obtained, along with the estimated experimental errors, are reported in Table 1. The α and β values are normalized to an electric field of 1 V/cm. To get the actual dimensional change for a sample of length l and width d , it is necessary to multiply α and β in Table 1 by the factor $(l/d)V$.

The experimental conditions in which α and β were obtained are the following:

Sample 1 The applied electric fields ranged between 0.007 kV/cm and 0.6 kV/cm. Within this interval the values of the α and β are constant.

Sample 2 The values of Table 1 correspond to an electric field of 0.377 kV/cm. We observed that for this material α is electric-field dependent, while β is not. The dependence of α upon electric field decreases with temperature and is very small at 77 K.

Sample 3 The applied electric field ranged from 0.2 kV/cm to 1 kV/cm. The values in Table 1 are for an electric field of 0.2 kV/cm. As in Sample 2, β is field independent. The dependence of α upon electric field at three different

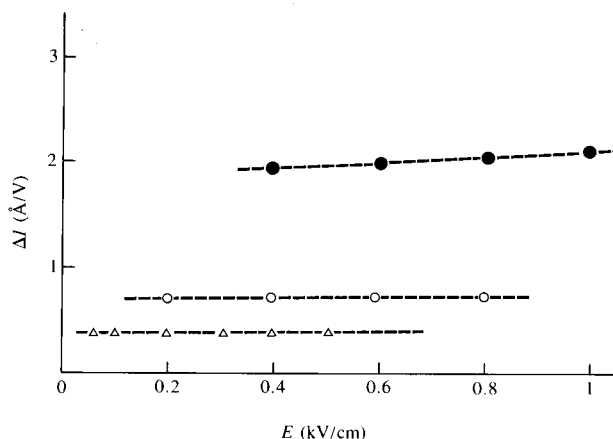


Figure 3

Electric-field dependence of the α parameter of Equation (1) for Sample 3, at $T = 4.15$ K (Δ), 77 K (○), and 293 K (●). The data are normalized as in Table 1.

temperatures is shown in Figure 3. The α field dependence for this specimen is also very small at 77 K and negligible at 4 K.

Sample 4 The applied electric field ranged from 0.06 kV/cm to 0.27 kV/cm; as in Sample 1, α and β are field independent.

In all the samples $\Delta l/l_0$ typically relaxed for 30 minutes after an abrupt electric-field change. The analysis to obtain the coefficients was performed with data for $t < 200$ s (except for Sample 4 at 278 K, for which the coefficients were from the data for $t < 15$ s). For Sample 4, the coefficients obtained from the analysis of the data at long relaxation times are inconsistent with the coefficients obtained from short relaxation times. This was not the case for Samples 1, 2, and 3, or for Sample 4 at 77 K.

• For 2 K $< T < 77$ K

Figure 4 shows the data for temperatures from 2 K to 50 K. In this temperature range the behavior of the piezoelectric

Table 1 Values of the α and β coefficients of Equation (1) when the applied electric field is changed 1 V/cm (α and β are in units of Å/V). Samples are numbered as in the text.

Sample	293 K		273 K		77 K	
	α	β	α	β	α	β
1			0.99 ± 0.02	0.065 ± 0.010		
2	1.85 ± 0.04	0.021 ± 0.004			0.79 ± 0.02	0.011 ± 0.002
3	1.92 ± 0.04	0.028 ± 0.005			0.72 ± 0.02	0.011 ± 0.02
4			1.26 ± 0.03	0.022 ± 0.04	1.28 ± 0.03	0.015 ± 0.003

