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Cite as: Appl. Phys. Lett. **73**, 3851 (1998); <https://doi.org/10.1063/1.122914>

Submitted: 22 September 1998 • Accepted: 22 October 1998 • Published Online: 23 December 1998

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Piezoelectric measurements with atomic force microscopy

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(Received 22 September 1998; accepted for publication 22 October 1998)

An atomic force microscope (AFM) is used to measure the magnitude of the effective longitudinal piezoelectric constant (d_{33}) of thin films. Measurements are performed with a conducting diamond AFM tip in contact with a top electrode. The interaction between the tip and electric field present is a potentially large source of error that is eliminated through the use of this configuration and the conducting diamond tips. Measurements yielded reasonable piezoelectric constants of X-cut single-crystal quartz, thin film ZnO, and nonpiezoelectric SiO₂ thin films. © 1998 American Institute of Physics. [S0003-6951(98)01852-X]

When an electric field is applied to a piezoelectric material, it strains due to the converse piezoelectric effect. Measurement of these small piezoelectric displacements is important in materials characterization, and several techniques,¹⁻⁴ including atomic force microscopy (AFM)⁵⁻⁸ have been employed. The AFM technique has the advantage of being able to measure the piezoelectric effect at single points on a sample, and the tip can be rastered to generate piezoelectric images.⁶ Most AFM piezoelectric measurements have been of the phase of the piezoelectric response rather than the magnitude. Ferroelectric domains have been imaged by recording the piezoelectric phase.^{5,7-9}

A few studies have employed AFM to determine the magnitude of the piezoelectric effect. Gruverman *et al.*⁸ performed measurements of lead zirconate titanate (PZT) thin films by applying a voltage between a conducting tip and a back electrode. In this configuration, it is more difficult to accurately determine the electric field, but the measurements can be performed readily and directly upon the piezoelectric material. Also, the piezoelectrically excited region may be constrained by the surrounding material. Zavala *et al.*⁶ made similar measurements on PZT, and in addition performed measurements with a conducting tip on an electrode. In this configuration, the tip supplies current to the electrode and also follows the piezoelectric motion.

We present an improved AFM method optimized for measuring the magnitude of the piezoelectric effect. Measurements are performed with the tip in contact with a top electrode and current is supplied to the electrode through an external probe. This configuration offers several advantages. The applied field is well defined; the piezoelectrically excited region is large compared to the tip radius; electrical contact to the top electrode is reliable; and tip degradation will be reduced since minimal current passes through the tip.

In an ideal AFM piezoelectric measurement, an electric field is applied to a piezoelectric material, and the tip accurately follows the piezoelectric motion. However, a significant challenge of AFM piezoelectric measurements is that the tip motion can be due to a combination of piezoelectricity, electrostriction, and electrostatic interactions between the

tip and electric field present.¹⁰ If the piezoelectric is driven by a voltage at a frequency f , the piezoelectric motion will also be of frequency f , since the piezoelectric constant represents the linear relation between the strain and applied field. The higher order electrostrictive term depends upon the square of applied field, and would therefore be at frequency $2f$. The displacement of the tip due to its interaction with the electric field also exhibits an f component. So a measurement of the f component of the tip deflection is a superposition of piezoelectric motion and motion due to the electrostatic interaction. In order to accurately measure the magnitude of the piezoelectric constant, the interaction between the tip and electric field must be minimized. The need to reduce this tip-field interaction determines the type of tips that can be used in piezoelectric measurements.

