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# **Preparation of Thin Film Polyvinylidene Fluoride Shock Wave Pressure Transducers**

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U.S. DEPARTMENT OF COMMERCE  
National Bureau of Standards  
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Annual Report  
Contract No. N6092187WRW0082  
J. W. Forbes, Contract Officer

Prepared for:  
**Naval Surface Weapons Center  
White Oak  
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**U.S. DEPARTMENT OF COMMERCE, C. William Verity, *Secretary***  
**NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director***



## ABSTRACT

Thin film polyvinylidene fluoride transducers have been prepared by a poling procedure which involves the repeated cycling of the applied electric field over the ferroelectric hysteresis curve. Using this procedure, it is possible to prepare a uniformly poled transducer and to establish, with precision, the final remanent polarization. The remanent or permanent polarization is calculated by measuring the total poling charge and subtracting from it that which arises from the resistance and capacitance of the specimen. By this method we were able to prepare ten gages with the same remanent polarization with a precision of 0.5%. X-ray observations and measurements of density on an unpoled specimen show that the film is biaxially oriented, contains both  $\alpha$  and  $\beta$  phase crystals, and is 50% crystalline. A poled specimen with a remanent polarization of  $5.7 \mu\text{C}/\text{cm}^2$  showed a decrease in the  $\alpha$  phase content with a corresponding increase in the  $\beta$  phase, but the overall crystallinity remained unchanged. We observe that polarization saturation is a function of the maximum poling field and it is suggested that the existence of space charge at the surface of the polar crystals presents an opposing local field to the crystallites and moderates the ferroelectric switching over a wide range of applied fields.

## INTRODUCTION

The procedure which we have used to carry out the poling of polyvinylidene fluoride (PVDF) thin film gages is based on work performed by Bauer.<sup>1</sup> This technique involves increasing the applied electric field in small increments, and, at each field level cycling many times over the ferroelectric hysteresis curve. In so doing each electrode spends approximately equal time in the positive and negative state thereby producing a symmetric distribution of space charge in the specimen. Since space charge affects the magnitude of the internal electric field, a symmetric distribution of charge will result in a symmetric distribution of permanently oriented dipoles. The symmetry of permanent polarization in specimens poled by this procedure has been observed by carrying out thermal pulse experiments.<sup>2</sup> In these experiments, 26  $\mu\text{m}$  thick poled specimens were observed to have uniform polarization throughout the interior 16  $\mu\text{m}$  but to possess a slight decrease in polarization for the 5  $\mu\text{m}$  distance nearest each surface.<sup>2</sup> Uniformity of the polarization is a particularly important criterion for PVDF shock gages because a shock wave, while traversing the thickness of the film, will produce an electric charge vs time profile which mirrors the polarization vs thickness profile in the gage.

Another advantage of using the Bauer poling technique is that the final remanent polarization of the gage can be precisely controlled. This control is achieved by calculating the remanent polarization,  $P_r$ , in real-time and adjusting the field in small increments to produce a gage with the desired value of remanent polarization. The calculation of  $P_r$  involves correcting the observed poling charge by subtracting from it that which is due to ohmic conductivity and that which is associated with the capacitance of the PVDF thin

film. The data which we present here will show that we have been able to fabricate shock gages whose remanent polarization was controlled within a precision of 0.5%.

## EXPERIMENTAL APPARATUS AND PROCEDURE

The PVDF Film The PVDF film which we used for our experiments was manufactured by Rhone-Poulenc,<sup>3</sup> and was obtained from a selected master batch which is kept at Sandia National Laboratories by R. Graham.<sup>4</sup> The thickness of this film is 26  $\mu\text{m}$ . Our x-ray observations and measurements of density indicate that the material is biaxially oriented, contains both nonpolar  $\alpha$  and polar  $\beta$  crystalline phases, and has a crystallinity of 50%. The as-received material has a density at room temperature of 1.8060 gm/cm<sup>3</sup>.

In cooperation with J. Forbes and D. Tasker of the Naval Surface Weapons Center<sup>5</sup>, an electrode pattern was established which minimizes the mutual inductance (and resultant ringing effects at high frequencies) of the leads to the gage sensitive area. This pattern is shown in Figure 1 and was deposited on the film using a vacuum deposition technique in two steps. First, chromium was deposited on the film, and second, gold was deposited on top of the chromium. The thickness of the chromium layer was 20 to 30 nm and the thickness of the gold layer was approximately 100 nm. The central circular area, which is 0.5 cm in diameter, is the sensitive gage area.

The Poling Procedure Poling of the PVDF film is carried out at room temperature with the film immersed in fluorinert, a dielectric fluid from the

3M Co..<sup>3</sup> During poling, a pressure of  $3.1 \times 10^6$  Pa (450 psi) is applied to the central area of the film by a hydraulic ram. The purpose of the applied normal force is to maintain the smoothness of the film surface which will wrinkle if left unconstrained. A cross section of the poling cell is shown in Figure 2.

Poling commences at a low applied field, 1.15 MV/cm (3000 V for these gages). Using a bipolar power supply, the field is cycled sinusoidally between + and - 1.15 MV/cm at a frequency of 0.05 Hz. After one hour of cycling, the field is increased by 0.4 MV/cm and cycling continues for an hour at this new field level. This procedure continues up to 2.75 MV/cm after which the incremental increase is lowered to 0.2 MV/cm. The current vs field hysteresis curve is monitored using a programmable digital oscilloscope. The corrected hysteresis curve and remanent polarization are obtained using software which subtracts the current due to ohmic conductivity and specimen capacitance from the observed total current. (Details regarding the current correction calculation are contained in the Appendix.) As the desired remanent polarization is approached, a linear extrapolation is used to determine the field increment which will yield the specified value of  $P_r$ . The absolute error in the measurement of  $Q_r$ , the remanent polarization charge, is estimated to be  $\pm 3\%$ , but owing to the difficulty in calculating the effective area of the electrode, the error in the measurement of  $P_r$  is estimated to be  $\pm 10\%$ . The relative precision with which  $P_r$  can be obtained from gage to gage is discussed below.

