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SECOND REPORT ON THE THERMAL EVALUATION OF FORMVAR RESIN

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ABSTRACT

The purpose of this work is to develop a method of evaluating electrical insulating materials with the end in view of being able to predict the life of an insulation when operated at a given temperature.

The method described herein involves the determination of the electrical properties of Formvar Resin at various temperatures after aging under special conditions. Numerous plots of data are given to illustrate the changes in electrical properties which Formvar Resin undergoes with time.

AUTHORIZATION

The thermal evaluation of electrical insulating materials described in this report is part of a project to study and evaluate electrical insulating properties of materials now used and proposed for use as insulating materials. This work is authorized by BuShips letter S62-2(335) SRD-346/46, dated 3/11/46, authorizing the development of new and improved methods of evaluating electrical insulations.

PROBLEM STATUS

This is an interim report on the problem; work is continuing.

SECOND REPORT ON THE THERMAL EVALUATION OF FORMVAR RESIN

Statement of the Problem

It is the purpose of this paper to present the results of a series of studies on the changes which certain vinyl polymers undergo at elevated temperatures (105-225° C); the ultimate object of the tests aims to develop a non-destructive method of evaluating the life expectancy of an electral insulant. Earlier work on this general problem was reported in Naval Research Laboratory Report P-2915.*

The reasons for using an insulating varnish in electrical apparatus are threefold; a varnish confers mechanical strength, electrical strength, and moisture resistance on the assembly. A material may be mechanically poor but electrically good, e.g., bentonite films, or vice versa, cement. Again a material may be mechanically and electrically good, but only when dry, e.g., cellulose paper. Any attempt, then, to correlate the characteristics of a dielectric with the life expectancy of the material must take cognizance of the mechanical, chemical, and electrical properties as a concatenated whole.

Previous Methods

The employment of dielectric strength as a measure of the suitability of an insulant suffers precisely from the lack of correlation with mechanical effects.† "Both time and temperature have the effect of decreasing the mechanical strength of insulations, eventually resulting in its becoming brittle, and causing it to disintegrate. Class A insulation does not fail by electrical breakdown upon arrival of some critical temperature but by mechanical deterioration. Hence the electrical strength of insulation cannot be correlated with its mechanical strength." ‡

Other investigators,§** after assuring themselves of the suitable characteristics of a material, have studied the effects of impurities on the substance by changes in power factor or tensile strength. In some cases correlations have been found between the change

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* Bukey, J. R., NRL Report P-2915, "Thermal Evaluation of Formvar Resin," 29 July (1946)

† Montsinger, V. M., "Thermal Characteristics of Transformers," Gen. Elec. Rev. 49; 31 April (1946)

‡ Knowlton, A. E., Standard Handbook for Electrical Engineers. McGraw-Hill, 1941. Section 6 "Transformers, Regulators and Reactors" pp. 537-632

§ Piper, J. D., et al. "Liquid Dielectrics," Ind. Eng. Chem. (Ind. Ed.), 31; 307 (1939)

** Clark, F. M., "Dielectric Stability of Mineral-Oil Treated Insulation," Ind. Eng. Chem. (Ind. Ed.), 31; 327 (1939)

in power factor with time and the life expectancy of the insulation. These are usually exceptional cases for the power factor, as a measure of loss, has meaning only when the dielectric coefficient is constant.*

Tensile, compressive, flexural and abrasive strengths of materials have served as qualitative aids in evaluating new varnishes and adhesive coatings;† they all suffer from the totally destructive character of the test.

METHODS

Properties of Polyvinyl Formal

The material employed for this work was a commercial polyvinyl formal produced by Shawinigan Company called Formvar (15/95) "E." "The starting point in the preparation of this material is vinyl acetate. In the preparation of polyvinyl formal, polyvinyl acetate is hydrolyzed in the presence of acetic acid. Considerable time elapses after the aldehyde has been added and the polyvinyl formal precipitated with water. In the final product there are various proportions of reagents, the extent of each being controlled by the conditions of the reaction."‡ Thus, for example the material employed here was claimed by the manufacturer to be 95 percent formal and the remainder ester and alcohol. P-ter-amylphenol§ when added to the reaction mixture during acetalation prevents discoloration of the products. Some of this material may be found absorbed on the polyvinyl formal.

