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PIEZO- AND PYROELECTRICITY OF POLY(VINYLLIDENE FLUORIDE) FROM PLASMA POLING

by

10 John E. McKinney — G. Thomas Davis

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theory of Mopsik and Broadhurst, which expresses these coefficients as linear functions of polarization. The theory gives an accurate representation of the piezoelectric coefficients, but underestimates the pyroelectric coefficients considerably.

In addition, x-ray measurements from various techniques were made on both drawn and undrawn PVDF samples. For the unpoled samples the results are in good agreement with those in the literature. Some changes in spacings and intensities were observed with noing for both the drawn and undrawn samples. As yet we do not have a clear interpretation of these results in terms of crystal structure changes resulting from polarization.

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Piezo- and Pyroelectricity of  
Poly(vinylidene fluoride) from Plasma Poling

J. E. McKinney and G. T. Davis

Abstract

A plasma poling device is described which allows essentially instantaneous poling of polymeric films at room temperature at very high fields. Using poly(vinylidene fluoride) both undrawn (comprising essentially pure  $\alpha$  conformation) and biaxially drawn (both  $\alpha$  and  $\beta$  conformation) were polarized in this manner. The polarization of the sample was determined from current integration measurements. The piezoelectric and pyroelectric coefficients were then measured and compared with those estimated from the polarization theory of Mopsik and Broadhurst, which expresses these coefficients as linear functions of polarization. The theory gives an accurate representation of the piezoelectric coefficients, but underestimates the pyroelectric coefficients considerably.

In addition, x-ray measurements from various techniques were made on both drawn and undrawn PVDF samples. For the unpoled samples the results are in good agreement with those in the literature. Some changes in spacings and intensities were observed with poling for both the drawn and undrawn samples. As yet we do not have a clear interpretation of these results in terms of crystal structure changes resulting from polarization.

PIEZO- AND PYROELECTRICITY OF  
POLY(VINYLLIDENE FLUORIDE) FROM PLASMA POLING

By

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I. Introduction

This paper is a preliminary report which includes a description of the plasma poling process, the consequences of this kind of poling in terms of piezo- and pyroelectric response, and an attempt to interpret the results in terms of molecular structure.

Conventional poling procedure involves heating the polymer sample to an elevated temperature, applying an electric field for a brief period of time, and then cooling to room temperature under the field. Recently, Southgate<sup>1</sup> has demonstrated that by applying corona discharge at high potential in the vicinity of a poly(vinylidene fluoride) (PVDF) sample, with the opposite side at ground potential, the poling process is essentially completed in several seconds at room temperature. There is also the advantage that higher fields may be obtained by corona or plasma poling. Since poling times are so short it is relatively easy to measure the total charge on the sample from poling, as demonstrated by Southgate. After the applied field is removed, the residual charge is a measure of the irreversible or "permanent" polarization from which it is possible to derive expressions for the piezo- and pyroelectric responses.

The piezoelectric and pyroelectric response from polymers has been related to polarization, which is linear with applied field for amorphous polymers<sup>2</sup>. Since the charge has been measured during the poling process in our work, these data give us the opportunity to evaluate the theory in terms of experimental quantities for semicrystalline polymers as well. It will be shown that the model predicts the piezoelectric response within experimental accuracy; however, some refinement appears to be necessary for the pyroelectric response. On the other hand, some of the experimental values of quantities appearing in the theory are somewhat dubious, it is not appropriate to pass judgement at this time.

There has been considerable recent interest and some speculation on the nature of apparent phase transitions in PVDF resulting from poling at high fields. For example, such behavior is indicated by changes in infrared spectra as observed by Southgate<sup>1</sup>. We are presently conducting investigations along these lines employing various X-ray and IR-techniques.

II. Theoretical Relationships between Piezo- and Pyroelectric Coefficients and Polarization

Using an Onsager type reaction field model, Hopsik and Broadhurst<sup>2</sup> have derived the relation for the polarization

$$P = (\epsilon_{\infty} + 2)(N\mu_0/3V) \langle \cos \theta \rangle \quad (1)$$

where  $\epsilon_{\infty}$  is the limiting high frequency dielectric constant of the medium,  $N$  is the number of dipole moments each of strength  $\mu_0$  in total volume  $V$ , and  $\langle \cos \theta \rangle$  is the average cosine of the dipole orientation angle  $\theta$ . The above relation gives the permanent polarization in a homogeneous, non-conducting, polarizable medium.