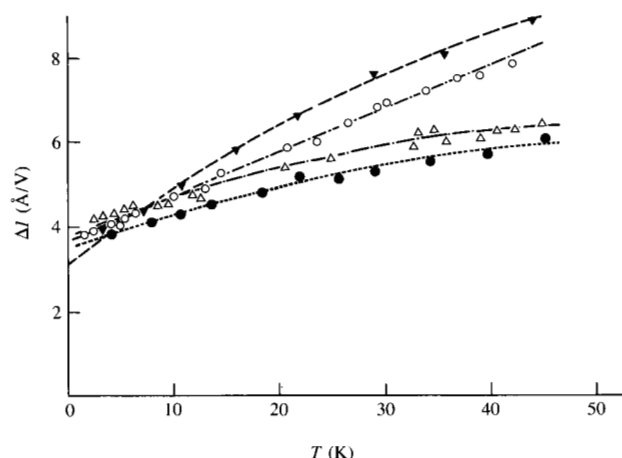


Figure 4

Experimental data for Δl for $T < 50$ K. Sample 1, $\circ$; Sample 2, Δ ; Sample 3, $\bullet$; and Sample 4, $\blacktriangledown$. The data are normalized as in Table 1.

Table 2 Coefficients of Equation (2). Values have been normalized as in Table 1.

Sample	$\alpha(0)$	c	n	Range of validity
1	0.36 ± 0.01	0.010 ± 0.001	1	$T < 40$ K
2	0.37 ± 0.01	0.016 ± 0.001	0.75	$T < 45$ K
3	0.34 ± 0.01	0.015 ± 0.001	0.75	$T < 45$ K
4	0.31 ± 0.01	0.024 ± 0.002	0.85	$T < 45$ K

deformation with the temperature is very simple. For all the samples, the dimensional changes can be described by

$$\alpha(T) = \alpha(0) + cT^n. \quad (2)$$

In Equation (2), $\alpha(T)$ is the instantaneous length change, namely without relaxation. The relaxation aftereffect is negligible at temperatures less than 45 K; above this temperature, the relaxation aftereffect increases with increasing temperature. Table 2 lists the values of the $\alpha(0)$, c , and n obtained by fitting the experimental data to Equation 2. The $\alpha(0)$ and c values have been normalized in the same way as the α and β coefficients of Table 1.

Summary

This work has shown that the use of piezoelectric ceramics in the scanning system of the STM requires an understanding of how the piezoelectric deformation is effected by temperature, electric field, and the environment in which it is used. For instance, during the bake-out of the STM ultrahigh-vacuum system, the piezoceramics are heated to almost 200°C for several hours, and this thermal treatment induces structural changes that may affect their

calibration. Measurements of Sample 4 heated in vacuum at 180°C for ten hours indicated a significant increase for the α and β coefficients. Further work must be done, with emphasis on the effect of temperature stabilization on the coefficients in Equations (1) and (2).

The relaxation aftereffect is important because of its temperature dependence. The constant, β , in Equation (1) increases proportionally with a power of temperature, i.e., as T^k . For Sample 2, k is near 0.5.

The use of these materials for low-temperature operations requires the knowledge of the parameters in Equation (2).

We wish to point out that Equation (1) allows the calculation of the actual change in length when different periodic driving voltages are applied for low-frequency scanning.

In conclusion, this work has examined the temperature and electric-field dependence of the piezoelectric response of several ceramics. The results show principally that the relaxation aftereffect is temperature dependent and becomes unimportant at low temperatures where the α coefficient has a simple temperature dependence.

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References

1. G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel, *Phys. Rev. Lett.* **49**, 57 (1982).
2. R. W. Basedow and T. D. Cocks, *J. Phys. E: Sci. Instrum.* **13**, 840 (1980).
3. K. W. Plessner, *Proc. Phys. Soc. A* **69**, 1121 (1956).
4. R. Street and J. C. Woolley, *Proc. Phys. Soc. A* **62**, 562 (1949).
5. M. Davis and N. Thompson, *Proc. Phys. Soc. B* **63**, 847 (1950).
6. R. Villar, M. Hortal, and S. Vieira, *Rev. Sci. Instrum.* **51**, 27 (1980).
7. A. M. Baro, R. Miranda, J. Alaman, N. Garcia, G. Binnig, H. Rohrer, Ch. Gerber, and J. L. Carrascosa, *Nature* **315**, 253 (1985).

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