The polyvinyl formal has the following properties:**††

TABLE I

Specific gravity	1.2-1.3
Refractive index	1.5
Heat distortion point	71-77° C
Water adsorption	0.6-1.3 percent
Solubility:	Soluble in tetrachlorethane and ethylene tetrachloride
	Slightly soluble in dioxane and toluene-butanol
	Very slightly soluble in the higher ketones
	Insoluble in alkalies and hydrocarbons

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* Race, H. H., "Capacitance and Loss Variations with Frequency and Temperature in Composite Insulation," Trans. Am. Inst. Elec. Engrs. 52; 682 (1933)

† Mulvey, B., "Process Testing of Film Continuity on Formex Fine Wire," Gen. Elec. Rev., 49; 46 (Nov. 1946)

‡ Delmonte, J., Plastics in Engineering, Penton Pub. Co. (1942)

§ Wakeman, R. L., The Chemistry of Commercial Plastics, Reinhold (1947)

** Plastic Materials Manufacturers' Assoc., Tech. Data on Plastics (1945)

†† Hippel, A., et al. "Tables of Dielectric Materials," MIT, Lab. for Insulation Research (NDRC, Div. 14), Report V., III (1944)

Tensile Properties:

°C	Tensile Strength	% Elongation
-30	12,000 psi	4
0	10,000	4
60	2,500	7
80	200	20

Dielectric Strength: 1200-1600 volts per mil (vpm) 0.003" sample; short time; 1/8" electrodes.

Examination of these data indicates that the material suffers serious plastic distortion at 71-77° C eventuating in it losing over 90 percent of its tensile strength accompanied by a five-fold increase in elongation. Under such conditions the viscosity of the plastic has changed appreciably and an appreciable change in the electrical constants is to be expected. This is shown later in Figure 1.

Preparation of Films

To 500 ml of Eastman tetrachlorethane, 28 grams of Shawinigan Formvar "E" were added slowly with vigorous agitation at room temperature. The dispersion was filtered through cheesecloth to remove the undissolved particles and then poured into a freshly cleaned, dry glass plate, 900 sq cm in area. The solvent was permitted to evaporate overnight at room temperature. The plate with the tightly adhering film was then submerged in a pan of water, and after the film separated, it was dried between filter papers and permitted to come to room temperature at equilibrium conditions. Films prepared in this way were found to be 0.005 ± 0.0005 cm in thickness over an area of 300 sq cm. Microscopic examination at 430X indicated a fairly continuous pellicle possessing no birefringent particles or gelled areas.

Determination of Electrical Characteristics

A piece of aluminum foil 4 cm in diameter and .008 cm in thickness was covered on one side with a homogeneous section of a freshly cast and dried film and warmed slightly to permit the plastic to fashion itself snugly against the metal. A silver electrode 2.5 cm in diameter was then painted on the plastic from a dispersion of Dupont No. 4133 silver paste in absolute alcohol. The composite was placed in an Elliott cell* and thoroughly dried at 105° C for two hours; the temperature was then lowered to -60° C by using a mixture of dry ice and isopropyl alcohol. The audio-frequency characteristics were determined on a General Radio 716-AR Capacitance Bridge employing a Hewlett-Packard 200-BR Oscillator as a frequency source and a General Radio 814-A Amplifier for the RCA-155 Oscilloscope used as a detector.

DATA OBTAINED

Electrical Properties of Polyvinyl Formal

The variation of the dielectric constant (hereafter called dielectric coefficient) of polyvinyl formal with temperature is shown graphically in Figure 1. A rapid change in

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* Elliott, M. A., et al. "Dielectric Identity Test for Plasticizers," Ind. Eng. Chem. (Anal. Ed.) 19, 10 Jan. (1947)