With semicrystalline polymers some modification is necessary to take into account the fact that polarization exists only within the crystalline region and the amorphous regions can be slightly conductive. Although the conductivity of poly(vinylidene fluoride) is very small, a gradual decay of polarization results from migration of real charge through the amorphous regions to the crystal surfaces<sup>3</sup>.

In order to adapt the continuum model<sup>2</sup> to a semicrystalline medium we assume<sup>4</sup>

$$P = x \frac{3\epsilon_s}{2\epsilon_s + \epsilon_c} P_c \quad (2)$$

where  $x$  is the crystalline fraction based on volume,  $P_c$  is the total polarization of the crystalline region, and  $\epsilon$  is the relative permittivity with subscripts  $s$  and  $c$  pertaining to sample and crystal respectively. The dipole moments in the amorphous region are assumed to be randomized at temperatures above  $T_g$  after removal of the external poling field. To account for the real charge redistribution mentioned above, we include an additional term in eq.(1) which becomes

$$P(t) = x \frac{3\epsilon_s}{2\epsilon_s + \epsilon_c} \left[ \frac{N\mu_0}{3} \frac{\epsilon_c + 2}{V_c} \langle \cos \theta \rangle - \frac{Q'(t)}{A_c} \right] \quad (3)$$

$A_c$  is the total area of the crystals, and  $Q'$  is a time dependent charge at the crystal surfaces which satisfies the boundary conditions

$$Q'(0) = 0 \text{ and } \lim_{t \rightarrow \infty} P(t) = 0$$

From application of the experimental definitions

$$d_p = (1/A) [\partial(AP)/\partial p]_T \quad (4a)$$

$$\kappa = -(1/A) [\partial(AP)/\partial T]_p \quad (4b)$$

where  $d_p$  is the hydrostatic piezoelectric coefficient,  $\kappa$  is the pyroelectric coefficient,  $A$  is the electrode area, and  $T$  and  $p$  are the temperature and pressure. The final expressions become

$$d_p = P_0 \beta_c \left[ \frac{2\epsilon_c + 1}{6} + \phi \frac{J_1(\phi)}{J_0(\phi)} \nu \right] \quad (5a)$$

$$\kappa = P_0 \alpha_c \left[ \frac{2\epsilon_c + 1}{6} + \phi \frac{J_1(\phi)}{J_0(\phi)} \left( \nu + \frac{1}{2T\alpha_c} \right) \right] \quad (5b)$$

where  $P_0$  is the initial value of  $P(t)$  after poling,  $\beta_c$  and  $\alpha_c$  are the crystal compressibility and thermal expansion,  $\phi$  is the dipole fluctuation angle,  $\nu$  is the Gruneisen constant, and  $J_0$  and  $J_1$  are Bessel functions of first kind of order 0 and 1, respectively.

More explicit detail of the development of eqs.(5) will be included in a later publication. In brief, it is assumed that the cell dimension (along the chain axis) change with temperature or pressure is negligible. Pressure and temperature dependence in  $\epsilon_c$  are taken from the Clausius Mossotti equation. Considerable simplification in the expressions for  $d_p$  and  $\kappa$  ensues from evaluating these quantities at the ultimate polarization [ $P(t) = 0$ ] when crystal polarization is compensated by real charge at the crystal liquid interface.

### III. Experimental

#### A. Apparatus

The plasma chamber used to generate plasma for the poling process was adapted from a commercial plasma cleaner. The electrode assembly is shown schematically in Fig. 1. An aluminum electrode of 2.5 cm diameter is evaporated on one side of each polymer film sample. The sample is clamped as shown with the bare side exposed to the plasma. The chamber (containing air) is evacuated to and maintained at about 200 mT during the poling process. Water vapor and other condensable gases are removed with a liquid nitrogen trap.

Voltages up to 10 kV may be applied. The corresponding charge transfer is measured with a charge amplifier in series with the high voltage circuit. Electrical leakage was found to be negligible over the short poling times used. From the charge transfer the polarization of the sample was determined.

After poling, a graphite electrode was painted on the bare side of each sample. The electrodes were shorted and the samples stored for at least twelve hours for surface charges to dissipate. The piezoelectric coefficients were then measured using a pressure cell<sup>5</sup> imposing nearly constant rate of pressure change. From the slopes of the pressure-time curves and corresponding currents,  $d_p$  is determined. In an analogous manner the pyroelectric coefficient  $\kappa$  is determined from the measurement of current resulting from nearly constant rate of temperature change.

