

## X-Ray Investigations of Shellac Wax Electrets

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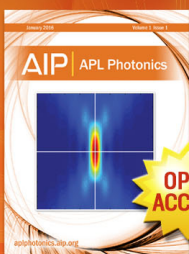
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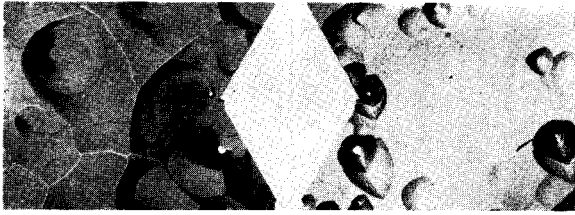


FIG. 2. Correlation between the etch pits produced by the RC-1 etch on the Ga {111} and the As {111} faces of GaAs. The Ga {111} face is to the left and the As {111} face is to the right,  $\times 54$ .

tively. The etching is performed at room temperature without agitation; pits are produced in about 3 min. The pits on the arsenic face resemble those produced by the Schell etch on the gallium face in that they are triangular pyramids; on the gallium face, conical pits are produced.

To correlate these etch pits with dislocations, a Ga {111} face was etched for 12 min in a solution containing 1 part  $\text{HNO}_3$  and 2 parts  $\text{H}_2\text{O}$  (Schell's etch) and photographed [Fig. 1(a)]. Following this, the surface was etched, without prior polishing, for an additional 1.5 min. in the RC-1 etch. The same area of the specimen appearing in Fig. 1(a) was again photographed and is shown in Fig. 1(b). The removal by the RC-1 etch of about  $15\ \mu$  from the surface is indicated by the disappearance of the scratch seen in Fig. 1(a). It is apparent that no new pits have formed and that there exists a 1:1 correspondence between the pits in Fig. 1(a) and (b). The same result was obtained when the specimen is first etched in the RC-1 etch and then in the Schell etch. Since it is known that the pits produced by the Schell etch correspond to emergent edge dislocations,<sup>3</sup> it is concluded that the pits produced on the Ga {111} plane by the RC-1 etch must also correspond to edge dislocation sites.

To show that the etch pits on the As {111} face also correspond to emergent edge dislocations, it is necessary to demonstrate that a 1:1 correspondence exists between the etch pits on (111) and ( $\bar{1}\bar{1}\bar{1}$ ).

If GaAs exhibited gross cleavage parallel to {111}, this demonstration would be simplified because it would then be possible to etch two mirror faces which were atomically close. Unfortunately, GaAs exhibits macrocleavage on planes of the form {110}.<sup>6</sup> Hence, the technique used was to reduce a specimen to a thickness of about  $50\ \mu$  with a chemical reaction using a reagent consisting of equal parts of HF,  $\text{HNO}_3$ , and  $\text{H}_2\text{O}$ .<sup>1</sup> The specimen was then placed in the RC-1 etch for 5 min, and corresponding areas on the mirror faces were photographed. The As {111} face can be seen to the right of the center of Fig. 2, and the Ga {111} face to the left. Even though the shape of the pits is different, it can be seen that a 1:1 correspondence exists between the center of the pits on the Ga {111} and those on the As {111} faces. The bright regions associated with the two pits near the edges of the two faces are due to the transmission of light through the specimen. Due to the fragility of the specimen at a thickness of  $50\ \mu$ , no attempt was made to polish the {111} faces prior to etching which would have produced a cleaner background. Since the desired correspondence between the pits on the Ga and As faces was found, it is concluded that the etch pits on the As {111} face also denote the sites of emergent edge dislocations.

In summary, it has been shown that the pits produced by the RC-1 etchant on the {111} faces of GaAs correspond to emergent edge dislocations.

<sup>1</sup> H. A. Schell, *Z. Metallk.* **48**, 158 (1957).

<sup>2</sup> H. C. Gatos and M. C. Lavine, in *Progress in Semiconductors* edited by A. Gibson, F. Kroger, and R. Burgess (John Wiley & Sons, Inc., New York, 1963), 7th ed.

<sup>3</sup> M. S. Abrahams and L. Ekstrom, in *Properties of Elemental and Compound Semiconductors*, edited by H. C. Gatos (Interscience Publishers, Inc., New York, 1960).

<sup>4</sup> The term edge dislocation is meant to include the familiar  $60^\circ$  dislocation of the zinc-blende structure.

<sup>5</sup> J. L. Richards and A. J. Crocker, *J. Appl. Phys.* **31**, 611 (1960).