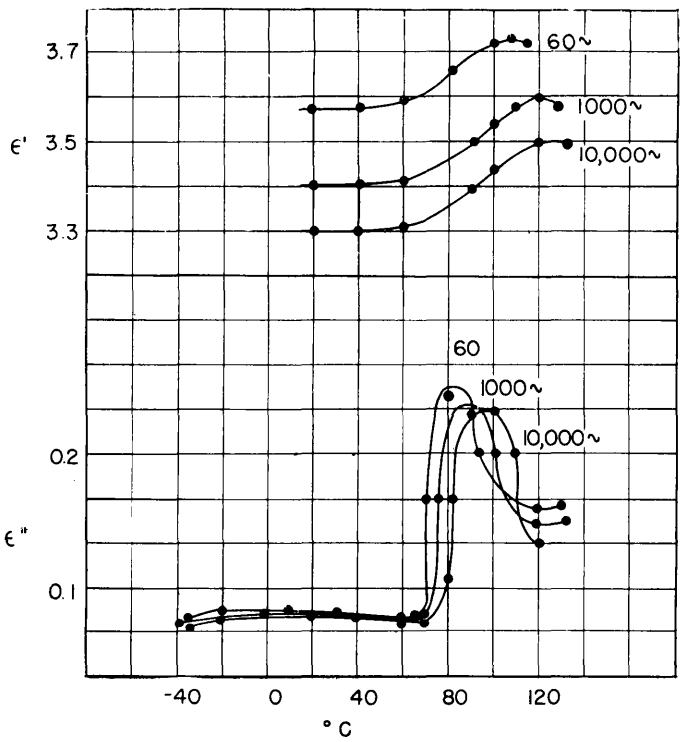

Fig. 1 - Electrical Properties of Polyvinyl Formal

the dielectric coefficient, ε' is observed in the region 70-90° C, which is the same temperature range at which the viscosity of the plastic changes abruptly. The loss factor, ε'' rises to a maximum corresponding to the primary absorption peak at the frequencies indicated on the curves. Incorporation of tri-cresyl phosphate in the film reduces the viscosity of the plastic appreciably, resulting in a shifting of the loss-factor maximum to lower temperatures; simultaneously a broadening of the curve is observed as shown in Figure 2. Although the last figures do not distinguish more than one absorption peak in each loss-factor curve, closer examination, as shown in Figure 3, indicates that at lower temperatures and frequencies the losses tend to reach a maximum at about -20° C and that they recede to a minimum at 72° C. This phenomenon has been described by Garton* as occurring in glycol-phthalate resins,† in shellac and in polymerized glyptal. He failed to observe the absorption peak in rosin or in unpolymerized glyptal. Fuoss‡ has developed an explanation which was applied successfully by Busse§ to the interrelation of mechanical and electrical properties of polyvinyl chloride. Methyl methacrylates also undergo this phenomenon.**

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* Garton, C. G., "The Dielectric Characteristics of a Chemically Pure Synthetic Resin," J. Inst. Elec. Engrs., 85; 625 (1939)

† Kienle, R. H., and Race, H. H., "The Electrical, Chemical and Physical Properties of Alkyd Resins," Trans. Electrochem Soc., 65; 87 (1934)

‡ Fuoss, R. M., "Electrical Properties of Solids," J. Am. Chem. Soc., 63; 381 (1941)

§ Busse, W. F., et al. "Dielectric Properties of Elasticized Polyvinyl Chloride," J. Am. Chem. Soc., 63; 361 (1941)

** Morgan, S. O., and Yager, W. A., "Dielectric Properties of Organic Compounds," Ind. Eng. Chem. (Ind. Ed.), 32; 1528 (1940)