X-ray measurements were made with an automatic scanning diffractometer and a powder camera using Nickel-filtered-Cu  $K\alpha$  radiation in both cases. For the powder camera, the sample film was cut into thin strips with dimensions approximately 3 x 0.3 x .025 mm. These were inserted and sealed in a capillary of 0.3 mm diameter.

#### B. Samples

Two kinds of commercial PVDF semicrystalline samples were studied. The first is a biaxially stretched (blow molded) film for which the polymer chains tend to orient in the plane of stretch. The stretch process converts a portion of the non-polar  $\alpha$  (TGTG' conformation) phase to the polar  $\beta$  (planar zig-zag) phase. The second sample is undrawn and contains essentially only the  $\alpha$  phase. Both kinds of film are about 25  $\mu\text{m}$  in thickness.

### IV. Results and Discussion

Fig. 2 gives the initial polarization  $P_0$  (as measured from the charge remaining after the applied field is removed) plotted against the applied field, or applied voltage, for the biaxially drawn sample. About three seconds are required to reach 90% of the ultimate value of charge at room temperature. As indicated, the data are somewhat erratic. Subsequent hysteresis loops, however, reveal smooth responses with applied field.



In all cases, except the one indicated by the triangle where the field was reversed, the plasma was at negative potential with respect to the electrode. As seen from the figure, there is essentially no polarization below one MV/cm. There is no indication of saturation in the polarization and dielectric breakdown prevents attaining fields much above 3.5 MV/cm. The numbers on the data points indicate correspondence to those on figures which follow.

According to the theory, for which the results are given in eqs.(5), the piezo- and pyroelectric coefficients are linear functions of the initial polarization  $P_0$ . The experimental values applied to these equations are given in Table I. Values of  $\alpha_c$  and  $\beta_c$  have been taken for the  $\alpha$  (non-polar) phase since these quantities for the  $\beta$  (polar) phase are apparently not available in the literature. The value of  $\alpha_c$  was obtained from the expansions of the unit cell dimensions<sup>6</sup>. The value of  $\beta_c$  was estimated by somewhat devious means from macroscopic compressibility measurements of a 68% crystalline fraction sample<sup>7</sup>.

In Fig. 3 the piezoelectric coefficient  $d_p$  is plotted against the initial polarization  $P_0$ . The straight line is a plot of the equation

$$d_p = 2.11 \times 10^{-10} P_0 \quad (\text{SI units})$$

which is evaluated from eq.(5a) taking the values given in Table I. In this case the agreement between theory and experiment are essentially within data scatter.

The corresponding pyroelectric response is shown in Fig. 4. The straight line represents the equation

$$\kappa = 2.97 \times 10^{-4} P_0 \quad (\text{SI units})$$

evaluated from eq.(5b) again using the experimental values from Table I. There is considerable underestimation by the theory for which the ratio, theoretical/experimental, is about 2/3. At this time we do not know why  $\kappa$  is underestimated.

We also polarized an undrawn PVDF sample composed of the "non-polar"  $\alpha$  phase. At an applied field of 1.75 MV/cm the responses are  $d_p = 13 \text{ pC/N}$  and  $\kappa = 3.1 \text{ nC/cm}^2\text{K}$ . A charge meter malfunction prevented us from obtaining a polarization value on this sample.

There is considerable interest in apparent morphological changes during the poling process. (See for example, references 1 and 8.) From reflection x-ray diffractometer measurements on biaxially drawn PVDF (comprising both  $\alpha$  and  $\beta$  phases), we have observed changes in structure at discrete intervals during the poling process. Fig. 5 shows the diffraction intensities with angle  $2\theta$  before and after poling at 3.4 MV/cm. Note that the (100), (020) and (120) reflections from the  $\alpha$  phase essentially disappear after poling, whereas the (110) of  $\alpha$  and the combined (110) and (200) of  $\beta$  appear to increase slightly. At this time it is not clear if these increases are significant. At  $36^\circ 2\theta$  some changes occur, but, unfortunately, it is not possible to distinguish between contributions from  $\alpha$  and  $\beta$  phases in this region.

