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Application Note

Piezoelectric Characterization of Bulk and Thin Film Ferroelectric Materials using Fiber Optics

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Abstract

In this study, the use of a fiber optic technique for the measurement of the piezoelectric properties of ferroelectric bulk and thin film samples was investigated. The strain and piezoelectic properties (namely the d_{33} coefficients) were measured using the MTI-2000 Fotonic Sensor, which uses the principle of the optical lever to resolve very small changes in sample displacement (1 Å). Using this technique, we were able to detect the very small strains associated with the converse piezoelectic effect for PVDF films and bulk PZT samples, and correlate the results with data acquired from direct piezoelectric effect measurement. Comparison of the data sets prove that the optical lever would be a useful optical technique for measuring of the d_{33} values of ceramic thin films, such as BaTiOy ZnO, and PZT.

Introduction

Voigt's[1] analysis of the free energy of crystals showed that it is possible to measure the d_{33} of a crystal by two different static methods. The first uses the direct piezoelectic effect and is typically called the normal load method. In this technique, the change in the charge of a sample is measured as a function of the change in applied stress. The stress is applied by placing a load on a metallic tip oriented perpendicular to the film's surface. With the application of the stress, the sample strains and the equilibrium arrangement of the charged species is disturbed, resulting in the formation of a net charge across the material. This charge appears as a voltage drop across a reference capacitor, in parallel relation to the sample, whose capacitance value must be much larger than the sample in order to fulfill the free-field condition.[2] The voltage is measured using a multimeter or an electrometer and used to calculate the d_{33} of the material. Detailed descriptions and discussions of this technique can be found in papers by Lefki and Dormans[2] and K. No, et. al[3].

The other technique used to measure the d_{33} of materials, especially thin films, is the shrain induced method, which uses the indirect or converse piezoelectric effect In this method, measurements are made of the strain induced in a sample by the application of an external electric field perpendicular to the surface of the film. Although conceptually simple, measurement of the strain is difficult because the strains are on the order of angstroms.During the last 10 years or so, researchers have been studying and using optical techniques, interferometry in particular, to measure the small strains.[13] The appeal of interferometric systems is their ability to resolve very small changes in displacement (10^{-3} Å).[8] This is done by comparing the interference light intensity of a reference laser beam to that of a laser beam striking the surface of the sample before and after strain occurs.[9] Thorough discussions of the theoretical aspects of the use of interferometry to measure thin film strains can be found in [8], [9], and [12].

Although interferomety is a very useful technique for measuring the d_{33} of thin films, it is not the only high resolution optical method that can be used to measure such small strains. Optical levers can also measure small changes in displacement An optical lever is a noninterferometric device, where the power of a light beam striking a vibrating surface is modulated in such a way that the power of the reflected beam is proportional to the displacement of the surface.[14] An extremely useful rendition of this concept uses step-index fiber optics, where transmission fibers illuminate a surface and receiving fibers transmit the reflected light to a photocell. Developed by Kissinger[15],the displacement sensitivity of this technique is a result of the differential relationship between the distance separating the sample and the fibers, and the amount of light received and transmitted to a photocell. Changes in displacement cause the photocell to "see" an amplitude oscillation, in reflected light, which can be converted into a displacement change.[14] As a result of ik high sensitivity, versatility, and simplicity, the fiber optic based optical lever has found uses in a wide variety of applications, such as welding, electronics, acoustics, and biomechanics.[14,16,17].

This paper discuss our preliminary results acquired from a fiber optic based optical lever system, which uses the MTI-2000 Fotonic Sensor, to measure the d_{33} of bulk and film samples.

Experiment

The piezoelectric coefficients (d_{33}) of the bulk and film samples were measured using the MTI-2000 Fotonic Sensor and 2032RX High Resolution Module, which are manufactured by MTI Instruments Inc. of Albany, NY. The system is a modular design, where the base unit (the MTI-2000) houses the analysis electronics and communications interfaces and two bays for removable fiber optic probe modules. Because the probe uses a fiber optic based, optical lever to determine the displacement change, it is important to calibrate the equipment for each sample so that sample-sample changes in reflectivity can be taken into account and eliminated.