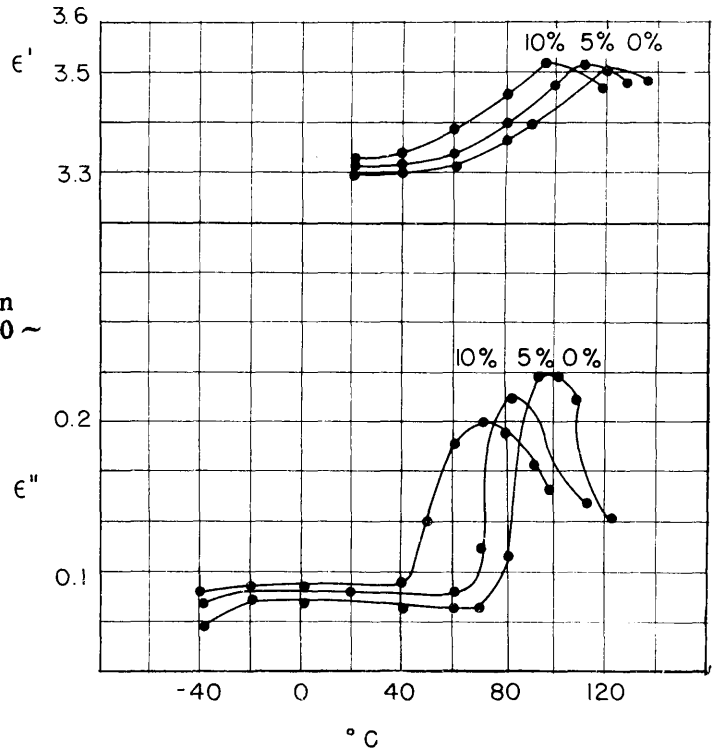

Fig. 2 - Effect of Plasticizer on Polyvinyl Formal at 10,000 ~

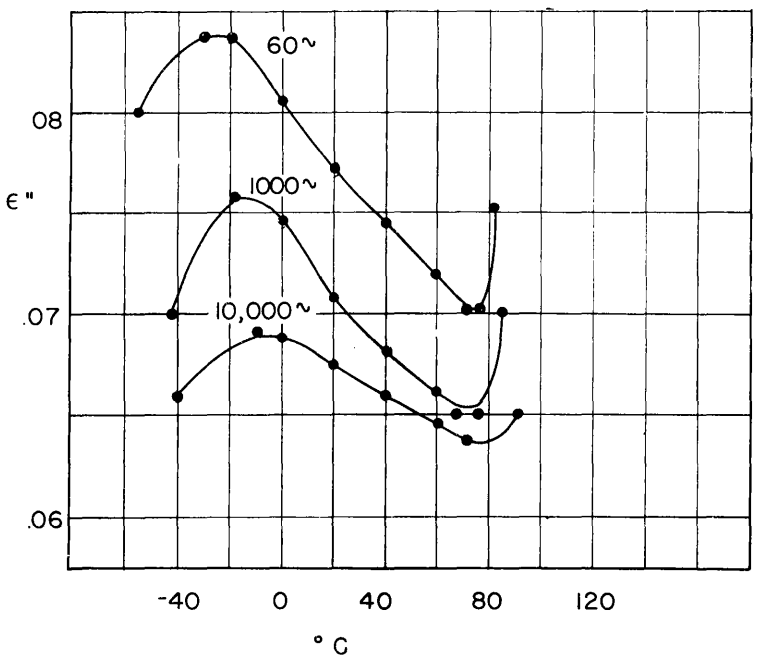

Fig. 3 - Low Temperature Absorption in Unaged Polyvinyl Formal

The minimum loss-factor value which occurs in the region 70-80° C corresponds to the brittle point or the internal melting point of the plastic. Such a point is of interest because it is a direct link between the mechanical and electrical properties of plastics. The brittle point-loss factor relationship has also been observed in cellulose.*

The relatively high value of the dielectric coefficient of polyvinyl formal indicates the presence of permanent dipoles. Debyes theoretical loss factor† is defined as

$$\epsilon''_{max} = \frac{\epsilon_0 - \epsilon_\infty}{2} = 0.72$$

where: ϵ''_{max} = maximum value of the loss factor

ϵ_0 = static dielectric constant

ϵ_∞ = dielectric constant at optical frequencies.

The range of molecular sizes, however, effectively reduces the loss factor at maximum absorption to a value of 0.25 which is a value about one-third that of the theory. The relaxation time, τ', of the dielectric is defined by the expression‡

$$\epsilon'' = (\epsilon_0 - \epsilon_\infty) \frac{\omega \tau'}{1 + (\omega \tau')^2}$$

where: ϵ'' = measured loss factor

ω = 2π times the frequency

τ' = relaxation time of the dielectric.

The value of τ' is 2.5×10^{-3} sec. The relaxation time of the dipoles is expressed as follows

$$\tau = \frac{1}{\omega \epsilon''} \frac{\epsilon_\infty + 2}{\epsilon_0 + 2} \left(\epsilon''_{max} \pm \sqrt{\epsilon''^2_{max} - \epsilon''^2} \right) = 2.10^{-3} \text{ sec.}$$

These are very slow rotational periods which may be atttributed to the unevenly substituted character of the polymer.