<sup>6</sup> G. A. Wolff, *Acta. Cryst.* **12**, 313 (1959).

## X-Ray Investigations of Shellac Wax Electrets

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SHELLAC wax electrets were prepared at different temperatures ranging from room temperatures to  $90^\circ\text{C}$ .<sup>1</sup> Samples were carefully prepared and photographed with a Philips x-ray diffraction unit PW 1010 using copper  $K$  radiation and a nickel filter. Shellac wax produced a strong pattern when x rays passed either in a direction perpendicular or parallel to the field. Nine distinct rings could be measured and analysis was made; see Table I.

TABLE I. Rings photographed with Cu  $K$  radiation on shellac wax electrets.

Sample No.	Bragg angle ( $\theta$ )	Spacing $d\text{\AA}$	Miller indices ( $hkl$ )	Intensity classification
Halo	6.1667	7.180	...	Halo
1	10.40	4.26	(110)	V. V. strong
2	11.75	3.78	(200)	V. strong
3	13.1667	3.385	...	Medium faint
4	14.7176	3.030	(210)	Strong
5	17.6917	2.503	(300)	Strong
6	19.7833	2.278	(310)	Strong
7	21.3667	2.116	(220)	Medium strong
8	24.4167	1.865	(400)	Medium faint
9	26.0917	1.758	(320)	Faint

The innermost halo as observed in practically all photographs can be explained in various ways. It may be caused by a small amount of general radiation or by the structure of component materials used for preparation of the electrets. Practically all the photographs showed a nonuniform intensity along the rings (110) and (200) enhanced in the direction of the field and reduced in the direction perpendicular to the field. The variation in intensity indicates a preferred orientation of crystallites inside the electret and it is maximum for electrets prepared at  $77.5^\circ\text{C}$ . This leads to the conclusion that at temperatures near the melting point of the wax, the orientation of dipoles is produced by a polarizing field.

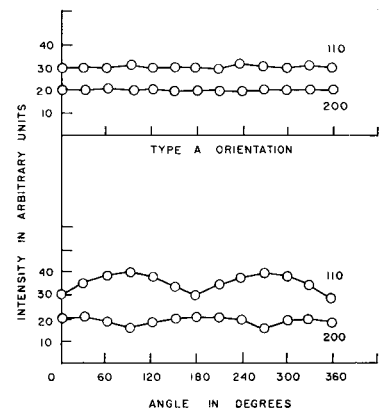
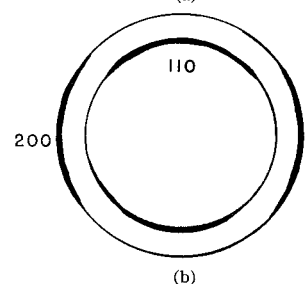


FIG. 1. Variation of intensity around (110) and (200) rings (type-A orientation). (a) The upper graph shows the case when the X-ray beam was parallel to the field and the lower graph shows the case when X rays passed perpendicular to the field. (b) Drawing from the original photograph.



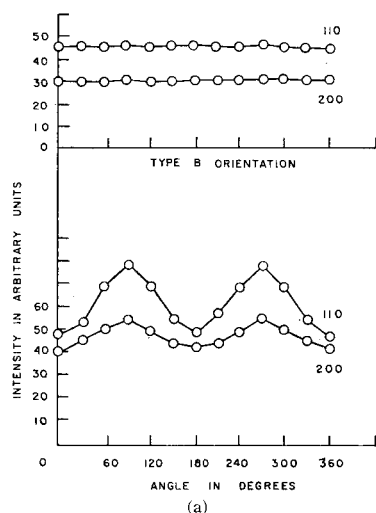
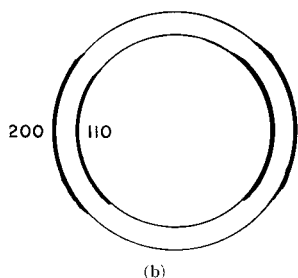
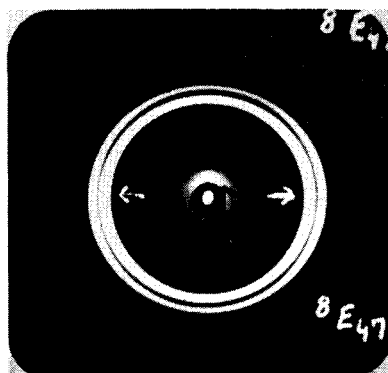


FIG. 2. Variation of intensity around (110) and (200) rings (type-B orientation). (a) The upper graph shows the case when the X-ray beam was parallel to the field and the lower graph shows the case when the X-ray beam passed perpendicular to the field. (b) The original photograph and its drawing.