Calibration is accomplished by placing the sample under the probe as shown in Figure 1a. By adjusting the distance between the fibers in the probe and the sample, it is possible to find the distance at which a receiving fiber is completely illuminated by reflected light from a neighboring transmitting fiber. This defines the optical peak which is the point where the photocell receives the maximum subtended power from the source (a tungsten lamp). The equipment then autocalibrates itself with respect to source power output to develop a calibration curve for received light power vs. displacement, as shown in Figure 1b. The front slope (range 1) of the calibration curve is very steep and exhibits linear behavior to within ±1% in the middle region. This means that very small changes in displacement appear as large linear changes in received light. In Figure 1b, the front slope has a value of 54.10 A/mV.

Figure 1a: Schematic of the displacement sensing mechanism for the fiber optic based optical lever

(Courtesy of Mechanical Technology Inc.)[18]

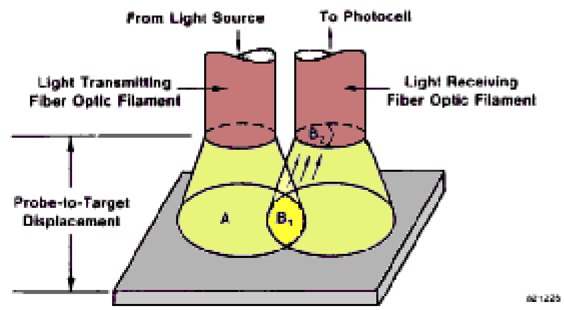

Fig. 2 Target Moving Away from Probe – Reflected Light Intensity Increases

Figure 1b: Calibration curve for a fiber optic based optical lever

(Courtesy of Mechanical Technology Inc.)[18]

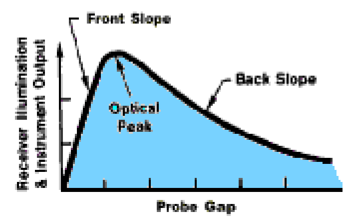

Fig. 3 Typical Fiber Optic Sensor Calibration Curve

Once calibrated, the 2032RX module is placed into high resolution mode, which increases the light output of the source and the probe resolution by a factor of ten, producing a front slope resolution of 5.410 Å/mV over a ±1% linear range of approximately 2.25 μm The resolution limit of the 2032RX probe is 2.5 Å without external filtering over a frequency range from dc to 150 kHz. External filtering and signal processing can improve the resolution to 1 Å or less over the same frequency range.[16]

The voltage output of the probe's photocell can be monitored with a digital oscilloscope by using the MTI-2000's analog output By setting up the scope to trigger upon application of the external voltage, the change in voltage caused by sample displacement can be captured, viewed, and measured directly on scope's screen. An HP 33120A function generator was used to generate 250 mHz unipolar (+) square wave pulses and a Tektronix 320 digital oscilloscope was used for signal acquisition. Figure 2 shows a typical output waveform from the MTI-2000.

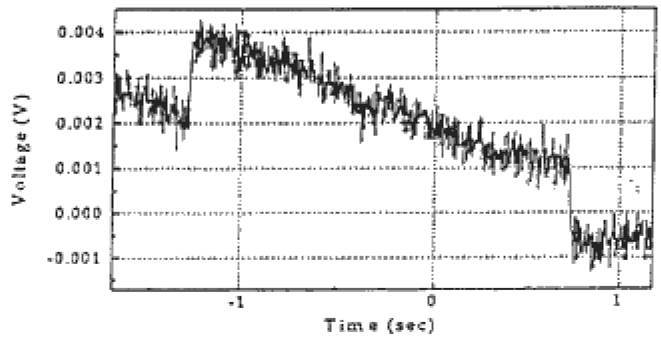

Figure 2: Typical signal acquired from the MTI-2000 (2 V p-p applied)

Multiplying the magnitude of the step change-in voltage detected by the oscilloscope by the probe's front slope factor (5.410 A/mV) gives the change in displacement of the sample when the voltage was applied. with the application of a unipolar pulse, the measured displacement is equal to the change in thickness of the sample, and the piezoelectric coefficient is calculated by dividing the thickness change by the applied voltage.