Electrical Properties of Aged Films

The properties of the polyvinyl formal disclosed thus far are those of freshly cast films of the material. The effect of aging the pellicles over a period of 24 hours at 175°C in an air oven changes the values of these constants appreciably as shown in Figure 4.

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* Kohman, G. T., "Cellulose as an Insulating Material," Ind. Eng. Chem. (Ind. Ed.) 31; 807 (1939)

† Debye, P., Polar Molecules, Chem. Cat. Co. (1929)

‡ Murphy, E. J., and Morgan, S. O., "Dielectric Properties of Insulating Material," Bell System Tech. J. 17; 642 (1938)

The dielectric coefficient is seen to increase appreciably with aging and the position of the loss-factor peak is observed to shift to higher temperatures. Concomitantly the minimum loss-factor point rises, indicating that the material remains in a non-plastic or embrittled state at higher temperatures; the rise of this point is therefore a measure of the increasing brittleness of the film. Since the life of an electrical insulation is related to the mechanical* embrittlement of the pellicle, the rate of change of the minimum loss-factor temperature is a measure of the life expectancy of the insulation. Any electrical test which can be correlated with this change in brittle point should be a measure of the life expectancy of the insulation.

The work of innumerable investigators† has shown that power factor determined under standard conditions results in, at best, a scattering of points. On the other hand, the determination of capacitance offers fewer interpretive difficulties. Figure 4 indicates that there is an appreciable change in the dielectric coefficient with aging, as well as a change in loss factor. The low-temperature absorption peak also shifts markedly, as shown in Figure 5, where the increase in the height of the curve is a measure of the loss of plasticizer or entrapped solvent, and the increasing abruptness of the minimum loss-factor value indicates the rising embrittlement of the plastic. The simultaneous changes observed, viz, the increase in dielectric coefficient with age and the increasingly high value at which the internal melting of the plastic occurs, are shown graphically in Figure 6. Since the rate at which the brittle point reaches a maximum