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When the same specimens are examined in transmission using photographic techniques, the (100) and (020) reflections are still readily apparent so that only certain crystal orientations with respect to poling field are affected in this manner.

In looking at the undrawn ( $\alpha$  phase) sample, the situation appears to be slightly different, as revealed by Fig. 6. The (110), (020), and (100) intensities seem to diminish slightly with poling; however, none of these disappear as with the (020) and (100) for the biaxially stretched sample. The important result is that an additional reflection seems to appear at  $20.7^\circ$ , which would coincide with the (110) and (200) of  $\beta$  phase shown on Fig. 5. Based on this evidence alone, one might speculate that there is a partial conversion from  $\alpha$  to  $\beta$  phase during poling. However, we have not observed any of the other reflections to be expected from  $\beta$  phase either by reflection in the diffractometer, by transmission in a flat plate camera or in a powder camera. The spacings obtained by us using the powder camera for the unpoled samples are in good agreement with those obtained in Refs. 11 and 12.

Our tentative conclusion is that the non-polar  $\alpha$  phase is modified under the influence of high electric fields to yield a polar crystal phase which persists after removal of the external field. The unit cell of the  $\alpha$  phase is non-polar but the TGTG' configuration within a given chain traverse of the crystal has a large component of dipole moment normal to the chain axis. The mechanism proposed would involve rotation of polymer chains about their own axes to align the dipole with the applied field resulting in a metastable state. Those crystallites oriented with the chain axis parallel to the surface of the film would be subjected to the largest interaction energy. It is clear that the applied field has altered the orientation of crystallites and perhaps the crystal structure, but the details are not yet resolved.

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Table I  
Experimental Values in SI Units of  
Quantities Appearing in Eqs. (5)

Quantity	Value	Reference
$\alpha_C$	$1.69 \times 10^{-4}$	6
$\beta_C$	$1.56 \times 10^{-10}$	6,7
$\epsilon_C$	3	9
$\nu$	4.55	10
$\phi$	0.284	2
T	300	-

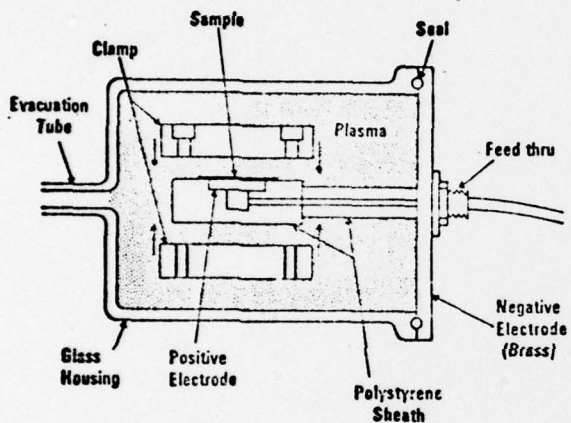


FIGURE 1: Schematic drawing of poling electrode assembly.

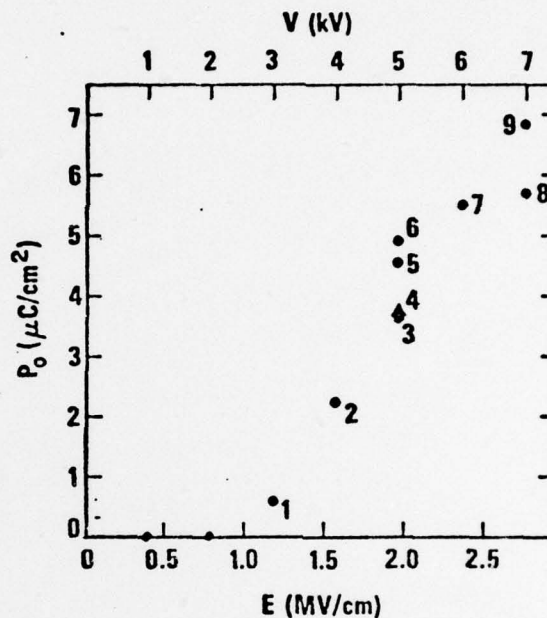


FIGURE 2: Initial polarization versus applied field (or voltage) for biaxially drawn PVDF.