The effect of frequency on the d_{33} of a sample (from 10 Hz to 60 kHz) was measured by placing the MTI-2000 into wide band mode (no low pass filtering) and setting up the digital oscilloscope to trigger on the function generator's bipolar sinusoidal pulse. This configuration allowed the scope to see a definitive trigger voltage, instead of relying on a seemingly random, noisy signal for the trigger. Using digital averaging, the background noise from the MTI-2000's signal was removed, leaving only the displacement pulse.

Results and Discussion

To verify that this technique is useful for measuring the very small strains induced in piezoelechic materials by the indirect effect, the MTI-2000 and 2032 RX module was used to characterize the d_{33}'s of two bulk PZT samples and a piece of PVDF film. The bulk samples, labeled 1A and 2B, were purchased from Channel Industries of San Barbara, CA. Each sample was 38 mm in diameter and 7 mm in thickness and had a proprietary composition. The d_{33}'s of the samples, determined by a Channel Industries with a Berlincourt dynamic d_{33} tester, were 554 and 565 pm/V, respectively. Figure 3 shows the results of the MTI Instruments experiments for the bulk PZT samples. The horizontal lines on the figure represent the average of the points for each sample. For the 2B sample, the average measured indirect d_{33} was 570±19 pm/V over the applied field range of 1-25 V/cm, which matches the Berlincourt measurement well. This d_{33} value corresponds to measured strains ranging from 5-85 Å, depending on the applied field. For verification, we tested the sample in the same locations with a direct piezoelectric effect tester. The direct d_{33} value was found to be 560±17 pC/N.

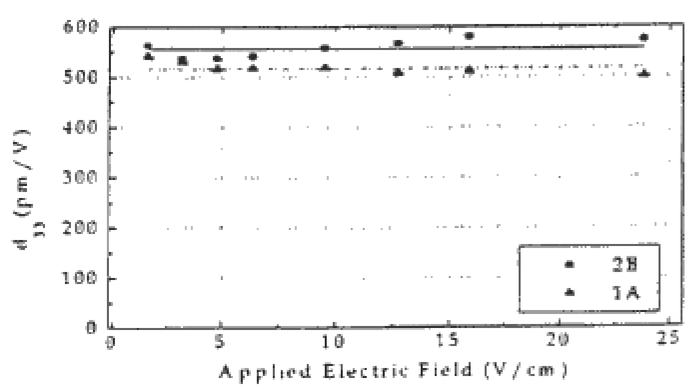

Figure 3: d_{33} vs. Applied field for bulk samples 1A and 2B

The average indirect d_{33} for sample 1A was 520±8 pm/V, well below the value of 554 pm/V obtained using a Berlincourt tester. The explanation for why the MTI-2000 measured d_{33},values for 1A are below the reported Berlincourt value is due to the electrode configuration (see Figure 4).