Density Measurements These experiments were carried out using the fluid displacement method for which the displacement fluid was water at 23°C. The weighing balance was a Cahn Electrobalance whose output was observed on a strip

chart recorder.<sup>3</sup> The weight of the sample in the fluid was observed to reach equilibrium in approximately 2 hours. The poled samples, for which we made density measurements, did not have the same electrode configuration as the gages but had rectangular aluminum electrodes which were 1 cm x 6 cm. When poling was complete, the aluminum electrodes were removed by placing the specimen in a solution of sodium hydroxide. This specimen was then used for both density and x-ray measurements. The uncertainty in the density measurement is estimated to be  $\pm 0.0005 \text{ gm/cm}^3$ .

X-ray Measurements Wide angle x-ray measurements were made using  $\text{CuK}\alpha$  radiation. X-ray patterns were recorded in transmission so that the (001) reflections of the  $\beta$  phase and the (002) reflections of the  $\alpha$  phase could be observed. Scans of the reflected x-ray intensity were made over a range of  $2\theta$  from 30 to  $46^\circ$ . The ratio of intensity of the  $\beta$  (001) reflection to the  $\alpha$  (002) reflection, weighted by their relative structure factors, was used to calculate the relative amounts of  $\alpha$  and  $\beta$  phase in the specimen.<sup>6</sup> Because these films are biaxially oriented in the manufacturing process, the  $2\theta$  scans were made for orientations of the specimen at 0, 45, and  $90^\circ$  with respect to the plane of the incident and reflected x-ray beam. An average intensity ratio was calculated from these three observations. The uncertainty in the measurement of the defined average intensity ratio is estimated to be  $\mp 10 \%$ .

Calculation of Specimen Crystallinity The crystallinity of the specimen was calculated by combining the x-ray and density data. If we let  $f_\alpha$ ,  $f_\beta$ , and  $f_A$  be the volume fraction of  $\alpha$ ,  $\beta$ , and amorphous phases in the specimen, and if we define  $k$  to be the ratio of the weighted x-ray intensities of the  $\beta$  and  $\alpha$

phases, then it follows that

$$f_{\alpha} + f_{\beta} + f_A = 1,$$

$$k = SI_{\beta}/I_{\alpha} = f_{\beta}/f_{\alpha},$$

$$f_{\alpha}v_{\alpha} + f_{\beta}v_{\beta} + f_A v_A = v_s,$$

where  $S$  is the relative structure factor of the  $\beta(001)$  and the  $\alpha(002)$  reflections,  $v_{\alpha}$ ,  $v_{\beta}$ , and  $v_A$  are the specific volumes of the  $\alpha$ ,  $\beta$ , and amorphous phases;  $v_s$  is the measured specific volume of the specimen (the reciprocal of the density). From these equations we have

$$f_A = \frac{v_s(k+1) - v_{\alpha} - kv_{\beta}}{v_A(k+1) - v_{\alpha} - kv_{\beta}}, \quad (1)$$

$$f_{\alpha} = \frac{v_A - v_s}{v_A(k+1) - v_{\alpha} - kv_{\beta}}, \quad (2)$$

$$f_{\beta} = kf_{\alpha}. \quad (3)$$

Using  $v_{\alpha} = 0.5197 \text{ cm}^3/\text{gm}$ ,  $v_{\beta} = 0.5074 \text{ cm}^3/\text{gm}$ , and  $v_A = 0.5980 \text{ cm}^3/\text{gm}$ , room temperature values for these quantities,<sup>7</sup> and  $S = 3$ ,<sup>6</sup> we are able to calculate the volume fraction of each phase if  $v_s$  is known.

The crystallinity,  $\chi$ , is defined as  $\chi = f_{\alpha} + f_{\beta}$  or  $\chi = 1 - f_A$ . We note from equation (1) that  $\chi$  is relatively insensitive to changes in  $k$  and we estimate the uncertainty in our measurement of  $\chi$  to be  $\pm 0.01$ .

## RESULTS AND DISCUSSION

Polarization Measurements In Figure 3, we show uncorrected current vs poling

field hysteresis curves which were obtained for four maximum poling fields in the range 1.10 to 2.70 MV/cm. The electric field was varied sinusoidally at 0.05 Hz for each curve, and each is a single cycle representative of the approximately 180 cycles made at each maximum field level. The peak in the current for each cycle occurs during ferroelectric switching. The total current is the sum of the displacement plus ohmic currents. Thus, in terms of the current density,

$$j = j_D + j_\rho = \epsilon dE/dt + dP/dt + E/\rho, \quad (4)$$

where  $\rho$  is the resistivity,  $\epsilon$  is the dielectric permittivity,  $E$  is the electric field, and  $P$  is the polarization. In order to obtain  $dP/dt$ , corrections are made by subtracting from the measured  $j$  a quantity equal to  $\epsilon dE/dt + E/\rho$ . In a hysteresis plot of  $j$  vs  $E$ , the effect of the ohmic current is to rotate the curve counterclockwise about the origin, and the effect of the capacitance current is to add to  $j$  in accordance with  $dE/dt$ . The software which we developed to make the corrections contains the following criteria: (a) the slope of the current vs field curve equals zero when  $P(E) = \text{constant}$ , i.e. at saturation; and (b) the value of the current equals zero for  $P(E) = \text{constant}$ .