Piezoelectric measurements were performed in contact mode with a commercially available AFM (Park Scientific M5) with the addition of a function generator (HP33120A) and a lock-in amplifier (SR510). A schematic of the measurement system is shown in Fig. 1. The function generator drives the piezoelectric capacitor. For the measurements described here, a frequency of 1 kHz was employed, and the peak-to-peak potential was varied up to 20 V. The 1 kHz frequency is above most environmental noise frequencies and well below the tip resonances. In our experiments, the applied voltage is stepped through computer control, and the AFM tip displacement at the frequency f is recorded using a lock-in amplifier. The piezoelectric constant can be determined from the slope of the resulting displacement vs ap-

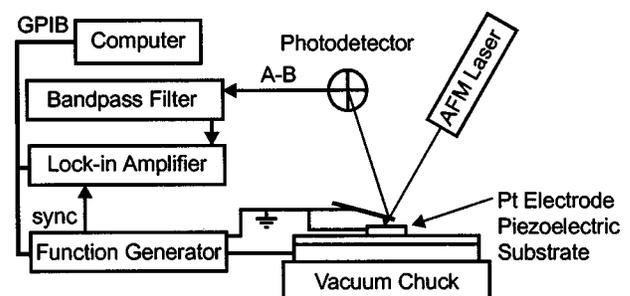


FIG. 1. Schematic of the AFM system showing the setup for piezoelectric measurements.

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TABLE I. Compilation of tip vibration constant measurements ($\text{\AA}/\text{V}$) of nonpiezoelectric, amorphous SiO_2 . A measurement labeled NL was at the noise level of our measurement system ($<0.004 \text{\AA}/\text{V}$). "In contact" measurements were performed with the tip in contact with the top electrode. "Tip as electrode" measurements were made with the tip serving as the top electrode, in direct contact with the SiO_2 surface. "Above electrode" measurements were performed with the tip $\sim 500 \text{\AA}$ above the electrode.

Measurement type	Silicon nitride tips force constant (N/m)				Grounded diamond tips force constant (N/m)			
	0.01	0.03	0.1	0.5	0.3	0.4	1.6	2.1
In contact	0.64	0.093	0.027	NL	NL	NL	NL	NL
Above electrode	29	5.2	1.2	0.13	NL	NL	NL	NL
Tip as electrode					0.063	0.018	0.009	NL

plied voltage plot. Commercially available Si_3N_4 (0.01, 0.03, 0.10, and 0.5 N/m) and conducting diamond tips (0.3, 0.4, 1.6, and 2.1 N/m) were used. The Si_3N_4 tips, which are commonly used for AFM, are electrically insulating. According to the supplier (Park Scientific), the conducting tips are prepared by deposition onto Si tips of 1000\AA of $0.1 \text{ }\Omega \text{ cm}$ p -type diamond. The conducting diamond tips are mounted on alumina squares and silver epoxy connects a wire to the chip. While other conducting tips are available, the diamond tips have been shown to be long lasting and suitable for scanning applications.^{11,12}

Measurements were performed on three types of samples: X-cut crystalline quartz ($10 \times 10 \times 0.25 \text{ mm}$) which serves as a piezoelectric standard, amorphous SiO_2 layers which do not show a piezoelectric response, and ZnO films which are commonly used in piezoelectric applications. Amorphous SiO_2 3000\AA thick was deposited by low pressure chemical vapor deposition on $0.01 \text{ }\Omega \text{ cm}$, n -type silicon substrates. Several $1 \text{ }\mu\text{m}$ thick, c -axis oriented ZnO films were deposited on $1 \text{ }\Omega \text{ cm}$ n -type silicon substrates by rf magnetron sputtering. Circular platinum electrodes $\sim 1000 \text{\AA}$ thick and $500 \text{ }\mu\text{m}$ in diameter were sputtered onto all samples. 5 mm diameter electrodes were deposited on some quartz crystals.

A direct method to compare the electrostatic interaction of different tip configurations is to drive the piezoelectric at frequency f , and measure the tip oscillation amplitude while the tip is above the electrode surface. With the tip out of contact with the electrode, the tip oscillation is due solely to the interaction with the electric field. These "above electrode" measurements of amorphous SiO_2 shown in Table I demonstrate that all of the Si_3N_4 tips interact with the electric field. Apparently, the Si_3N_4 tips held a surface charge which led to this large electrostatic interaction. Attempts at grounding the Si_3N_4 tips were unsuccessful, presumably due to the insulating character of the material. The tip oscillation amplitude was reduced by about a factor of 50 when measurements were performed in contact with the electrode; however, the tip-field interaction was still large enough to be a significant source of error for the low force constant tips. All grounded diamond tips showed no measurable interaction with the electric field when either above or in contact with the electrode. Measurements were also made with diamond tips used as the top electrode, in direct contact with the SiO_2 surface. In this case, the lower force constant diamond tips did measurably interact with the electric field.

