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B. Gross

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On Permanent Charges in Solid Dielectrics. II.* Surface Charges and Transient Currents in Carnauba Wax

B. Gross
National Institute of Technology, Rio de Janeiro, Brazil
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Permanent surface charges are produced in disks of carnauba wax by exposure to an external electric field. The nature of these charges is studied with a new method of analysis, which is based on the simultaneous measurement of the external current and of the induced charges on the electrodes of the capacitor containtaining the dielectric. The observed effects are explained by dielectric absorption and by transfer of charge from the electrode to the surface of the dielectric; this latter phenomenon is due to surface breakdown and continuous conduction currents. The results provide an adequate explanation for the electret.

ISPLACEMENTS of charge carriers within the dielectric and exchange of carriers between dielectric and electrodes take place during exposure of a solid dielectric to an external field and after withdrawal of the field and short-circuiting. These effects manifest themselves by the production of time-dependent currents in the external circuit and by the induction of electric charges on the electrodes of the capacitor containing the dielectric. This paper reports a systematic study of the permanent surface charges and transient currents for disks of carnauba wax and discusses their bearing on the interpretation of the electret. Use is made of the results achieved in a former research and published as Part I of this paper. A preliminary report² has already been published. A list of pertinent papers is given below;3 for a more complete bibliography we refer to the recent report given by Gutmann.4

I. DIELECTRIC TESTING WITH THE DISSECTIBLE CAPACITOR

1. The Dissectible Capacitor

Current and induced charges are measured with the dissectible capacitor⁵ (Fig. 1). The dielectric, in the form of a disk (diameter 7 cm, height 7-10 mm) is

* Paper handled by the Committee on Inter-American Scientific Publication.

¹ B. Gross and L. F. Dennard, Phys. Rev. 67, 253 (1945).

⁶ The origin of the dissectible capacitor as a tool in the study of dielectric phenomena goes back to Benjamin Franklin; See Bernhard Gross, Am. J. Phys. 12, 324 (1944), and J. Zeleny, *ibid.* 12, 329 (1944).

clamped to the lower, fixed electrode of the capacitor by means of a guard ring. The upper electrode (diameter 6.5 cm) can be lifted; it is suspended on an iron piston which moves in the field of a coil. Passing the current through the coil lifts piston and electrode; interruption of the current brings the electrode again into contact with the dielectric. Since the electrode is not rigidly fixed to the piston, but is allowed a certain freedom of motion, it adjusts itself easily to the surface of the dielectric and an adequate contact between dielectric and electrode is achieved. The weight of the movable system is 815 g; an airdamping device makes the movement of the system sufficiently smooth. Nickelplated and steel electrodes have been used without measurable difference in the results. A special shielding device closes over the dielectric every time the electrode is lifted, avoiding any influence of the polarized dielectric upon the measuring electrode during the act of measuring. The closing and opening of this device is accomplished by the movement of the piston. The movable electrode is connected with the measuring system, which is constituted by a one-fiber string electrometer and an adjustable air capacitor; this electrode and the connecting wires are insulated by sulphur insulators. The lower electrode is during polarization connected with the high voltage source; otherwise it is grounded. The guard ring and other metal parts of the capacitor are always grounded. Heating coils are placed around the electrode system for measurements at elevated temperatures. Temperature is read on a mercury thermometer; a thermocouple is also provided for. The entire capacitor, including the heater, is enclosed

2. Technique of Measurement

All measurements were made with samples of first-grade (yellow) filtered carnauba wax under conditions of very low humidity. For obtaining quantitative results one should avoid operations involving uncontrolled handling of the samples, like taking them out of the capacitor, exposing them to humid air, and wrapping them in tinfoil and unwrapping them. Thus during one experimental cycle the sample remained always in the

in a desiccator.

² Bernhard Gross, Phys. Rev. 66, 26 (1944).