A typical corrected current curve is shown in Figure 4. Upon integration of the current hysteresis curve, we obtain the  $Q_p(E)$ , the polarization charge hysteresis curve of Figure 5 from which we calculate a remanent polarization of  $6.00 \mu\text{C}/\text{cm}^2$ .

The specified remanent polarization for the group of ten gages which we

prepared was  $6.00 \mu\text{C}/\text{cm}^2$ . In order to realize the value of the poling field which will yield the target remanent polarization for each gage, we made a linear extrapolation of the remanent polarization vs poling field data from lower values of  $P_r$  to  $6.00 \mu\text{C}/\text{cm}^2$ . The results, shown in Table I, indicate the precision with which we are able to control the remanent polarization, in this case with a standard deviation of  $\pm 0.5\%$ .

TABLE I  
Remanent Polarization and Poling Field Measurements

<u>Gage</u>	<u><math>P_r</math> <math>\mu\text{C}/\text{cm}^2</math></u>	<u>E MV/cm</u>
1	6.00	2.78
2	5.98	2.77
3	6.00	2.84
4	5.99	2.79
9	5.98	2.21
10	6.00	2.77
11	6.09	3.19
12	5.98	2.77
13	5.99	2.85
14	5.99	2.70
Average	$6.00 \pm 0.5\%$	$2.77 \pm 8\%$

In Table I, we also note that the maximum poling field, which is necessary to achieve a polarization of  $6.00 \mu\text{C}/\text{cm}^2$ , has a standard deviation much larger than that for the remanent polarization. We attribute this to the presence of different amounts of internal space charge within each gage. We view the space charge as residing at the surface of the polar crystallites and presenting an internal field to the crystallite which opposes or diminishes the effect of the applied poling field. Considering that a distribution of space charge exists in each specimen, it follows that there is a distribution of local field

strengths. As a consequence, the poling field which is necessary for achieving a given remanent polarization will be a function of the particular space charge distribution within each gage. Since the space charge is an uncontrolled parameter during the processing of this film, it is not possible to prepare gages with identical remanent polarization simply by subjecting them to the same maximum poling field.

Another observation which we attribute to the presence of space charge is the dependence of the saturation polarization on the maximum poling field. As implied by the definition of saturation, one would expect that the saturation value of the polarization would remain constant as the maximum poling field increases. But for PVDF, this is not the case. As we increment the maximum poling field to a higher level, the saturation polarization also increases. Two phenomena occur on the molecular scale during poling: (a) dipoles within the  $\beta$  phase crystallites orient in the direction of the applied field if they experienced a field equal to or greater than the coercive field for PVDF (approximately 1.0 MV/cm), and (b) the  $\alpha$  phase crystallites are converted to a polar  $\alpha$  phase at fields near 1.0 MV/cm and subsequently to  $\beta$  phase crystals at higher field strengths.<sup>8</sup> As the poling field increases, the opposing effect of the space charge is overcome and more crystallites participate in the ferroelectric switching. Those crystallites which remain unswitched or unconverted to the polar  $\alpha$  and  $\beta$  phases at a given poling field will remain so until the field reaches the appropriate value.

We have made repeated attempts to prepare gages with remanent polarization greater than  $8 \mu\text{C}/\text{cm}^2$ . These attempts have all met with failure because of

electrical breakdown which occurs at poling fields in the range from 3.0 to 3.5 MV/cm. Seven out of eight gages failed at the edge of the electrode indicating that the electrode design and/or the electrode material are the source of the breakdown problem. Fabrication of gages using different electrode materials and employing sputtering rather than vacuum deposition may improve the breakdown strength of this design.

X-ray Measurements In Figures 6 and 7, scans of the x-ray intensity vs  $2\theta$  are shown for three orientations of the control (unpoled) specimen and for a poled specimen whose remanent polarization was  $5.7 \mu\text{m}/\text{cm}^2$ . At zero degree orientation, the ratio of the intensity of the  $\beta$  (001) reflection to the  $\alpha$  (002) reflection is the largest of the three orientations. This indicates that, during processing, the principal stretching and extension of the film has occurred in a direction coincident with zero degrees. Such an interpretation is based on the well known observation that the  $\beta$  phase is formed by extensional strain.<sup>9</sup>

The largest difference in the x-ray data obtained from poled and unpoled specimens is seen at the  $90^\circ$  orientation. Here, a significant increase in the  $\beta$  phase intensity at the expense of the  $\alpha$  phase intensity has occurred for the poled specimen. Thus, the poling field for this specimen, 2.6 MV/cm, was large enough to produce some conversion from the  $\alpha$  to the  $\beta$  phase. It is interesting that, at zero degrees, there is no difference in the x-ray scan between the poled and unpoled specimens. The reason for the lack of conversion from the  $\alpha$  phase to  $\beta$  phase along this direction is unclear.

From the x-ray measurements we calculate a value of  $k$  by taking a weighted average of the  $k$  values obtained from the three orientations, i.e.

$\langle k \rangle = 0.25k_0 + 0.5k_{45} + 0.25k_{90}$ . This average  $k$ , shown in Table II, is used in equations (1) through (3) to calculate the volume fractions of the  $\alpha$ ,  $\beta$ , and amorphous phases.

