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Characterization of piezoceramic crosses with large range scanning capability and applications for low temperature scanning tunneling microscopy

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We have developed a large amplitude piezoceramic scanner which should have numerous applications. Scanning tunneling microscopy (STM) and other scanning probe microscopies predominantly use piezoceramics for the scanning elements. Similarly adaptive optics, high resolution lithography, and micromanipulators are other examples of research which regularly utilize piezoceramic scanners. We present a new geometry for a piezoceramic scanner which allows for both high resolution (~nanometers) and large amplitude (~400 μm) displacements. The cross-shaped geometry makes it possible to produce extremely long pieces with very high tolerances. We have shown its effectiveness by using it as the major component of a low temperature STM (LTSTM). This LTSTM is unique in two distinct ways: the scan range at low temperature is a factor of 10 larger than those reported and the coarse, approach mechanism is a single component piezoceramic—making coarse approach *in situ* much quieter and easier than in other designs. © *1995 American Institute of Physics.*

I. INTRODUCTION

Since its invention in 1982, the scanning tunneling microscope (STM)[1] has revolutionized the study of surfaces and nanoscale objects. The scanning tunneling microscope's (STM's) ability to image and manipulate individual atoms is well known.[2] Used in this way, the STM utilizes a high resolution scanner with a very limited scan range. However, there are other areas of research which require the STM to have a long range scanning capability but where atomic resolution is not necessary. These areas include nanolithography,[3] nanoscale manipulation,[4] imaging the lattice of fluxoids (in a superconductor),[5] or studying mesoscopic phenomena. Additionally, adaptive optics and other areas of research that utilize micromanipulators would benefit from a large range piezoceramic ("piezo") element controllable from the nanometer scale to hundreds of microns.

We have developed a low temperature STM (LTSTM), capable of multirange scanning and having electronically controlled coarse positioning. The main component of this microscope is a long piezoceramic "cross."[6] The configuration of the LTSTM may be easily changed to accommodate a small or large scanning range while retaining the electronic coarse approach capability. The availability of a coarse approach mechanism allows one to mechanically decouple the sample and the scanning probe microscope (SPM) (via soft springs) from the remainder of the low temperature or ultrahigh vacuum (UHV) environment (no rigid mechanical linkages being required). The LTSTM's compact design allows for insertion into the restricted geometry of most cryostats and can easily be used at room temperature or UHV. The coarse approach mechanism is simple to implement and reliable at both room and low temperatures. It is controlled electronically and therefore the only mechanical coupling to the LTSTM is through small diameter wires.

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II. RESPONSE OF A POLARIZED CROSS

The LTSTM is fully interfaced to commercial scanning electronics and software[7] with additional electronics made to drive the coarse positioning mechanism and long range scanner (see Fig. 1). It is 17.78 cm in length and has as its largest diameter 2.24 cm. The main component of the LTSTM involves a long piezoceramic cross;[8] it is cross shaped in the plane perpendicular to its long axis (see Fig. 2). The polarization of each of the legs of the cross is indicated by arrows in Fig. 2(b).

The cross is 8.57 cm long, 0.7 mm thick, and 6.4 mm wide and is machined from a piezoceramic ingot having a square cross section, after which it is plated and poled (see Appendix for repoling procedure). Made in this way, a long cross may be easily machined with very high tolerances unlike piezo tubes which are usually extruded and are susceptible to deformities. The cross is made from a high efficiency piezo by the Ragspace Corporation[8] (PKR-7M) having piezoelectric constants of $d_{31}=-350\times10^{-12}$ m/V and $d_{33}=800\times10^{-12}$ m/V. This material is ferroelectric. Consequently a large, critical electric field opposing the polarization direction will cause the polarization to change. Another related consequence is a nonlinear response for large electric fields (although still lower than the critical field) opposing polarization. This nonlinearity may be seen in Fig. 5(a), a large range scan. In the upper right portion of the scan, the