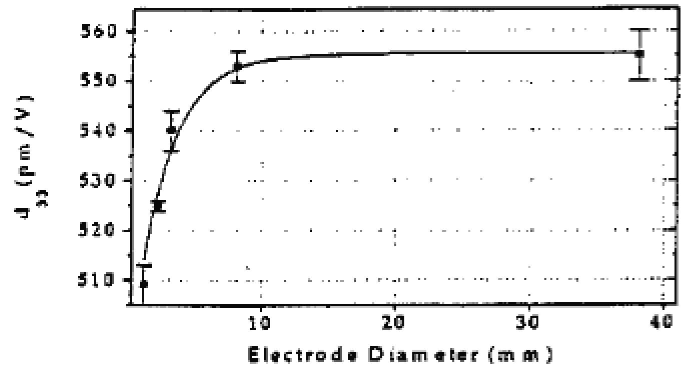

Figure 4: Effect of electrode diameter on the indirect d_{33} of sample 1A

For 2B and the Berlincourt tests, the samples had silver electrodes that completely covered the top and bottom faces. To test how the MTI-2000 would behave under more film-like electrode conditions, we polished off the top electrode of the 1A sample and used a mask overlay to deposit 1, 2, 3, and 7 mm Pt electrodes onto the PZT surface. The results mirror those seen in PZT films, where the measured indirect d_{33} of the sample is dependent on the top electrode diameter,[11, 12] The d_{33} measured by the direct piezoelectric effect was found to be 554 ±19 pC/N.

Figure 5 shows the results of testing a polyvinylidene fluoride (PVDF) film on the MTI-2000. Although the exact d_{33} value of the PVDF film is not known, the literature shows that the expected d_{33} value for PVDF is within 15-30 pm/V.[19,20] Therefore, the average measured value of 22±0.7 pm/V is well within the expected range. Direct piezoelectic tests showed the d_{33} to be 19±2 pC/N, which compare well with the MTI-2000 results.

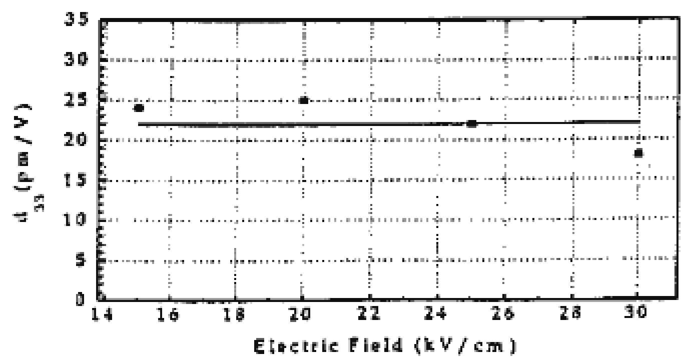

Figure 5: d_{33} vs. Applied field for PVDF film

Figure 6 shows the frequency dependence of the d_{33} of sample 1A. under an applied field of 7 V/cm, for a frequency range of 10 Hz to 60 kHz. This gradual decrease in d_{33} with an increase in frequency has been reported by Li et al[13] and shows that the MTI-2000 is working properly within this frequency range.

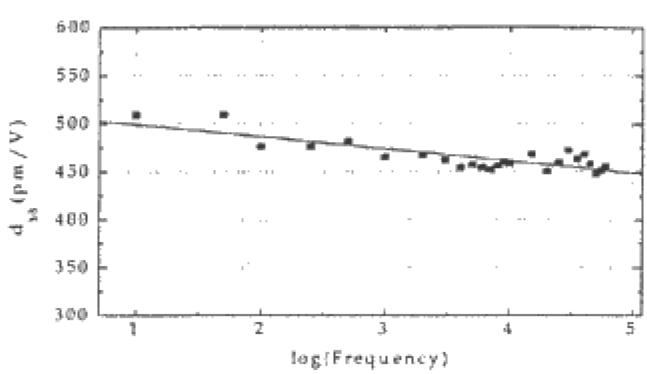

Figure 6: d_{33} vs. frequency for sample 1A

Conclusions

Based on our preliminary work, the MTI-2000 Fotonic Sensor, a fiber optic based optical lever instrument, does an excellent job in measuring the indirect piezoelectric behavior of a material. The high displacement sensitivity of a fiber optic lever is a result of the differential relationship between the distance separating the sample and the fibers, and the amount of light received and transmitted to the photocell. Changes in sample displacement cause the photocell to "see" an amplitude oscillation in reflected light, which can be converted, by using the probe's slope factor, into a relative displacement change. Using this technique, the d_{33} of two bulk PZT samples and a PVDF film were measured using the MTI-2000. These results compare well with d_{33} values measured using the normal load technique (direct piezoelectric effect).

Acknowledgements

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