The general nature of the diffraction patterns and the values of spacings indicate the rhombic modification of long chain compounds. In shellac wax electrets prepared at 77.5° and 80°C, the photographs showed two types of orientations and this result was further confirmed by photographs of many other electrets prepared at the same temperature. In the case of type-A orientation, the maximum intensity of rings (110) is in the direction of the applied field and in the case of (200) ring, enhancement of intensity is perpendicular to the field direction (Fig. 1). In the case of type-B orientation, the maximum intensity of both the rings (110) and (200) is in the direction of the applied field (Fig. 2). The temperature, polarizing field, and time are the only factors which determine the conditions for the occurrence of type-A and type-B orientations. The present results support the theoretical model for type-A and type-B orientation given by Müller,<sup>2</sup> Tiku,<sup>3</sup> and Kakiuchi.<sup>4</sup> The orientational distribution of dipoles was calculated in the present investigation using the dispersion angle.

In general, the dipoles may be randomly oriented in relation to some selected frame of reference or they may be oriented about a particular axis, depending on experimental conditions. Preferred orientation may, therefore, be defined simply as distribution of

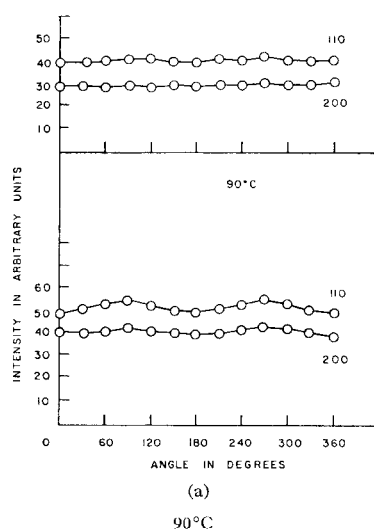
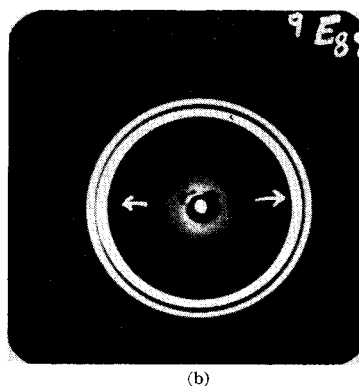


FIG. 3. Variation of intensity around (110) and (200) rings in the case of electrets prepared at 90°C. (a) graphs; (b) original photograph.



crystal orientation which occurs at a critical temperature. It is only a crystallographic condition and does not depend upon the shape of the dipoles as disclosed by microscopic studies. Only x-ray diffraction studies can give any evidence of preferred orientation and it is best described by a pole figure.<sup>5</sup> It has been observed that the orientation increases with the field strength but that the upper limit is at 10 kV/cm, after which the amount of orientation seems to be constant. X-ray studies also indicated that in electrets prepared at 90°C, the probability of orientation of dipoles is lessened, due to the high mobility at that temperature and only one type of orientation namely type-B was observed (Fig. 3). This confirms the observation that temperatures above 77.5°C contribute to the lessened orientation which is named type B. The dipole orientation is important in the formation of the electrets and it is concluded that the temperatures near the melting point of electrets forming materials have a significant influence on the final charge and the stability of such electrets.<sup>1</sup> At low temperatures, the heterocharge into a homocharge takes many hours. In conclusion, the oriented dipoles are responsible for the stability of homocharge as found in the present investigations on shellac wax electrets. The properties of shellac wax electrets do not disappear when layers are removed from the surface. These electrets, when stored, should be short-circuited. Irradiation of these electrets with x-ray reduces the surface charge but after some time, the total charge is recovered.

\* O. P. Puri, Ph.D. thesis, Saugar, India (1961): work supported by the University Grants Commission.

<sup>1</sup> O. P. Puri, *J. Phys. Soc. Japan* **18**, 1115 (1963).

<sup>2</sup> A. Müller, *Proc. Roy. Soc. (London)* **A138**, 514 (1932); *Proc. Roy. Soc. (London)* **A120**, 369 (1938).

<sup>3</sup> T. Tiku, *Proc. Phys. Math. Soc. (Japan)* **14**, 63 (1932).

<sup>4</sup> Y. Kakiuchi, *Sci. Pap. I. P. C. R.* **40**, 189 (1943).

<sup>5</sup> F. Happel, *Institute of Physics Publication* (Reinhold Publishing Corp., New York, 1960), p. 481.