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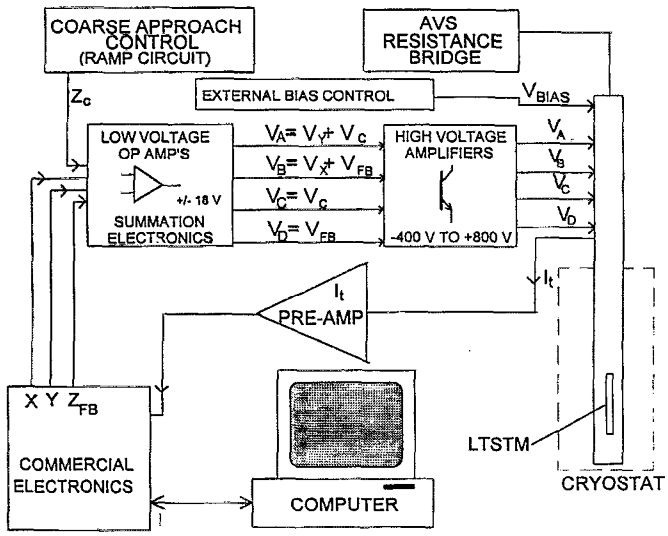

FIG. 1. Block diagram of the experimental setup.

electric field opposes the polarization and the grid appears slightly nonlinear. In the bottom right portion of the scan, the field is parallel to the polarization and the grid is linear. A calibration program may be applied to a scan such as this to correct for the nonlinearity but has not been done to date. The lowest resonant frequency of the cross is 15 kHz. The hysteresis of the cross has also been studied. The results are shown in Fig. 3.

Applying voltages to the different quadrants of the cross allows it to scan in three orthogonal directions.[9] We define X and Y as the scanning directions, Z_{COARSE} the coarse approach direction and Z_{FB} the feedback direction (parallel to Z_{COARSE}). V_X, V_Y, V_{COARSE}, and V_{FB} are defined as the corresponding voltages that are applied to the cross to obtain movement in these directions. If the quadrants of the cross are labeled A, B, C, and D (see Fig. 2), the combinations of voltages required for three-dimensional movement may be represented by

$$\begin{aligned} V_A &= V_Y + V_{\mathrm{COARSE}}, \\ V_B &= V_X + V_{\mathrm{FB}}, \\ V_C &= V_{\mathrm{COARSE}}, \\ V_D &= V_{\mathrm{FB}}. \end{aligned} \tag{1}$$

First we consider movement in the Z direction. If a voltage (e.g., $+V_{\mathrm{COARSE}}$) is applied equally to quadrants A and C

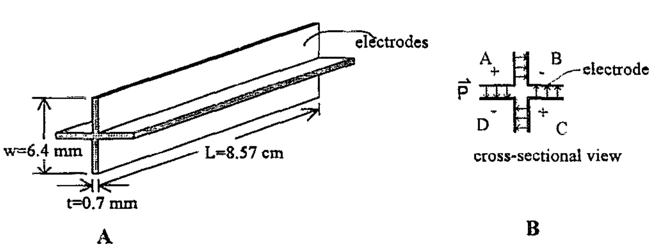

FIG. 2. Diagram of piezoceramic cross: (a) dimensions of cross; (b) cross section indicating polarization of cross.

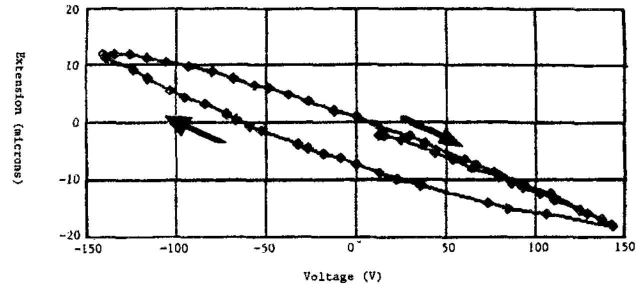

FIG. 3. Hysteresis of piezoelectric cross.

of the cross ($E \| P$), the cross will expand in the direction parallel to the polarization and contract perpendicular to it (see Fig. 4(a)). The net result is that the cross shrinks along the Z axis. To move in the Y direction, a scanning voltage (e.g., $+V_Y$) is applied to only the A quadrant. This causes only one quadrant to shrink along the Z axis. In this case, the net result is movement along the Y direction (see Fig. 4(b)). In order to use the cross as a three-dimensional scanner, the voltages for X, Y, and Z must be electronically added and applied to the four quadrants as described in Eq. (1).