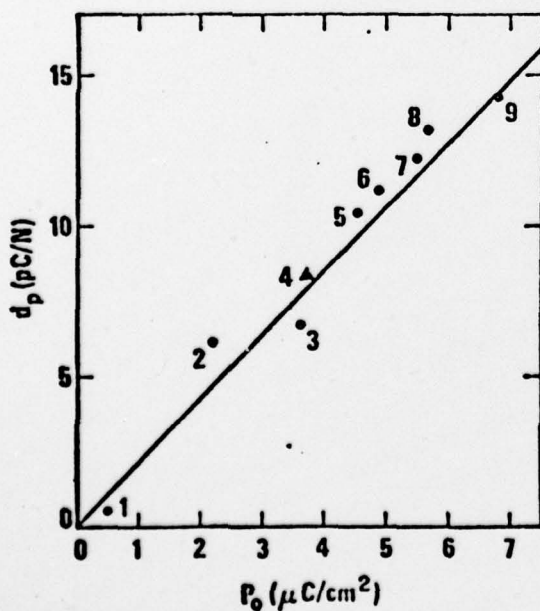


FIGURE 3: Piezoelectric coefficient  $d_p$  versus initial polarization for biaxially drawn PVDF.

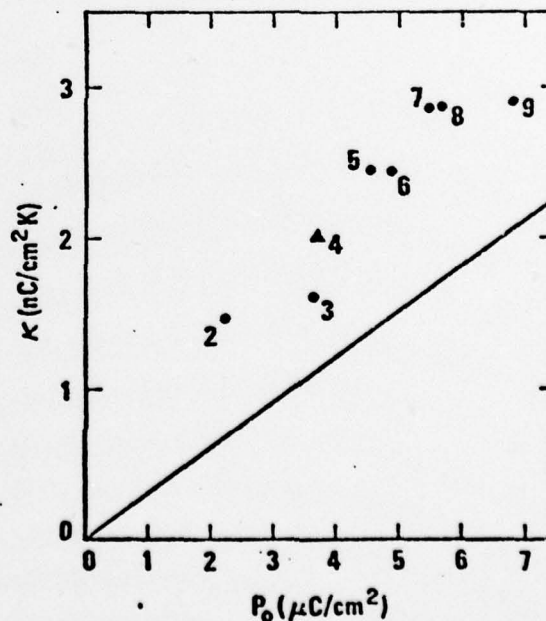


FIGURE 4: Pyroelectric coefficient  $\kappa$  versus initial polarization for biaxially drawn PVDF.