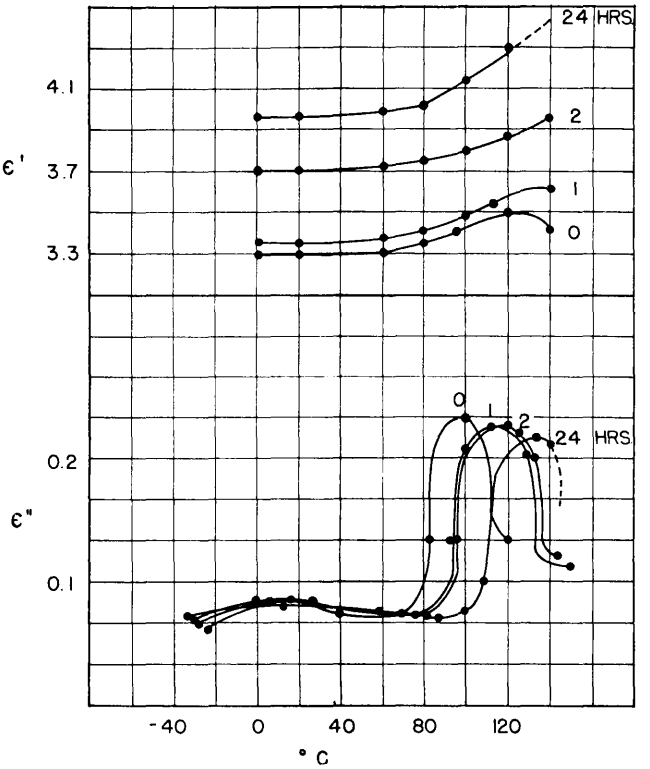

Fig. 4 - Effect of Thermal Aging (175° C) on Polyvinyl Formal at 10,000 ~

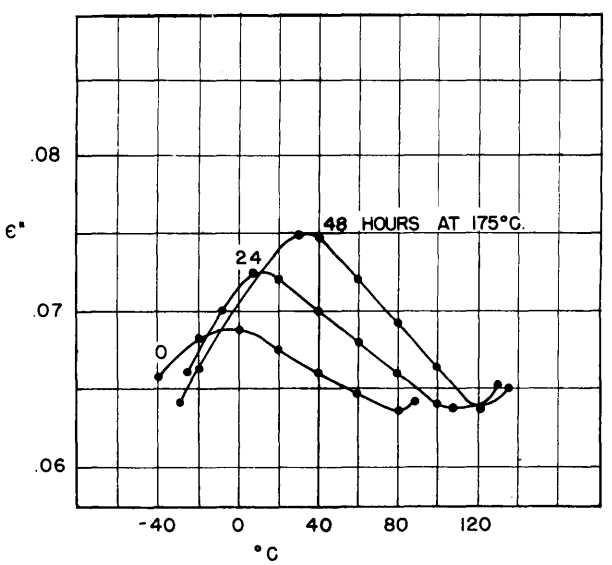

Fig. 5 - Effect of Aging on the Low Temperature Absorption in Polyvinyl Formal - 100,000 ~

* Billig, E., "Mechanical Stresses in Transformer Windings" J. Inst. Elec. Engrs. 93; 227 (1946)

† Debing, L. M. "Dielectric Properties of Phenolic Resins," Trans. Electrochem. Soc. 90; 24 (1946)

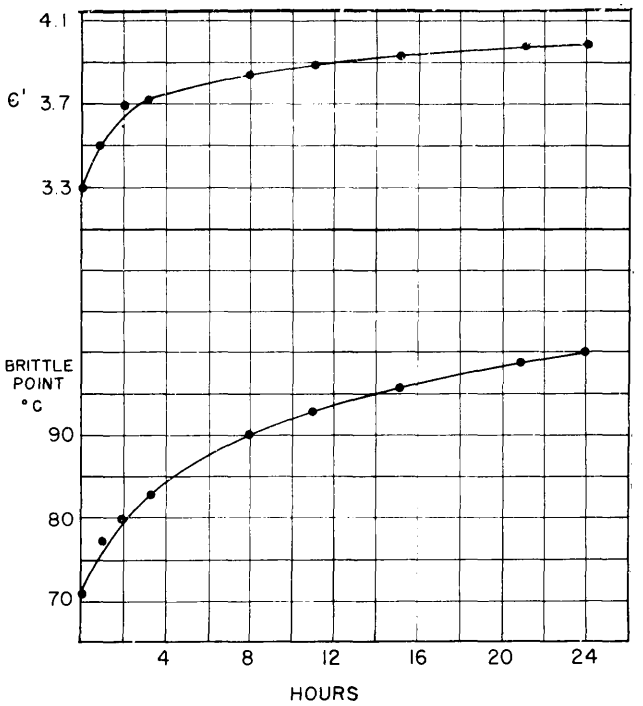

Fig. 6 - Effect of Thermal Aging (175° C) on Polyvinyl Formal

The physical properties of the films were:

TABLE II

Thickness	.001- .003 inches
Specific Gravity	1.2 - 1.3
Tensile Strength (25° C)	7000 psi
Elongation	125 percent
Color	Colorless - Yellow
Transparency	High
Dielectric Strength	1200 - 1800 vpm

The dielectric coefficients and loss factors of this material at various frequencies and temperatures are given in Figure 7. The loss factors are broad and appear at relatively low temperatures for the basic component which is polyvinyl formal. These phenomena indicate the plasticizing effect of the cresol and this idea is substantiated in Figure 8, where the gradual change in position of the loss-factor peak is observed to rise appreciably accompanied by a large increase in dielectric coefficient. The dielectric coefficient is found to increase with is dependent on the quantity and volatility of the plasticizer, it would appear that the incorporation of a stable plasticizer of low volatility and high boiling point would lead to a plastic of high flexibility.

Properties of Formex G. E. Enamel - 9825

A sample of the commercial material employed as an enamel in the manufacture of Formex magnet wire was secured as an example of a commercial formula employing polyvinyl formal as a base and containing high-boiling plasticizing agents.

Films were cast from the varnish on glass in a manner similar to that employed in making polyvinyl formal pellicles. When thin films were required, tetrachlorethane was employed as a diluent. The films were air-dried at room temperature to prevent the interaction of the cresylic acid with excess formaldehyde.