To get into feedback, a high, positive voltage (V_{COARSE}) is applied to quadrants A and C. This results in movement of the tip away from the sample. The sample is then moved close to the tip by means of an 80 thread/in. screw and an optical microscope. To get into feedback, the voltage on quadrants A and C is slowly ramped down until a tunneling

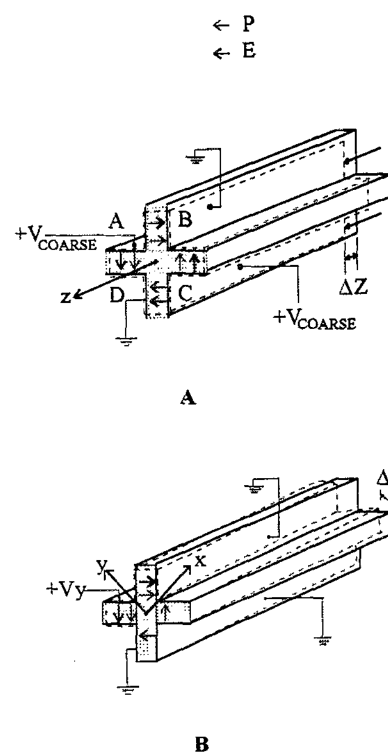

FIG. 4. (a) Schematic of how the piezoceramic cross changes length when $+V_{\mathrm{COARSE}}$ is applied to the A and C quadrants. The dotted lines indicate how the cross would change when the voltage is applied. The electric field is parallel to the polarization direction. (b) A schematic of how the cross scans in the Y direction when $+V_y$ is applied to quadrant A.

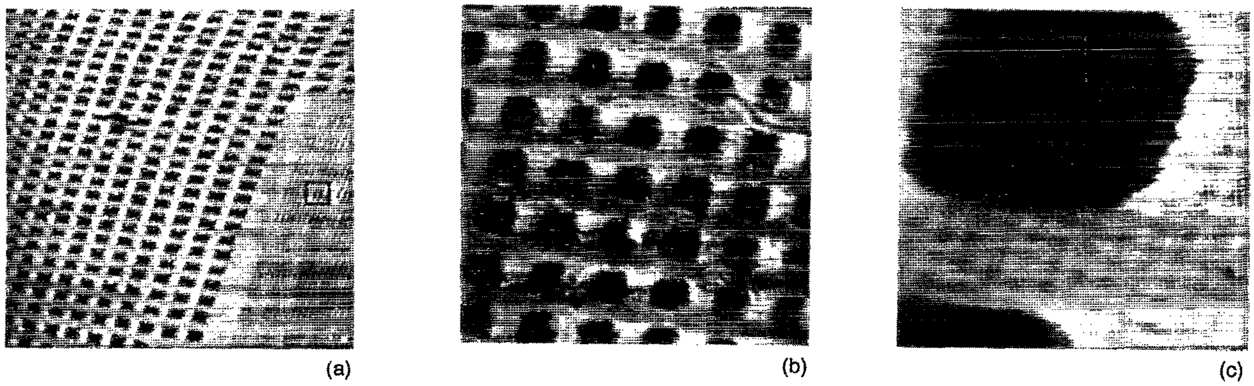

FIG. 5. Scans of a niobium-coated silicon grid: (a) A scan in the large area, large height mode; the cross as XY scanner and Z feedback piezo, the tube is grounded. Scan range is 225 μm. (b) A scan in the large area, high resolution height mode; cross as XY scanner, the tube as Z feedback piezo. Scan range is 15 μm. (c) A scan in the high resolution mode; the tube as XY scanner and Z feedback piezo. Scan range is 3 μm.

current is established. At this point, the coarse approach voltage is held constant and the feedback signal (V_{FB}) is applied to quadrants B and D; a positive feedback signal results in the tip moving closer to the surface, a negative signal results in the tip retracting from the surface. All electrical connections to the piezos are made via 0.1-mm-diam wire and are anchored to the LTSTM housing in order to better decouple the piezo elements from any mechanical vibrations.

III. ELECTRONICS

The electronics used for the coarse approach mechanism and scanning signals were designed for producing high voltages (maximum voltage swing 1.2 kV). A transformer and voltage multiplier circuit are used to convert 120 V_{ac}, 60 Hz to regulated -400 to $+800$ V_{dc}. Low voltage OP AMP's are used to add the input signals (the ramp for the coarse approach, the feedback and the XY scan signals). These signals are then amplified by high voltage transistors which are in the OP AMP feedback loop to preserve linearity. This circuit allows output voltages between -400 and $+800$ V (see Fig. 1). The output of the high voltage circuit is very quiet, linear, and has minimal coupling between the quadrants during scanning.