TABLE II

Density and X-Ray Results

<u>Specimen</u>	<u>Density g/cm<sup>3</sup></u>	<u><math>\langle k \rangle</math></u>	<u><math>f_\alpha</math></u>	<u><math>f_\beta</math></u>	<u><math>f_A</math></u>	<u><math>\chi</math></u>
Control	1.8060	3.5	11%	39%	50%	50%
$P_r = 5.7 \mu C/cm^2$	1.8099	5.3	8%	43%	49%	51%

Density Measurements The results of the density and x-ray measurements on two specimens are shown in Table II. The increase in density with increasing remanent polarization is associated with the conversion of the lower density  $\alpha$  phase crystals to higher density  $\beta$  phase crystals. Within the limits of experimental uncertainty, we observe that the crystallinity remains constant.

SUMMARY AND CONCLUSIONS

By using the Bauer polarization procedure, we have been able to prepare ten gages with a remanent polarization of  $6.00 \mu C/cm^2$  to a precision of 0.5%. From density and x-ray measurements we calculate that the crystallinity remains constant during poling but there is a significant conversion of  $\alpha$  phase crystallites to the polar  $\beta$  phase. The observation that each gage requires a unique maximum poling field to achieve the same remanent polarization is attributed to the presence of different distributions and amounts of space

charge in each gage. The observed increase in the saturation polarization with increase in the maximum poling field is also attributed to the effect of space charge which resides at the surface of polar crystals and creates a local electric field which diminishes the effect of the applied poling field. Our attempts to increase the remanent polarization to values greater than  $8.0 \mu\text{C}/\text{cm}^2$  were not successful because electrical breakdown occurred at the edges of the electrodes before reaching this level of polarization.

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3. Identification of a commercial product is made to facilitate experimental reproducibility and does not imply endorsement by NBS.
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## APPENDIX

### Data Analysis

The poling voltage is in the form of a high voltage sine wave and is applied across the dielectric PVDF film at a frequency of 0.05Hz. The measured variable is the current through the dielectric. A signal proportional to the high voltage and a signal from a current amplifier are fed into a programmable digitizing waveform recorder. Both signals are digitized with 12-bit resolution. Fifty seconds of data are collected at a sampling rate of 20 samples/second (1024 data points). This results in an effective sampling of approximately 400 data points per cycle.

Figure 3 shows the recorded raw data. The following is a summary of the analysis program:

1. Both waveforms are multiplied by the appropriate proportionality constants to give applied voltage (V) and measured total current ( $\mu\text{A}$ ).
2. By dividing the applied voltage by the sample thickness (entered as a constant), the applied field, E, is computed (V/cm). A smoothing of the digitized data is performed (a 10 point smoothing) which should not result in significant degradation due to the sinusoidal nature of the field.
3. A complete cycle is chosen for analysis.
4. The resistive component of the current is calculated and subtracted from the total current,  $I_T$ . The resistive term is calculated by drawing a straight line between  $I_T(E=E_{\text{max}})$  and  $I_T(E=E_{\text{min}})$  and storing the value of the slope. The field is multiplied by the slope to give the resistive current, which is then subtracted from the total current.
5. Next, the capacitive component of the current is calculated and subtracted from the total current. To do this, we subtract an elliptically shaped current vs field function (see reference 1) so that  $dI/dE = 0$  at  $E_{\text{max}}$  and  $E_{\text{min}}$ . The resultant current is the polarization current,  $I_p$ .
6. To calculate the remanent polarization, the  $I_p$  curve is integrated to give the polarization charge  $Q_p$  as shown in Figure 5. The remanent polarization expressed as charge per unit area is:  $P_r = Q_p(E=0)/A_e$ , where  $A_e$  is the area of the electrode.

From this analysis we can obtain a value of the resistivity and dielectric constant of PVDF. Typical values are  $\rho = 6.0 \times 10^{12}$  ohm-cm and  $k = \epsilon/\epsilon_0 = 17.5$  at 0.05 Hz and room temperature.



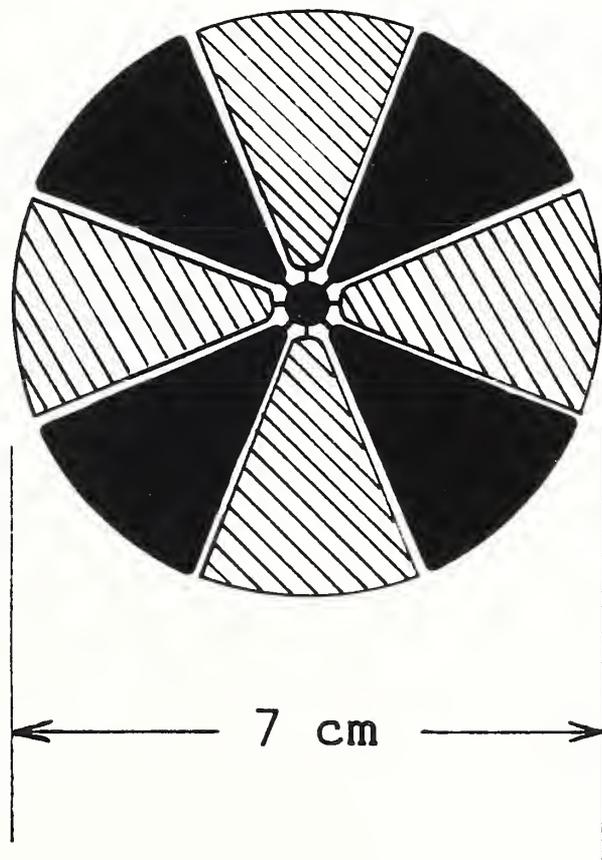


Figure 1. The electrode pattern for the shock wave pressure gages is shown. The central circle in the pattern has a diameter of 0.5 cm and is the sensitive area of the gage. The dark and shaded regions emanating from the central area are the electrical leads on opposite sides of the clear thin film.