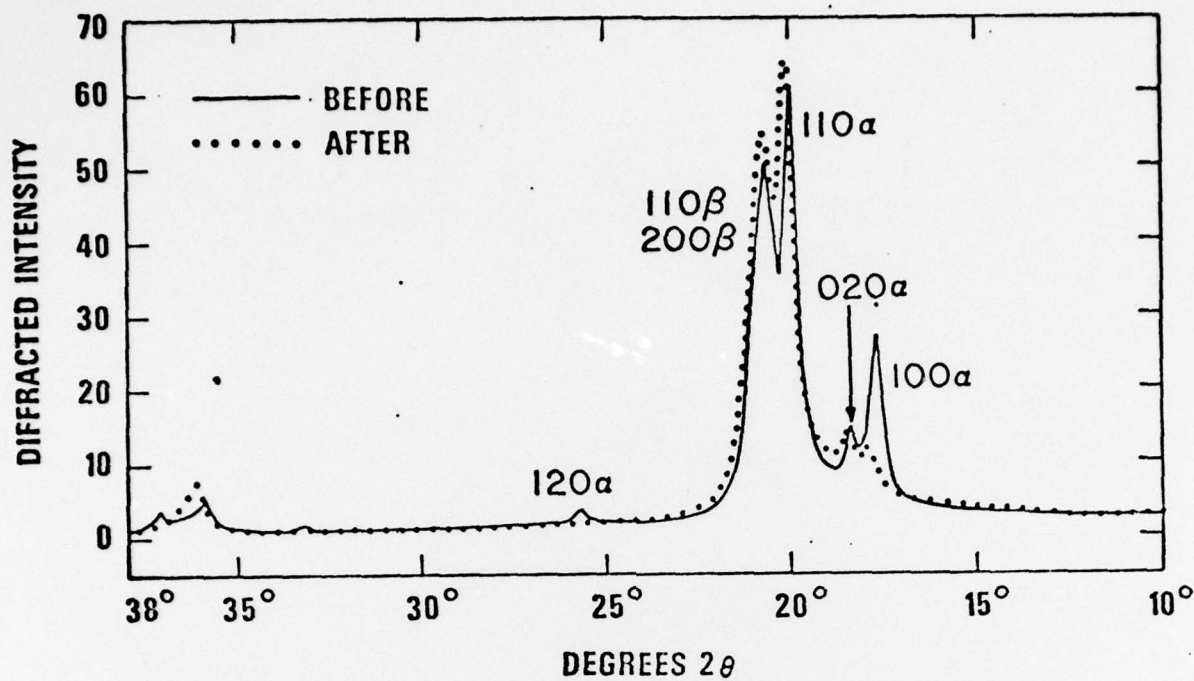


FIGURE 5. X-ray diffractometer scan for biaxially drawn PVDF before (—) and after (...) poling at 3.4 MV/cm.

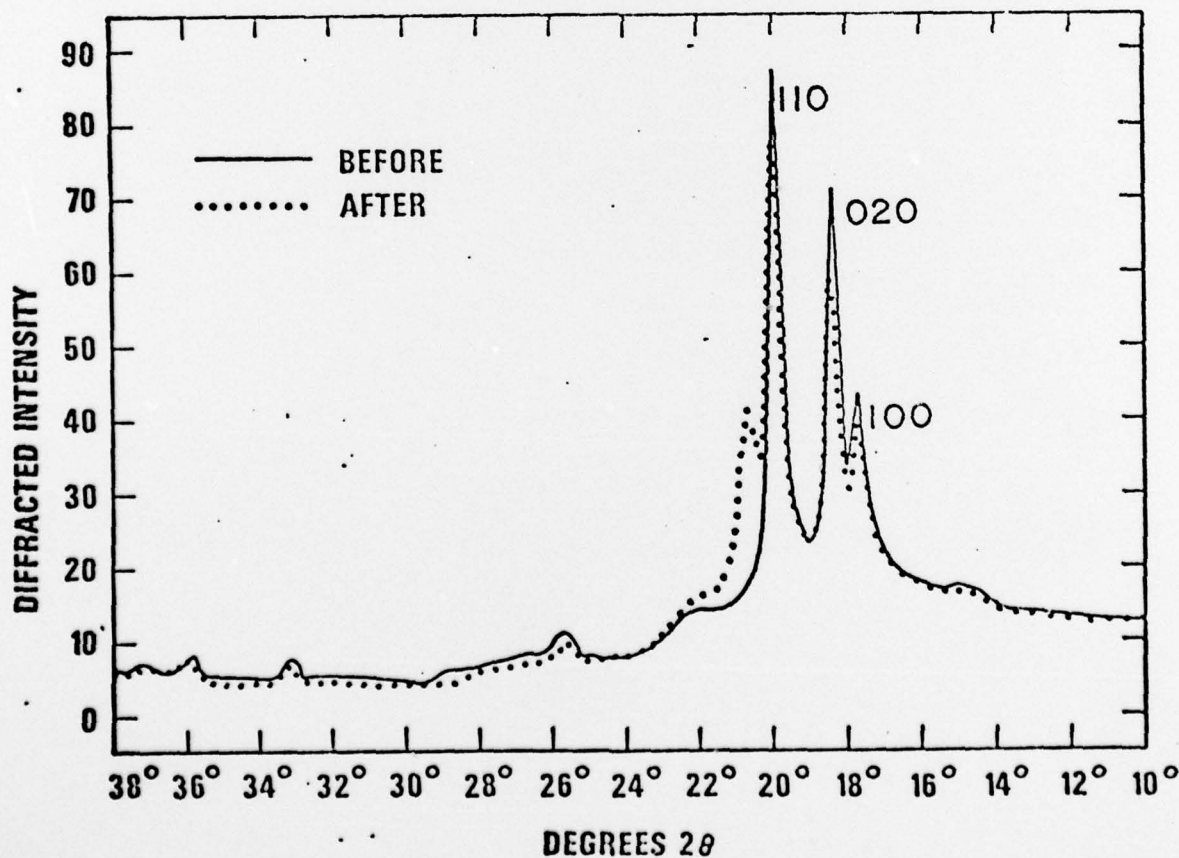


FIGURE 6. X-ray diffractometer scan for undrawn PVDF before (—) and after (...) poling at 1.7 MV/cm.

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