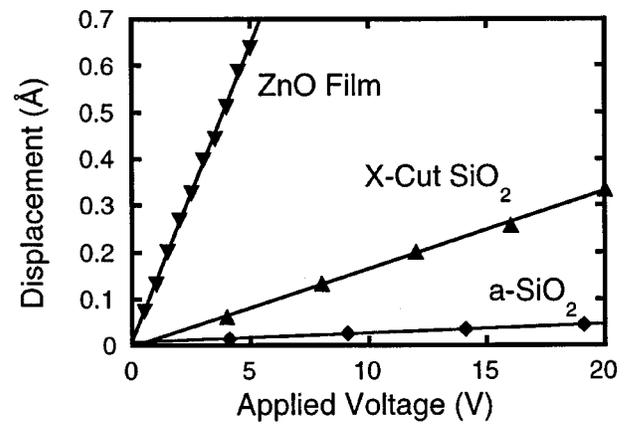


FIG. 2. Examples of piezoelectric measurements of thin film ZnO, X-cut quartz, and thin film amorphous SiO_2 . Piezoelectric constants for the measurements shown were $d_{33}=0.13 \text{\AA}/\text{V}$, $d_{11}=0.019 \text{\AA}/\text{V}$, and $d=0.002 \text{\AA}/\text{V}$, respectively. The amorphous SiO_2 measurement is at the experimental noise level ($<0.004 \text{\AA}/\text{V}$). The displacements and voltages are both peak-to-peak values.

Both the tip material and the force constant are important considerations in eliminating the tip-field interaction. For all the measurements in Table I that were above the measurement noise level, higher force constant tips interacted less with the electric field than lower force constant tips of the same material. The larger force constant tips, in general, require the use of a larger contact force. For a given tip, the measurements were independent of the contact force used. Therefore, the measurements indicate that the force constant is the parameter governing the size of the tip-field interaction, rather than the magnitude of the contact force applied during measurements. Both the 0.3 and 0.4 N/m diamond tips showed no measurable tip-field interaction even though they have smaller force constants than the 0.5 N/m Si_3N_4 tip which did interact with the electric field in the "above electrode" measurements. The measurements indicate that a conducting tip held at the same potential as the top electrode has a smaller tip-field interaction than an equivalent insulating tip. Therefore, conducting tips are the better choice for piezoelectric measurements. Also, the 0.5 N/m Si_3N_4 tips tended to fail (i.e., begin interacting with the electric field) after only a few measurements; whereas, the diamond tips withstand months of imaging and piezoelectric measurements.

Several example piezoelectric measurements using the conducting diamond tips are shown in Fig. 2. Before a piezoelectric measurement, an "above electrode" measurement is performed to verify that the tip is not interacting with the electric field. Measurement of several ZnO thin films yielded piezoelectric constants in the range of $0.02\text{--}0.13 \text{\AA}/\text{V}$. The measured values never exceeded the single crystal value (within experimental error) of $d_{33}=0.124 \text{\AA}/\text{V}$ for ZnO. Measurements on 5 mm electrodes on X-cut quartz yielded $0.014\text{--}0.019 \text{\AA}/\text{V}$, which is below the accepted value of $d_{11}=0.023 \text{\AA}/\text{V}$. Measurements performed at a single point over a period of an hour showed $<5\%$ variation. This level of stability was consistent for points of both high and low measured piezoelectric constants. Since the $\sim 1000 \text{\AA}$ radius AFM tip interacts with such a small area of the electrode, the measurements will be affected by microscopic

variations in piezoelectric film or electrode quality. For this reason, the measurements were position dependent.