³ O. Heaviside, Electrical Papers, Vol. 1, pp. 488; M. Eguchi, Proc. Phys. Math. Soc. Japan. Ser. 3, 1, 326 (1919); 2, 169 (1920); Phil. Mag. 49, 179 (1925); S. Mikola, Zeits. f. Physik 32, 476 (1925); K. Nakata, Proc. Phys. Math. Soc. Japan. Ser. 3, 9, 179 (1927); E. P. Adams, J. Frank. Inst. 204, 469 (1927); M. Ewing, Phys. Rev. 36, 378 (1930); R. D. Bennett, Phys. Rev. 37, 103 (1931); O. J. Johnson and P. H. Carr, Phys. Rev. 42, 912 (1932); P. Jaeger, Ann. d. Physik 21, 481 (1934); A. Gemant, Phil. Mag. 20, 929 (1935); Rev. Sci. Inst. 11, 65 (1940); Phys. Rev. 61, 79 (1942); G. Groetzinger and H. Kretsch, Zeits. f. Physik 103, 337 (1936); Thiessen, Winkel, and Herrmann, Physik. Zeits. 37, 720 (1936); W. M. Good and J. D. Stranathan, Phys. Rev. 56, 810 (1939); G. E. Sheppard and J. D. Stranathan, Phys. Rev. 60, 360 (1941); B. Gross, Anais Acad. Brasil. Ci. 15, 63 (1943); 17, 219 (1945); Partington, Planer, and Boswell, Nature 158, 835 (1946).

capacitor and the desiccator was not opened. This excluded the possibility of polarizing the dielectric in the molten state. But the samples, which we have polarized at an elevated temperature below the melting point, produce electric fields, the intensity and permanency of which reach the values mentioned in the literature for polarization in the molten state.

The circuit employed (Fig. 2) allows for measurement of the total external current J(t) and the induced charge q(t) of the removable electrode.

3. Analysis of the Currents

The electric charges $\pm q$ found on the electrodes of a shorted capacitor are due to induction by fields, which originate in other charges residing in the dielectric. Observation of the induced charges of the electrodes therefore gives information about the nature and timedependence of the charges in the dielectric. Quantitative information is gained, when use is made of the general equation, which according to Maxwell's theory connects currents and charges:

$$J(t) = i(t) + dq/dt, \tag{1}$$

where J(t) is the external current, i(t) is the conduction current across the dielectric-electrode interface, and dq/dt is the displacement current across the interface.

The simultaneous measurement of J(t) and q(t) permits the decomposition of the current across the interface into its two components; i(t) is a measure for the transfer of charge carriers between dielectric and electrode and dq/dt is a measure for the displacement of charge carriers within the dielectric. This method of measurement gives more insight into dielectric behavior than the usual practice of measuring either J or q; its field of application may conveniently be extended to dielectric testing in general.

4. The Field at the Surface of the Dielectric

The electric field E produced by the dielectric (in a shorted condition) is given by

$$E = 1.16 \times 10^{13} q/A \text{ volt/cm},$$
 (2)

where the area A is given in cm² and q in coulombs.

II. EXPERIMENTAL RESULTS

1. Dielectric Absorption and Heterocharge

All effects, which occur when low or medium polarizing voltage is applied at room temperature, can be explained by dielectric absorption. Figure 3 gives the typical aspect of the curves obtained under such conditions (V=950 volt and T=30°C). The current shows the familiar behavior characteristic for every solid dielectric and for carnauba wax already discussed in I. The charge of the electrode jumps from zero to a finite initial value, when the polarizing voltage is applied; then it increases continuously. At the moment of the short-circuit a jump of q of the same value as before, but in the contrary direction, is observed, followed by a slow decrease. The polarity of q does not change; the electrode, which during polarization was connected with the positive pole, carries always a positive charge, the adjacent surface of the dielectric a negative one. The displacement current obtained by differentiation of the function q(t) (actually $\Delta q/\Delta t$ is given) is found to coincide within the precision of the measurement with the measured values of the external current J(t). Thus no conduction current flows across the dielectric-electrode interface; this proves that no exchange of charge carriers between dielectric and electrode is taking place.