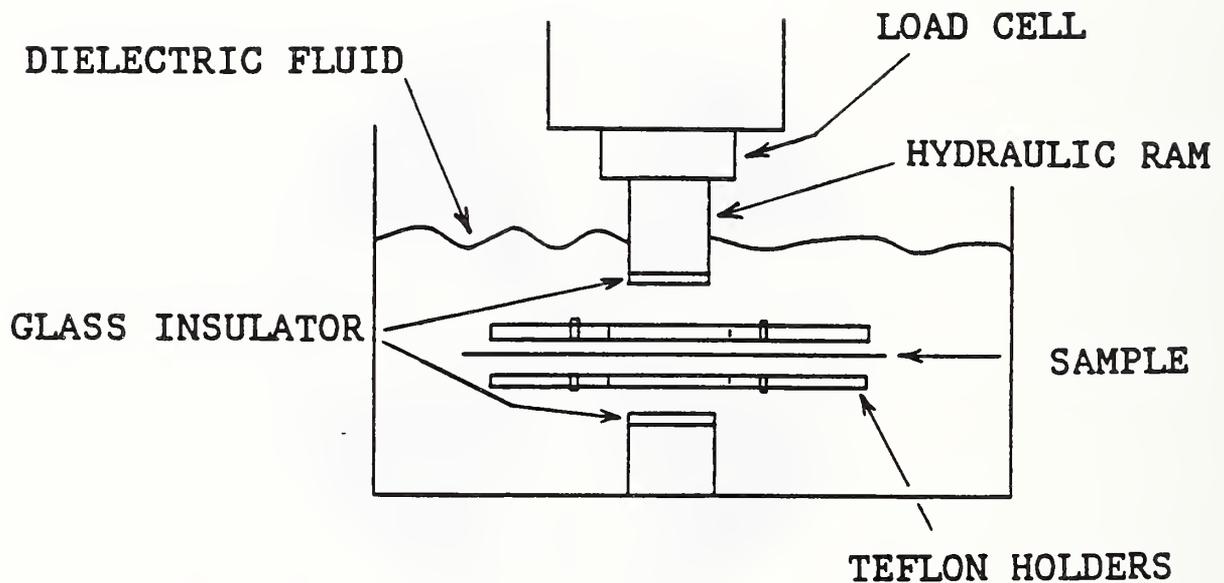


Figure 2. A cross sectional diagram of the poling cell is shown. The flat glass insulators impinge upon the face of the gage through circular openings in the teflon holders; they transmit the normal force of the hydraulic ram to the sensitive area of the gage. The teflon holders support the PVDF film and electrical leads.

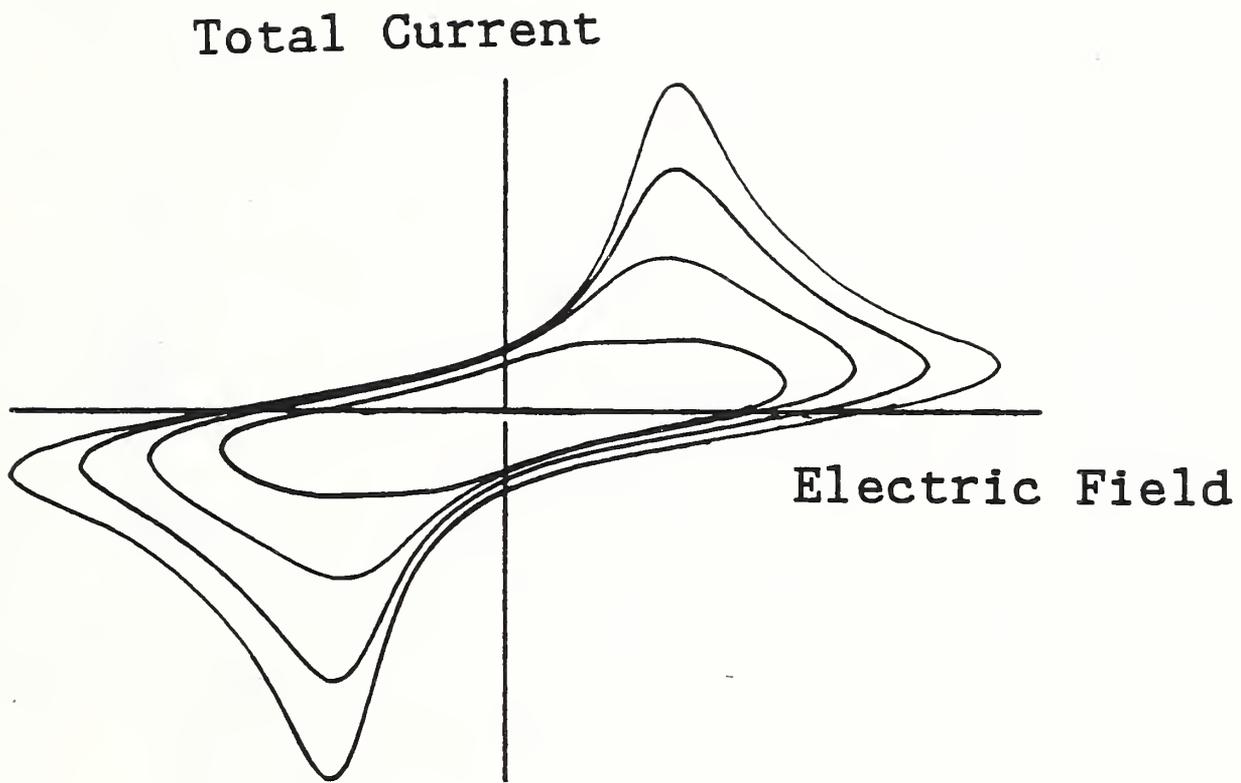


Figure 3. Typical uncorrected current hysteresis curves are shown. The maximum poling fields for these curves are 1.54, 1.92, 2.31, and 2.69 MV/cm. The peak current for the curve with  $E_{\text{max}} = 2.69$  MV/cm is approximately  $2.5 \mu\text{A}$ .