Besides the tip-field interaction that has been minimized, another source of error is in measuring the constant for converting the photodiode voltage signal to a displacement. This calibration constant is measured by lowering the tip (with the AFM scanner) while the tip is in contact with the surface. The tip deflection is simultaneously measured with the photodiode. The scanner has been independently calibrated with a known step height, so the photodiode signal is calibrated to the known scanner displacement. One component of the conversion constant error is the due to the scanner calibration error. This error would result in a constant offset in our piezoelectric measurements. This error is estimated to be at most $\pm 10\%$. The conversion constant error when combined with the nearness of the X-cut quartz piezoelectric constant to our experimental noise level ($\sim 0.004 \text{ \AA/V}$) partially explains the low measured values for the X-cut quartz piezoelectric constant. Another potential source of error is discussed by Kholkine *et al.*² in reference to single beam interferometric measurements of piezoelectric samples bonded to a substrate. An implicit assumption in the measurement is that the displacement of the top electrode is equal to the entire piezoelectric displacement. Any compliance of the back electrode would reduce the measured piezoelectric constant. We are currently addressing these issues.

We have demonstrated that an AFM can be used to measure the magnitude of the piezoelectric effect. The interaction between the AFM tip and the electric field can be a major source of error in piezoelectric measurements. This tip-field interaction is greatly reduced when grounded con-

ducting diamond tips are used in piezoelectric measurements with the tip in contact with a top electrode, and the current is supplied to the electrode through an external probe. Measurements with the diamond tips yielded reasonable piezoelectric constants of X-cut single-crystal quartz, thin film ZnO, and nonpiezoelectric SiO₂ films.

The authors acknowledge Dr. Ajay Singh, Hoon Ham, and Jaehwan Oh of NCSU, David Dreifus and Linda Plano of Kobe EMC, and Dr. Enrico Colla of EPFL, Switzerland for helpful discussions. This research was supported by Kobe Steel U.S.A., Inc., Electronic Materials Center.

- ¹W. P. Mason and H. Jaffe, Proc. IRE, 921 (1954).
- ²A. L. Kholkine, C. Wuetchrich, D. V. Taylor, and N. Setter, Rev. Sci. Instrum. **67**, 1935 (1996).
- ³H. Birk, J. Glatz-Reichenbach, Li-Jie, E. Schreck, and K. Dransfield, J. Vac. Sci. Technol. B **9**, 1162 (1991).
- ⁴Lie-Jie, C. Baur, B. Koslowski, and K. Dransfield, Physica B **204**, 318 (1995).
- ⁵T. Hidaka, T. Maruyama, M. Saitoh, N. Mikoshiba, M. Shimizu, T. Shiosaki, L. A. Wills, R. Hiskes, S. A. Dicarolis, and J. Amano, Appl. Phys. Lett. **68**, 2358 (1996).
- ⁶G. Zavala, J. H. Fendler, and S. Trolier-McKinstry, J. Appl. Phys. **81**, 7480 (1997).
- ⁷P. Guethner and K. Dransfield, Appl. Phys. Lett. **61**, 1137 (1992).
- ⁸A. Gruverman, O. Auciello, and H. Tokumoto, J. Vac. Sci. Technol. B **14**, 602 (1996).
- ⁹E. L. Colla, S. Hong, D. V. Taylor, A. K. Tagantsev, N. Setter, and K. No, Appl. Phys. Lett. **72**, 2763 (1998).
- ¹⁰K. Franke, J. Besold, W. Haessler, and C. Seegebarth, Surf. Sci. Lett. **302**, L283 (1993).
- ¹¹P. Niedermann, W. Haenni, N. Blanc, R. Christoph, and J. Burger, J. Vac. Sci. Technol. A **14**, 1233 (1996).
- ¹²S. J. O'Shea, R. M. Atta, and M. E. Welland, Rev. Sci. Instrum. **66**, 2508 (1995).