The jumps of q in phase with the voltage jumps of course are caused by the charging and discharging of the geometrical capacitance. The behavior of the timedependent currents and the coincidence of the J(t)and dq/dt curves is explained by dielectric absorption and agrees with the conclusions of the generally ac-

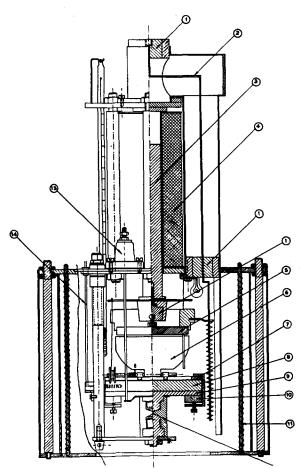


Fig. 1. Dissectible condenser.

- (1) Sulfur insulator
- Lead to measuring electrode Iron piston
- Magnetic coil
 Removable electrode
 Shielding device
 Guard ring
- Dielectric Fixed electrode Clamps Heater coil
- Insulator High voltage pole Thermocouple

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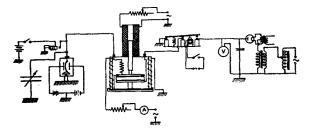


Fig. 2. Diagram of the circuit.

cepted theories of this effect. According to them, the following mechanisms may be effective: (a) hindered dipole orientation (Debye), associated with the rotation of dipole systems; (b) microscopical heterogeneity of structure (Maxwell-Wagner), associated with the displacement of ions over microscopical distances; (c) ionic conduction, associated with the formation of space charges. The two former effects do not involve any transfer of charge carriers between dielectric and electrodes and in the case of the last one this transfer plays a minor role. This being so, there is J = dq/dt. Formally absorption can always be accounted for by a time-dependent component of the volume polarization P(t). This leads to the appearance of surface charges s = -PA. It follows that

$$s(t) = \int_{t}^{\infty} J(t)dt. \tag{3}$$

The polarity of s is contrary to that of the corresponding electrode during polarization; thus according to Gemant's terminology s is a heterocharge. The relation (3) can be used generally for the computation of the heterocharge, if it is applied to the shorted system. Thus the value of the heterocharge at a time t after the short-circuit is given by the integral over the total discharge current from 0 to ∞ .

2. "Freezing-in" of the Heterocharge

A considerable fraction of the absorbed charge can be frozen in by an adequate heat treatment, as has been shown by Frei and Groetzinger⁹ and later discussed in I. Since dielectric absorption is always associated with a heterocharge, production of a permanently polarized dielectric carrying a heterocharge only is feasible. Such an experiment is shown in Fig. 4. A voltage of 175 v was applied at 70°C; after 30 min. the temperature was reduced to 40°C, then the system was short-circuited. Figure 4a shows the behavior immediately after the short-circuit, Fig. 4b that during reheating 2 weeks

⁶ The theory of this effect is due to G. Jaffé, Ann. d. Physik 16, 217 (1933). Space charges in electrets, which were polarized in the molten state, have been observed by Gemant (see reference 3) and by Thiessen, Winkel, and Herrmann (see reference 3).

⁷This relation between the absorption current and the surface charges of the electrodes of an absorptive capacitor is found in many papers on absorption, i.e. G. Jaffe (see reference 6).

8 A. Gemant, see reference 3.

later. The current behaves much as in I; the current surge during reheating indicates the liberation of a frozen-in charge. The sign of q again is that of a heterocharge; the value of q at first decreases, later it stays constant over the period of two weeks, which passes between the two phases of the experiment.¹⁰

3. Surface Breakdown

A new effect, transfer of charge from the electrode to the surface of the dielectric, sets in, when the applied voltage is sufficiently high and the temperature well above room temperature, or even when a very high voltage is applied at room temperature. This is shown in Fig. 5 giving the results of an experiment, in which 2500 v were applied at 50°C. Comparison with Fig. 3a shows various conspicuous differences: (a) The charge of the electrode repeatedly undergoes strong discontinuous variations. (b) The current now contains a steady state component; this is the ohmic current which appears at high temperatures. (c) The analysis of the current shows the existence of a conduction current between dielectric and electrode (by the inequality of J and dq/dt), which seems connected at least in part with the steady state current. The inset of Fig. 5 gives again the charge in function of time, but in such a way that the discontinuous variations of q are disregarded; thus after each jump the q values have been increased by an amount equal to the height of the jump.