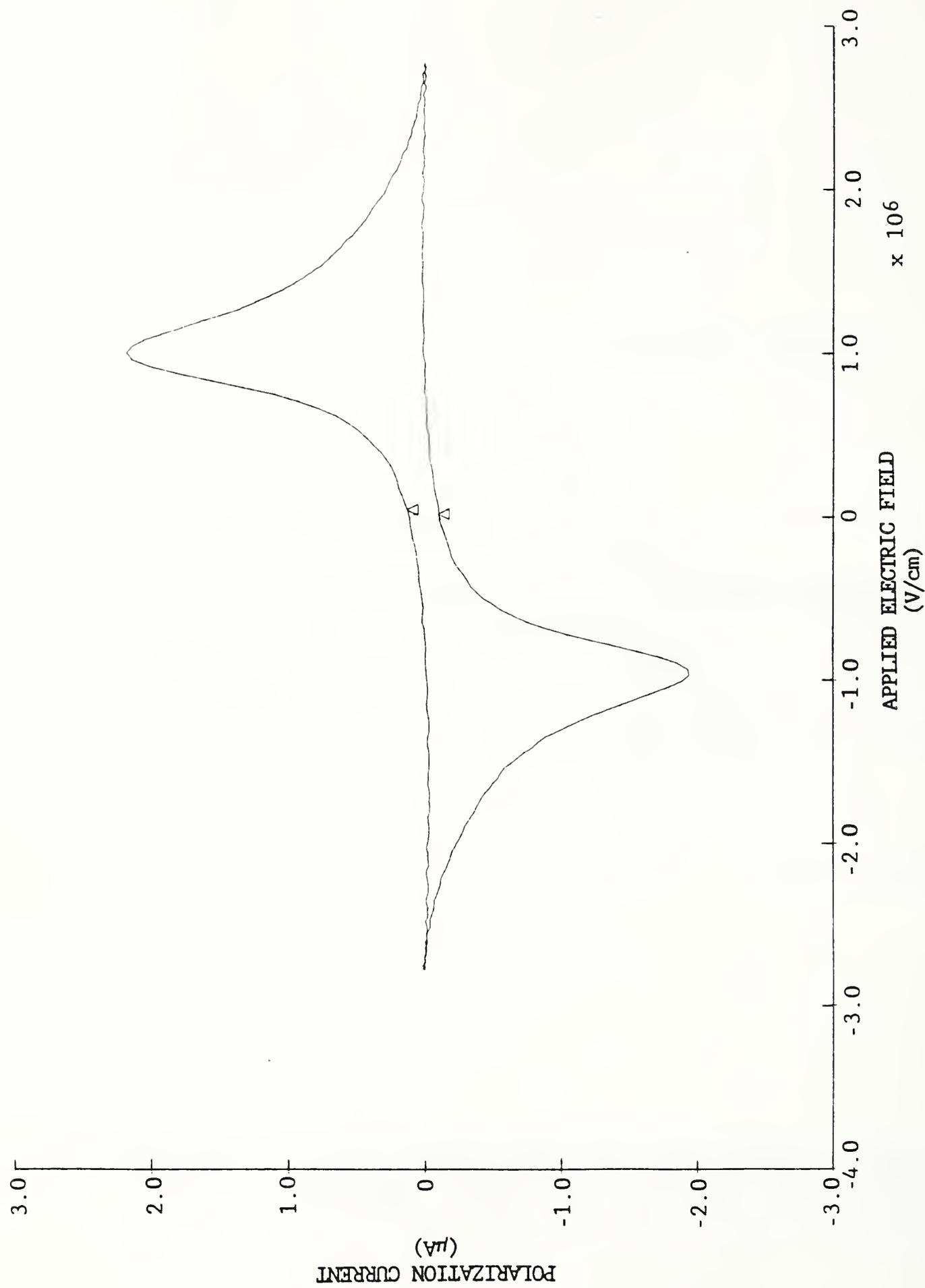


Figure 4. The polarization current hysteresis curve is shown for gage # 14. The polarization current was obtained by subtracting the resistive and capacitive currents from the total charging current.