IV. A DUAL SCANNING SYSTEM

The cross may be used as the sole piezo scanner. For large range or surveying scans, this is the best configuration. However, the feedback response of the cross in the Z direction is coupled to the X or Y directions because the piezo cross is not perfectly symmetrical (one or two of the legs may be slightly thicker than the others). Therefore, for smaller range scans in which this coupling becomes more apparent, a piezo tube (0.64 cm diameter, 1.27 cm in length) may be placed at the end of the cross to be used as the feedback piezo. With these two piezoceramic elements we are able to accommodate a wide range of samples—from the large area, large height samples down to small scale samples. For example, we are able to scan small features of interest which may be sparsely spread out over a large area. This capability is of particular interest for high vacuum or low temperature work in which is difficult to move the sample once the desired pressures or temperatures have been reached.

In the large range, large height scan mode, the cross is used as the coarse positioner, the XY scanner, and the Z feedback piezo. The tube electrodes are grounded together to avoid charge build up with temperature change in this configuration.[10] Figure 5(a) is a 225$\times$225 μm scan of a niobium-coated silicon grid taken in the above configuration. The large squares are 10$\times$10 μm with 6 μm separation, the smaller squares are 2$\times$2 μm with 1 μm separation.

In the large range, high resolution height mode, the tube is used as the Z feedback piezo instead of the cross. The cross is still used as the XY scanner. Figure 5(b) is a 15$\times$15 μm scan of the area indicated by a square in Fig. 5(a). It was taken in the large range, high resolution height mode.

In the high resolution scan mode, the cross is used only as a coarse positioner and the Z feedback and XY scanning signals are applied to the piezo tube.[11] For the highest resolution imaging, Styrofoam supports should be placed along the length of the cross and the LTSTM housing to reduce mechanical noise. An example of a scan taken in this mode is seen in Fig. 5(c). The scan range is 3$\times$3 μm.

The signals applied to the two piezos may be changed remotely during low temperature (or UHV) operation. Therefore, a large scan may survey the sample using the large range, large height mode. To investigate a small feature in this scan area, the voltages on the cross are held constant while the tube is used as a conventional high resolution scanner.

As the LTSTM is cooled to its base temperature, the thermal expansion of the piezos is differential in that contraction occurs in the direction parallel to polarization and expansion occurs in the direction perpendicular to polarization.[10] Since the polarization direction for the cross is perpendicular to its long axis, the cross expands along this axis as it is cooled. To compensate, a voltage is applied to the cross to keep the distance from the surface constant as the cross cools. Because of this large thermal expansion, the voltage required to compensate exceeds the breakdown voltage of the cross. Therefore, it is determined how far the tip must be brought out of tunneling range at room temperature

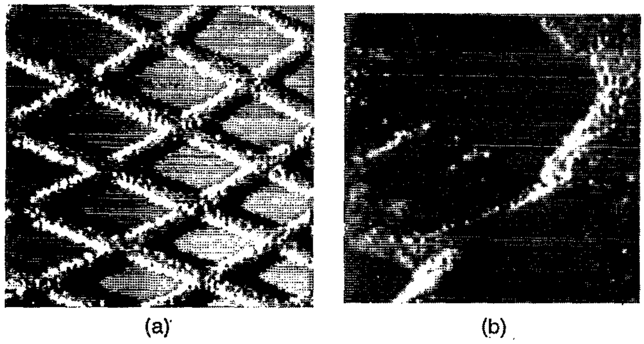

FIG. 6. (a) Room temperature scan of a 1000 mesh TEM grid. Scan size is 75×75 μm, scan voltage is ±40 V. (b) Low temperature (77 K) scan of 1000 mesh TEM grid. Scan size is 35×35 μm, scan voltage is ±40 V.