We conclude that a jump gives evidence for an instantaneous breakdown of the interface.¹¹ With in-

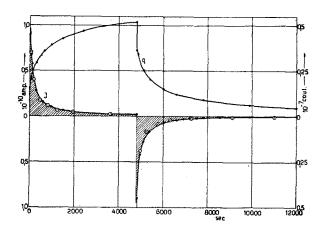


Fig. 3. Currents and charges for dielectric absorption. (Points: measured values; circles: calculated values of the displacement current.)

¹⁰ It has already been reported by Johnson and Carr, reference 3, that polarization with very low voltages yields electrets with heterocharges; for further measurements see Gemant, reference 3.

⁹ H. Frei and G. Groetzinger, reference 3.

¹¹ Since evidence for a crystalline structure of the electret has been found (M. Ewing, reference 3; A. Gemant, reference 3), a jump could possibly be thought of as being due to an adjustment of domains (see reference 4, p. 463). In the formal theory this effect is described by a discontinuous variation of the volume polarization P(t); thus it must be accompanied by a jump of J(t). But we have never observed discontinuous phenomena in the current-time curve except those caused by variations of the applied voltage.

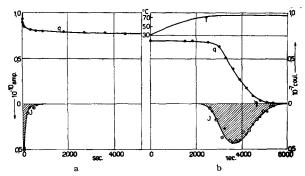


Fig. 4. "Freezing-in" of the absorbed charge (a) Current and charge after the short-circuit. (b) Current and charge during reheating, 2 weeks later. (Points: measured values; circles: calculated values of the displacement current.)

creasing polarization of the dielectric, the surface charges and, consequently, the field in the interface rise to high values. Finally the interface breaks down and a considerable amount of electricity is rushed from the electrode to the surface of the dielectric. This charge has a polarity, which is contrary to that of the original charge of the dielectric; it neutralizes part of the latter and thus reduces the field in the interface below sparking threshold.

During this experiment the density of the surface charge reached about 4.7×10^{-9} coul./cm² and the electric field 54,000 v/cm. But occasionally we have observed still higher values, in one instance a surface charge of 5.7×10^{-9} coul./cm², giving a field of 66,000 v/cm.¹²

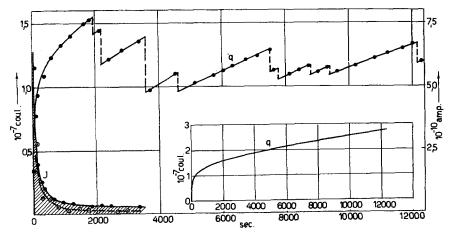
4. The Homocharge

The charge, which during a surface breakdown is transferred to the dielectric, does not dissipate at once. This can be inferred from the study of the behavior after short-circuit of a sample, where surface breakdown has occurred; (Fig. 6) (9800 v applied at 29°C for

2 hr.). Figure 6a shows that the behavior of J(t) and the slope of q(t) are very much the same as before (Fig. 3) and there is still i=0. But there is one significant new feature: the position of the q(t) curve relative to the vertical axis has changed, the whole curve being displaced downward by a constant amount; in consequence of this downward shift q undergoes a polarity reversal. Thus some time after the shortcircuit, the electrode which was connected with the positive pole of the voltage source, carries a negative charge and the other electrode a positive one, the polarities of the corresponding surfaces of the dielectric being, respectively, positive and negative. Figure 6b shows, that heating (22 hr. after the short-circuit) destroys these charges. But during this heating period there is $J\neq dq/dt$ and $i\neq 0$ over a considerable interval. This proves the occurrence of a continuous transfer of charge carriers between dielectric and electrode. The small external current flows in the same direction as during the period of application of the polarizing voltage. This is just what one has to expect when an ionic charge which during the application of the external field has gathered on the surface of the dielectric, dissipates by conduction within the dielectric (internal decay). But the fact that the measured current J is much smaller than the calculated displacement current dq/dt proves that only a small fraction of the ionic charge dissipates in this way; the bulk of the ionic charge dissipates by conduction across the dielectricelectrode interface (external decay). The value of i(t) is a measure for the rate of the external decay.