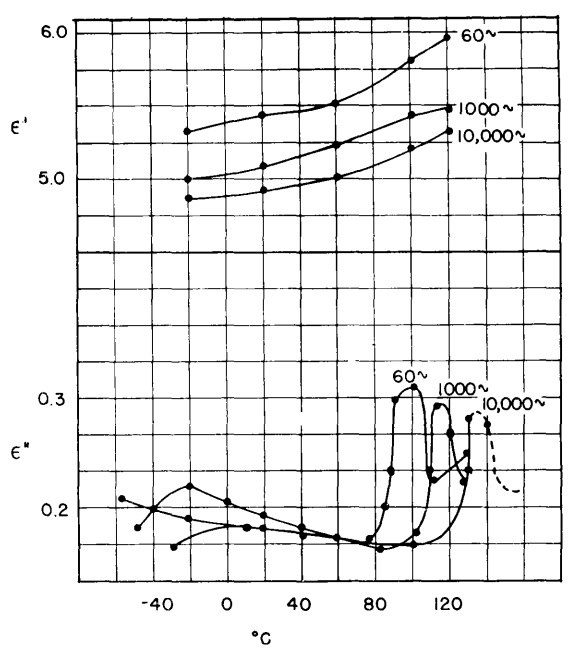

Fig. 7 - Electrical Properties of G. E. Formex Enamel No. 9825

aging and approaches a final value according to a parabolic relationship as shown in Figure 9. The Brittle Point (B.P.), as determined by loss-factor methods, also increases with time of aging at 225° C (Figure 9) according to the relationship,

$$B.P. = 108 + 3.75t - 0.185t^2 + 0.003t^3$$

where t is the time in hours.

The first derivative,

$$\frac{d(B.P.)}{dt} = 3.75 - 0.37t + 0.009t^2 = 0,$$

specifies the time t, at which the loss-factor minimum reaches its maximum temperature. On solving this expression, t = 20+ hours, which is an indication that the embrittlement of the varnish is complete in 20 hours at 225° C. This compares well with the value obtained by the more familiar bending tests.

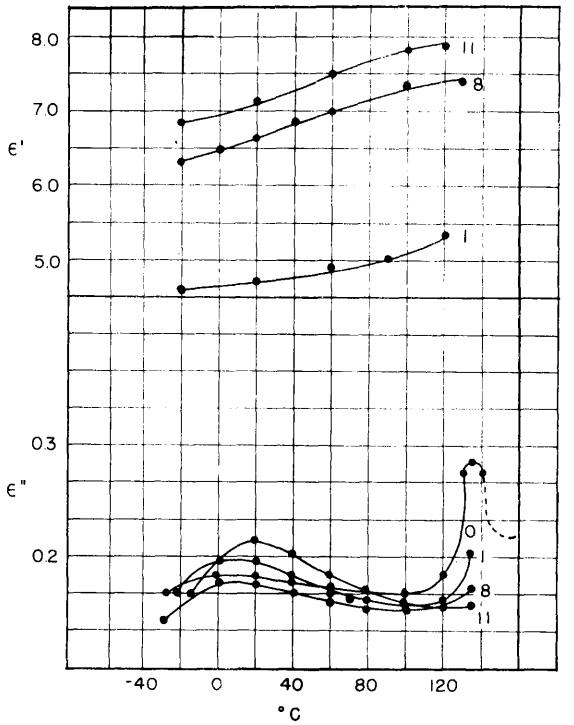

Fig. 8 - Effect of Thermal Aging on G. E. Enamel No. 9825 at 10,000~

The dielectric coefficient may be examined at various temperatures after aging at 225° C and is plotted in Figure 10. From these curves, the dielectric coefficient at any time after aging at 225° C may be expressed by the relation,

$$\epsilon'^2_{225} = 2t + 0.08\,K. \qquad (1)$$

where t is the number of hours at 225° C and K is the temperature (in degrees absolute) at which the dielectric coefficient is determined, the frequency being 10,000 cycles per second. A similar relationship may be determined empirically for the aging at 175° C (Figure 11). This was found to be

$$\epsilon'^2_{175} = 0.14t + 0.08\,K. \qquad (2)$$

It follows from this that a constant frequency the value of the constant A in the equation

$$\epsilon'^2 = At + B$$

is a measure of the rate of change of the dielectric coefficient with aging, and B is the temperature parameter. Differentiating