so that at low temperature, tunneling can be established. This is done by putting a 360° protractor on the 80 thread/in. screw and pulling the tip down a predetermined amount. Before base temperature is reached, a tunneling current is established and maintained so as to track the change is size of the piezos. Figures 6(a) and 6(b) are examples of room temperature and low temperature scans. The sample is a standard 1000 mesh TEM grid with squares 19×19 μm, bars 6 μm, and thicknesses of 13 μm. Both scans were taken in the large range, high resolution Z mode. The scanning voltages applied to the cross quadrants were ±40 V to obtain a range of 75 μm. Figure 6(b) is a low temperature scan (77 K). The scanning voltages applied to the cross quadrants were again ±40 V to obtain a scan range of 35 μm. It is worth noting that the reduction in scan range is significantly less than for other reported piezoceramics. In Fig. 6(b) the tunneling current becomes saturated at one corner of the image and zero within the square. This is because the piezoelectric coefficients for the piezo tube are reduced significantly at low temperature.

Another possible solution to the problem of thermal expansion, is to place the tip perpendicular to the long axis of the cross, place the sample vertically, and use one of the XY quadrants as the feedback direction (see Fig. 7). This will result in rectangular scans as the movement per volt along the long axis is significantly smaller than perpendicular to it. This configuration is being tested at this time.

V. PERFORMANCE

All piezos have been calibrated at room temperature using an optical He–Ne interferometer (λ=632.8 nm). The piezo tube calibration was determined to be 75.3 Å/V in Z, and 95.6 Å/V in XY directions at room temperature. At 77 K, this decreased to 37.5 Å/V in Z, 25.9 Å/V in XY, and at 4 K, 7 Å/V in Z, 9.2 Å/V in XY. The reduction in movement is consistent with other published results.[12] The piezo cross was calibrated at 69.4 nm/V in the Z direction and 800 nm/V in the X and Y directions at room temperatures. Interferometry was not performed on the cross at low temperatures. The results presented were obtained by scanning the calibration grid. The XY calibration was reduced to 288.2 nm/V at 77 K.

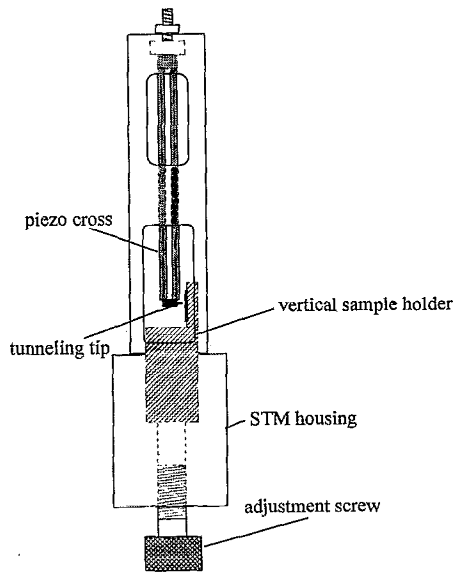

FIG. 7. Temperature compensated LTSTM with vertical sample holder.

The PKR-7M was less affected by the change in temperature than the PZT-5H being reduced by a factor of approximately 2 at low temperatures.

The maximum scan range of the LTSTM at room temperature is over 300 μm and has been shown to be at least 35 μm at low temperatures (77 K). The maximum scan range in the most common configuration which is large amplitude, high resolution Z, is limited by the piezo tube rather that the cross; in that if the sample plane and scanning plane are not perfectly parallel, the tube can adjust for change in heights of 3.31 μm at room temperatures and only about 330 nm at low temperatures (4 K).

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APPENDIX: REPOLARIZATION OF PIEZOCERAMICS

Periodically, it becomes necessary to repolarize the piezoceramics. Polarization may be destroyed when excessive voltage or heat is applied to the piezo. Additionally, the polarization of the piezos will decay with time and thermal cycling. To repolarize a cross, leads are placed on the electrodes (or faces) as indicated in Fig. 8. To protect the high voltage wire from shorting to other wire, all wires are encased in Voltrex tubing (0.38 mm nominal inner diameter, wall thickness 0.017 mm minimum, 600 V/0.001 in. dielectric strength).

The cross was placed in a glass test tube which was filled with silicone (Dow Corning, 200 fluid, 10^5 cs viscos-

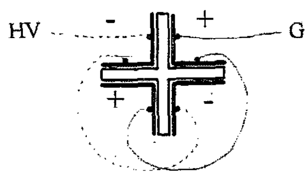

FIG. 8. Placement of leads on the piezo electrodes.

ity) to act as an insulator. This was put into a desiccator and pumped on with a roughing pump until all bubbles had risen to the surface (~30 min). Bubbles tended to form between the edges and along the corners of the piezos during polarization if this was not done. Next, the test tube was placed within a copper tube attached to a copper plate which acts as a radiator and heats the test tube evenly (see Fig. 9). The copper tube is partially filled with mineral oil or another fluid which acts as a bath. This assembly sits on the hot plate and conducts heat evenly around the test tube.