Thus one reaches the conclusion that at the moment of the short-circuit the dielectric carried two types of surface charges: (a) The charge s associated with dielectric absorption and (b) the charge f associated with surface breakdown. After the short-circuit, s decays according to the law of decay of the absorbed charge; since in the present case no heat treatment was

FIG. 5. Transfer of charge from the electrode to the surface of the dielectric during polarization. Inset: charge q(t); the curve is made continuous by adding at every jump the corresponding less of charge of the electrode. Circles: dq/dt.



¹² The role of breakdown phenomena has been emphasized by Mikola (see reference 3) and by Thiessen, Winkel, and Herrmann (see reference 3). Mikola's external polarization is to some extent the counterpart of the homocharge effect of the present paper, and his internal polarization is the counterpart of the heterocharge effect.

¹³ A two-charge theory was first formulated by Adams, reference 3.

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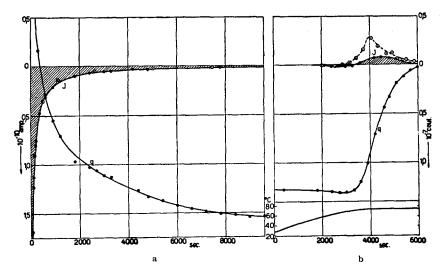


Fig. 6. Evidence for the existence of an ionic surface charge due to transfer of charge from the electrode to the dielectric. (a) Current and charge immediately after short-circuit. (b) Current and charge during heating, 1 day later. Circles: dq/dt.

given, s goes to zero. But f remains constant so long as the temperature is kept low. Thus when the heating started, only the charge f was left on the dielectric. The opposite polarities and different values of relaxation times of the two charges show that these charges are not of the same kind: s is due to a homogeneous volume polarization of the dielectric; f is an ionic surface charge. This explains why these charges coexist without mutual recombination. Since the sign of f is the same as that of the corresponding polarizing electrode, in Gemant's terminology f is a homocharge.

The induced charge of the electrode is now generally given by

$$q - q_0 = -(s + f),$$
 (4)

where q_0 is the charge of the geometrical capacitance. It is to be noted that the sign of f is contrary to that of s.

5. Coexistence of Homocharge and Heterocharge

Homocharge and heterocharge can be preserved together on the dielectric over long periods of time. For this purpose the volume polarization, and with it the heterocharge, must be frozen in by the usual heat treatment; the homocharge can be preserved even

without this treatment. Thus a sample was polarized with 950 v during 30 min. at 69°C; then the temperature was reduced to 35°C and the system was shorted. The behavior immediately after the shortcircuit does not reveal any novel feature, in particular no conduction current. A polarity reversal does occur, but later than in the foregoing experiment. Reheating started 19 hours after the short-circuit. It causes the appearance of a discharge current associated with the liberation of a frozen-in charge. But at the same time the absolute value of the induced charge of the electrode at first goes up, not down; later a maximum is reached and finally q tends to zero. During the period of increase there is still i=0, but shortly before the maximum of q is reached, a strong conduction current sets in, which soon predominates over the displacement current. No current reversal is observed (Fig. 7).

Interpretation in terms of the two-charge theory is straightforward. After the short-circuit a part of the absorbed charge decays, another bigger part remains frozen-in; the ionic charge remains undiminished. In consequence of the reheating, both charges decay, but the decay of the ionic charge begins later than that of

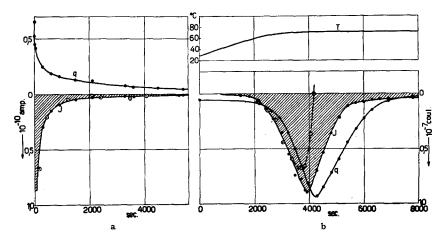


Fig. 7. Evidence for the coexistence of 2 types of charges on the surface of the dielectric. (a) Current and charge immediately after short-circuit. (b) Current and charge during reheating, 19 hours later. Circles: dq/dt.