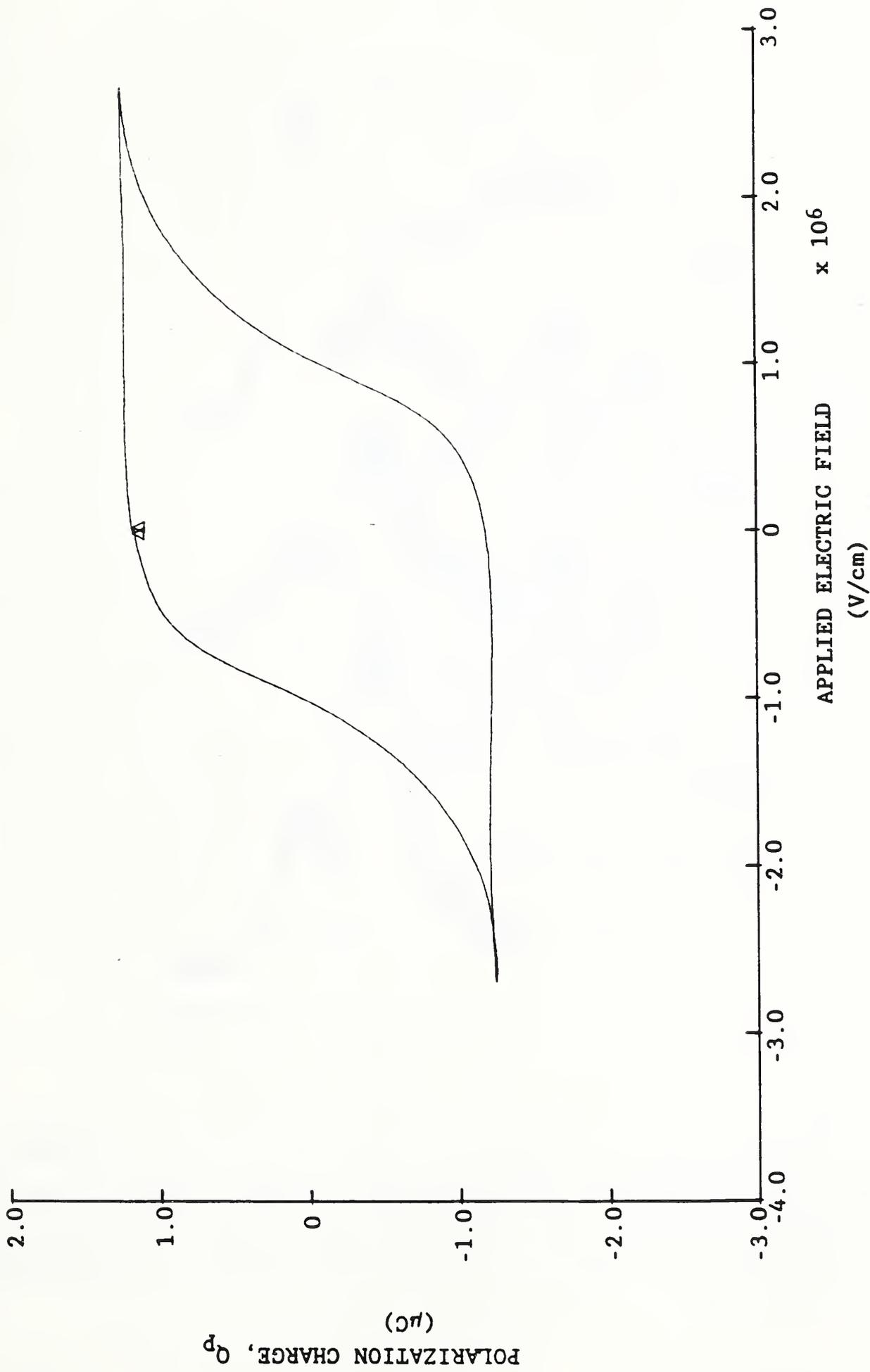
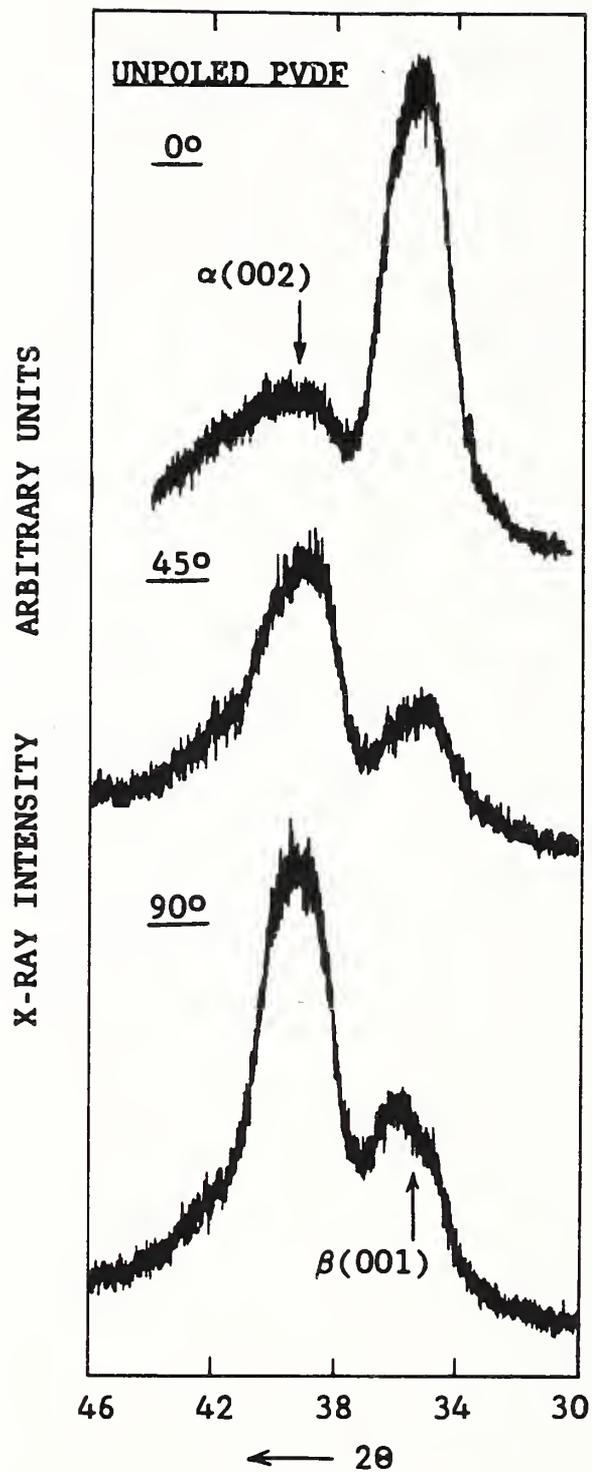


Figure 5. The polarization charge,  $Q_p$ , for gage # 14 is shown in a hysteresis plot. The remanent polarization is obtained by dividing the charge at zero field by the area of the gage electrodes,  $0.196 \text{ cm}^2$ . In this case,  $P_r = 5.99 \mu\text{C/cm}^2$ .