The piezo is then heated to the desired temperature.[13] The electrodes are grounded together[14] during heating to reduce stress and charge build-up. The high voltage source was a Kepco operational power supply, 0–2000 V, 0–10 mA, OPS 2000. Slowly (in increments of 50 V) the high voltage was decreased to −200 V after which the system was allowed to reach equilibrium for 15 min. This process was repeated until −1200 V had been reached. If the voltage was turned up too quickly, the long piezo tended to crack or break from the induced stress. The voltage remained at −1200 V

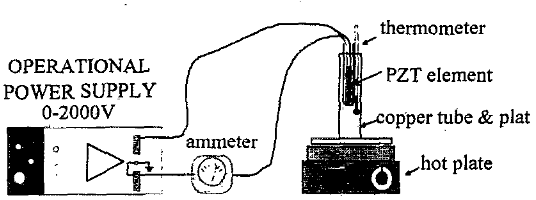

FIG. 9. Block diagram of the repolarization setup.

and at the desired temperature for 1 h after which the hot plate was turned off. The system was allowed to slowly cool to room temperature with the high voltage applied. Once at room temperature, the high voltage was slowly turned down leaving about −50 V applied before disconnecting from the high voltage power supply. This was used as a check by measuring the voltage on the piezo. It should hold the −50 V charge for a long time (days). If −50 V was not measured, then the repolarization was unsuccessful and must be repeated. The cross was cleaned off with acetone and the edges of the piezo was checked at this time for any arcing marks which may have occurred during polarization.

[1] G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel, Phys. Rev. Lett. **49**, 57 (1982).
[2] D. M. Eigler and E. K. Schweizer, Nature **344**, 524 (1990).
[3] G. C. Rosolen, A. C. F. Hoole, M. E. Welland, and A. N. Broers, Appl. Phys. Lett. **63**, 2435 (1993).
[4] M. A. McCord and D. D. Awschalom, Appl. Phys. Lett. **57**, 2153 (1990).
[5] In the low field, mixed state ($H \approx H_{c1}$), the spacing of the fluxoids may be on the order of microns. In order to fully investigate this state, a large scanning range at low temperature is required. A large scanning range is also useful as the field increases to better investigate fluxoids' interaction and movement.
[6] I. V. Lyubinetsky, P. V. Mel'nik, N. G. Nakhodkin, and A. E. Anisimov, Vacuum **46**, 219 (1995) and V. K. Adamchuk, A. V. Ermakov, I. V. Lyubinetsky, G. A. Zhitomirsky, and A. E. Panich, Prib. Tech. Eksp. **5**, 182 (1989).
[7] TopoMetrix Corporation, 5403 Betsy Ross Dr., Santa Clara, CA 95054-1162.
[8] Piezoceramic of Ragspace Corporation, PCR-7M, Physical Research Institute, 194 Stachki Ave., Rostov-on-Don, 344104, Russia.
[9] V. K. Adamchuk, A. V. Ermakov, I. V. Lyubinetskii, G. A. Zhitomirsky, and A. E. Panich, Prib. Tech. Eksp. **5**, 182 (1989).
[10] A. M. Simpson and W. Wolfs, Rev. Sci. Instrum. **58**, 2193 (1987).
[11] G. Binnig and D. P. E. Smith, Rev. Sci. Instrum. **57**, 1688 (1986).
[12] M. A. Dubson and Jeeseong Hwang, Rev. Sci. Instrum. **63**, 3643 (1992).
[13] The Curie temperature of the PKR-7M piezoelectric is 175 °C. However, silicones heated above 120 °C decompose into dangerous byproducts—among them benzene and formaldehyde, see MSDS sheets for complete listing. In order to fully repolarize the crosses, these operations should be performed under a hood.
[14] A change in temperature will introduce stress in the piezo which results in a voltage across the electrodes—in order to reduce stress as much as possible during the heating process, the electrodes should be grounded together.