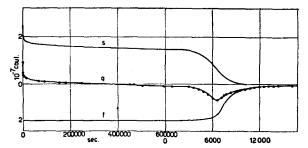


FIG. 8. Decomposition of the surface charge into homocharge f and heterocharge s. Reheating begins 600,000 sec. after the short-circuit. The time-scale for the heating period is changed.

the frozen-in volume polarization. Therefore at first only s decreases, while f remains constant; later f too decreases. This behavior, according to Eq. (4), gives the temporary increase of q shown in the measurement. The dissipation of f proceeds by continuous conduction currents across the dielectric-electrode interface and can be computed from the values of the conduction current i(t).

6. Computation of Heterocharge and Homocharge

The discharge current J(t) in the external circuit is due almost entirely to the decay of the volume polarization. Neglecting altogether the small contribution possibly arising from an internal decay of the homocharge, one obtains the value of the heterocharge s(t)at the time t from Eq. (3) as the integral over J(t) from 0 to ∞ . The value of the homocharge f then follows from Eq. (4) as the difference between the induced charge of the electrode and the value of the heterocharge. Such a computation is shown in Fig. 8 for an experiment similar to that just described, with a polarizing voltage of 2000 v applied at 65°C. It is worth mentioning that over a considerable period of time q is nearly zero; the current (not shown in the figure) is then very small also. An observer confining his attention to this interval, could easily conclude that "the electret has been discharged." But the analysis of the data in terms of heterocharge and homocharge shows that the measured value of q represents here as in many other cases but a small difference effect; both s and f exceed q by a considerable amount. It is in the range of possibility to avoid formation of one or the other of the two charges and to get in this way electrets, which produce much higher fields than those employed hitherto.

7. Behavior of an Uncovered Dielectric

The surface charge of an uncovered dielectric decreases rapidly after the electrode has been withdrawn. When the electrode is again brought into contact with the dielectric, the charge recovers. ¹⁴ This is shown in Fig. 9 for a sample which initially carried an ionic charge

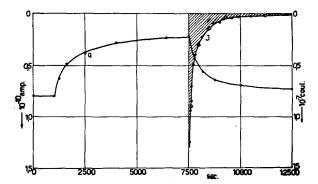


Fig. 9. Uncovering of a charged dielectric. The electrode is lifted at the point corresponding to 1000 sec. and is depressed at 7500 sec. (Points: measured values; circles: calculated values of the displacement current).

only. The reason for this behavior is found in the difference between the fields of the shorted and of the open electret. When the electrode is in contact with the dielectric, the field of the charge of the dielectric is concentrated mainly in the dielectric-electrode interface and the field in the interior of the dielectric is weak,15 When the electrode is removed, the situation is reversed and the main field of the charge of the dielectric is now concentrated in the interior of the dielectric. Thus removing of an electrode produces the same effect as does application of an external field; volume polarization and absorption occur. Accordingly new surface charges appear and partially neutralize the previously existing charges. But when the electrode system is reassembled, the primitive field distribution is reestablished and the field in the interior of the dielectric

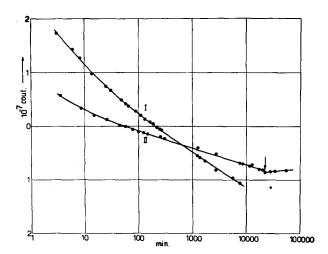


Fig. 10. Charge of the electrode against logarithm of time; I. Applied voltage 4500 v. II. Applied voltage 3000 v. At the point indicated by an arrow, the sample was reheated until the volume polarization had been destroyed.

 $^{^{14}\,\}mathrm{Such}$ effects have already been observed by M. Eguchi, reference 3.

¹⁵ The precise values of the field at every point depend on the thickness of the interface, the ratio of the dielectric constants of interface and dielectric, and the space charge distribution in the dielectric; but for the following qualitative considerations the knowledge of these precise values is not essential.