**Figure 6.** X-ray scans, taken in transmission, are shown for three orientations of the unpoled PVDF film. These scans show  $\alpha$  (002) reflections at  $2\theta = 39^\circ$  and  $\beta$  (001) reflections at  $2\theta = 35^\circ$ . The angle of orientation is the angle between the principal direction of strain in the film and the plane of the incident and reflected x-ray beam.

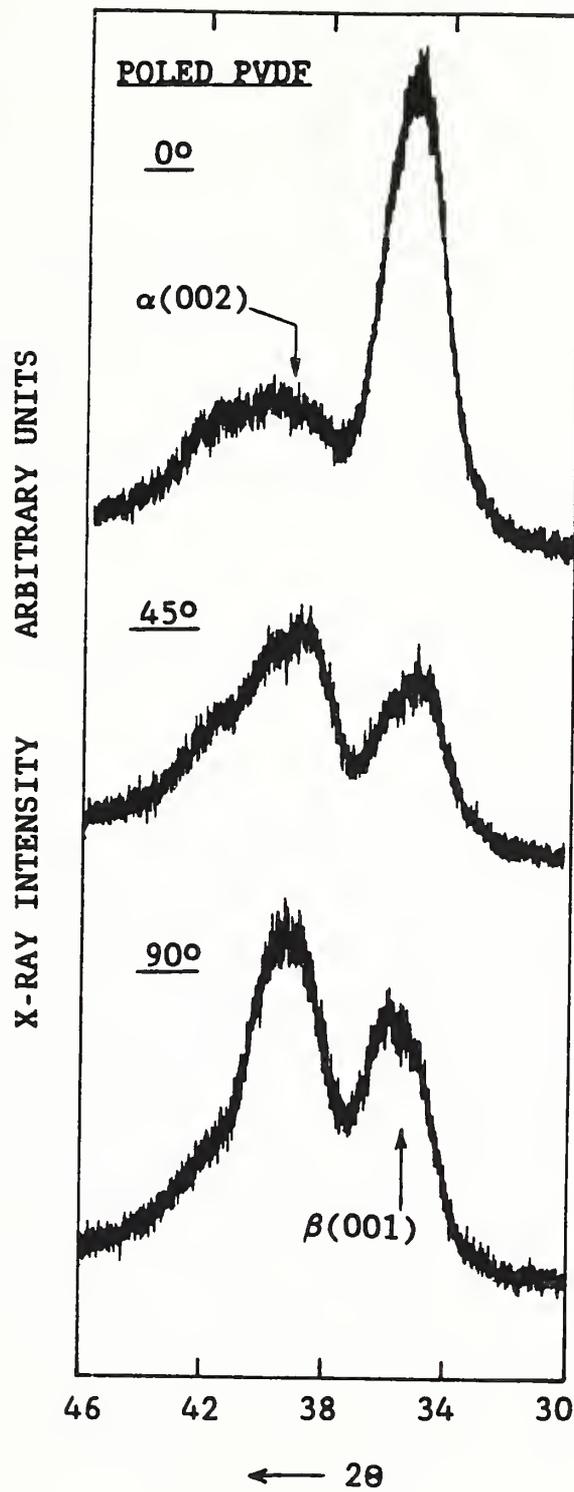


Figure 7. X-ray scans, taken in transmission, are shown for three orientations of the poled PVDF film. These scans show  $\alpha$  (002) reflections at  $2\theta = 39^\circ$  and  $\beta$  (001) reflections at  $2\theta = 35^\circ$ . The angle of orientation is the angle between the principal direction of strain in the film and the plane of the incident and reflected x-ray beam.



U.S. DEPT. OF COMM. <b>BIBLIOGRAPHIC DATA SHEET</b> <i>(See instructions)</i>	<b>1. PUBLICATION OR REPORT NO.</b> NBSIR 87 3680	<b>2. Performing Organ. Report No.</b>	<b>3. Publication Date</b>
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<b>10. SUPPLEMENTARY NOTES</b>  <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached.			
<b>11. ABSTRACT</b> <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> Thin film polyvinylidene fluoride transducers have been prepared by a poling procedure which involves the repeated cycling of the applied electric field over the ferroelectric hysteresis curve. Using this procedure, it is possible to prepare a uniformly poled transducer and to establish, with precision, the final remnant polarization. The remnant or permanent polarization is calculated by measuring the total poling charge and subtracting from it that which arises from the resistance and capacitance of the specimen. By this method we were able to prepare ten gages with the same remnant polarization with a precision of 0.5%. X-ray observations and measurements of density on an unpoled specimen show that the film is biaxially oriented, contains both $\alpha$ and $\beta$ phase crystals, and is 50% crystalline. A poled specimen with a remnant polarization of $5.7 \mu\text{C}/\text{cm}^2$ showed a decrease in the $\alpha$ phase content with a corresponding increase in the $\beta$ phase, but the overall crystallinity remained unchanged. We observe that polarization saturation is a function of the maximum poling field and it is suggested that the existence of space charge at the surface of the polar crystals presents an opposing local field to the crystallites and moderates the ferroelectric switching over a wide range of applied fields.			
<b>12. KEY WORDS</b> <i>(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)</i> density; ferroelectric hysteresis; piezoelectric shock gages; polymer transducers; polyvinylidene fluoride; remnant polarization; x-ray			
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