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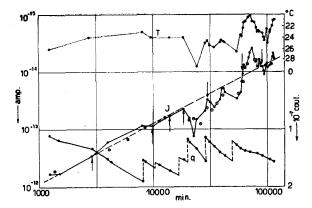


Fig. 11. Current, charge, and temperature of a sample polarized with a high voltage. The measurements extend over 80 days. The arrows indicate current values corresponding to a temperature of 24°C. Circles: dq/dt.

is reduced to its former low level. This is similar to the short-circuiting of a previously energized capacitor. The ensuing decay of the recently formed polarization restores the initial high value of q; it manifests itself also by the appearance of a discharge current J(t) of the same type as that observed after the short-circuit of an absorptive capacitor. Figure 9 shows this discharge current, which strongly confirms the ideas outlined here.

8. Decay of the Polarization of the Dielectric

Figure 10 gives for two experiments the value of qagainst the logarithm of time. In one case 3000 v were applied at 56°C during 10 min., in the other 4500 v at 67°C during 150 min.; in both cases the system was shorted when room temperature had been reached. The systematic variation of q was observed over several weeks; one and the same effect, the slow decay of the frozen-in charge, manifests itself over this period of time. In one of the experiments, the sample was reheated after 16 days of observation for a short time and only as much as was necessary for removing the frozen-in heterocharge. From this moment on (indicated in the figure by an arrow), the trend of q(t) is different; the increase of q now has given way to a very slow decrease obviously caused by a slow decay of the homocharge which after the short reheating had been left alone on the dielectric. This decrease was observed for nearly four weeks.

Finally Fig. 11 gives the result of an experiment, in which 9,000 v were applied at 71°C for 3 hours; the system was shorted, when room temperature had been reached. These measurements extend over 80 days. In a logarithmic time scale, $\log J$, the temperature, q and dq/dt, are given. The current undergoes a series of fluctuations. Inspection shows that they are correlated with fluctuations of the temperature and, therefore, caused by the enormous value of the temperature coefficient of the discharge current. When the current

values corresponding to a temperature of 24°C are selected (in the figure pointed out by arrows), they define with good approximation a straight line, with a slope equal to 1. Thus during all this time the current obeys the well-known, and frequently questioned, power law. The charge q at first increases regularly just as happened in the two former experiments. But when the intensity of the field in the interface attained a sufficiently high value, several discontinuous variations do occur. The slope of the curve q(t) coincides amazingly well with the measured values of J(t), following all the irregularities of the latter. Thus except during a surface breakdown there is no indication of conduction across the interface in spite of the high value of the field existing there. Thus the ionic charge does not dissipate unless breakdown occurs, and this is a rather rare event. One must conclude, that at room temperature a strong potential barrier impedes the transfer of carriers between dielectric and electrode; the height of the barrier is reduced at elevated temperatures.

III. SUMMARY

Our experiments lead us to the following conclusions: (a) The heterocharge is due to a volume polarization of the dielectric. 16 It is preserved, because this volume polarization can be frozen in. (b) The homocharge is an ionic surface charge caused by surface breakdown. (c) The sum of heterocharge and homocharge gives the free surface charge of the dielectric. This free charge together with the corresponding induced charge of the electrode constitutes an electric double layer. (d) At room temperature the double layer does not disappear by surface conduction, because the potential barrier at the surface of the electrode then prevents transfer of charge between dielectric and electrode; it does not decay by volume conduction, because it does not set up an electric field within the dielectric, provided its thickness is small compared with the thickness of the sample. (e) At elevated temperature continuous conduction currents flow between dielectric and electrode. because the height of the potential barrier is reduced, (f) The electric moment of the double layer is limited by breakdown phenomena occurring in the dielectricelectrode interface.

I am greatly indebted to the Director of the Institute, Professor E. L. da Fonseca Costa, who made this study possible.

¹⁶ We do not in general exclude the possibility of an ionic effect and of space charges of the type found by Jaeger (reference 3) and Gemant (reference 3). This effect will contribute to the heterocharge. But as Gemant has already pointed out, the time constant for the decay of the space charges will be much smaller than that for the decay of the frozen-in volume polarization, in consequence of the finite volume conductance of the dielectric. Therefore the permanent component of the heterocharge of the present paper must be due to a volume polarization. Unpublished measurements about discharge currents, in which the surface layers on both sides of the samples were mechanically removed before the sample was reheated, prove this point of view.