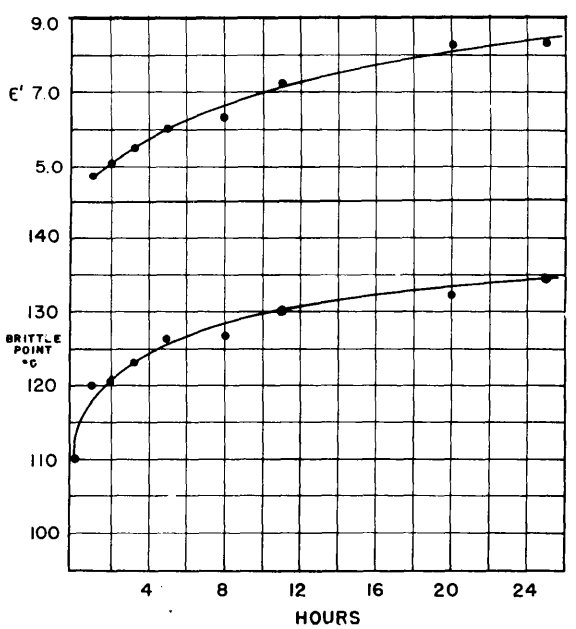

Fig. 9 - Effect of Thermal Aging (225° C) on the Dielectric Constant Brittle Point of G. E. Enamel 9825 at 10,000~

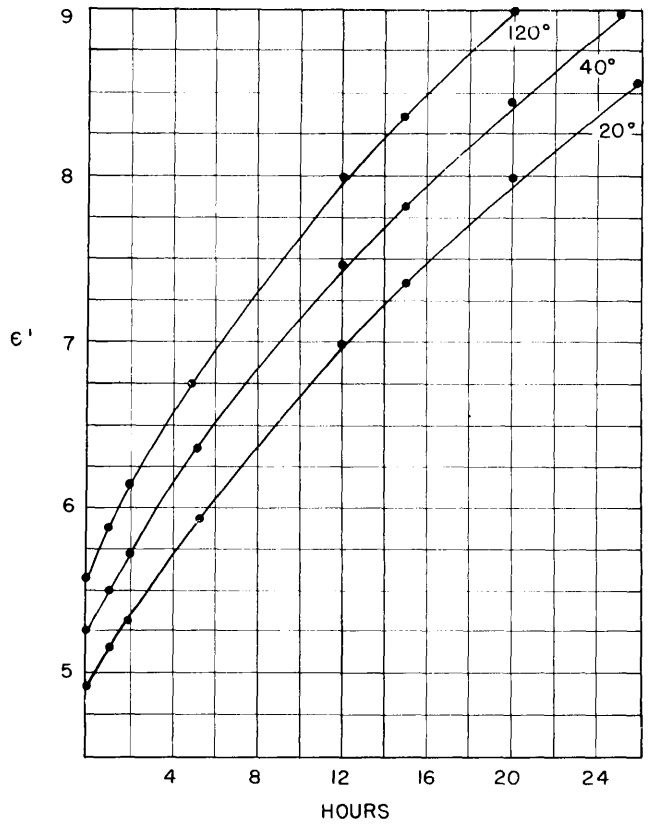

Fig. 10 - Effect of Thermal Aging on G. E. Enamel at 225° C - 10,000~

equations (1) and (2) and comparing the slopes, it becomes apparent that after t hours of aging at 125° C, the dielectric coefficient may be represented as

$$\epsilon'^{2}_{125} = 0.01\,t + 0.08\ K. \qquad (3)$$

Work on the experimental verification of equation (3) is underway at the present time.

The brittle point-vs-time curve (Figure 9) indicates that the varnish has reached its mechanical embrittlement after 20 hours of aging, at 225° C, or when a dielectric coefficient of 8 is reached. On solving for t in equation (3) at $\epsilon' = 8$, it is found that the varnish should be embrittled in 4,000 hours when maintained at a temperature of 125° C. This is the average value secured by G. L. Moses* for Class A insulation at 105° C, and is estimated to remain suitable as an insulator for a period of 20 years when maintained below 60°C.

CONCLUSIONS AND RECOMMENDATIONS

Mechanical Properties

In order to evaluate the phenomenon of increasing dielectric coefficient, it is well to compare the qualities of a freshly cast film with those of an aged coating. A freshly cast

* Miner, D. F., Insulation of Electrical Apparatus, Appendix B, McGraw-Hill (1941)

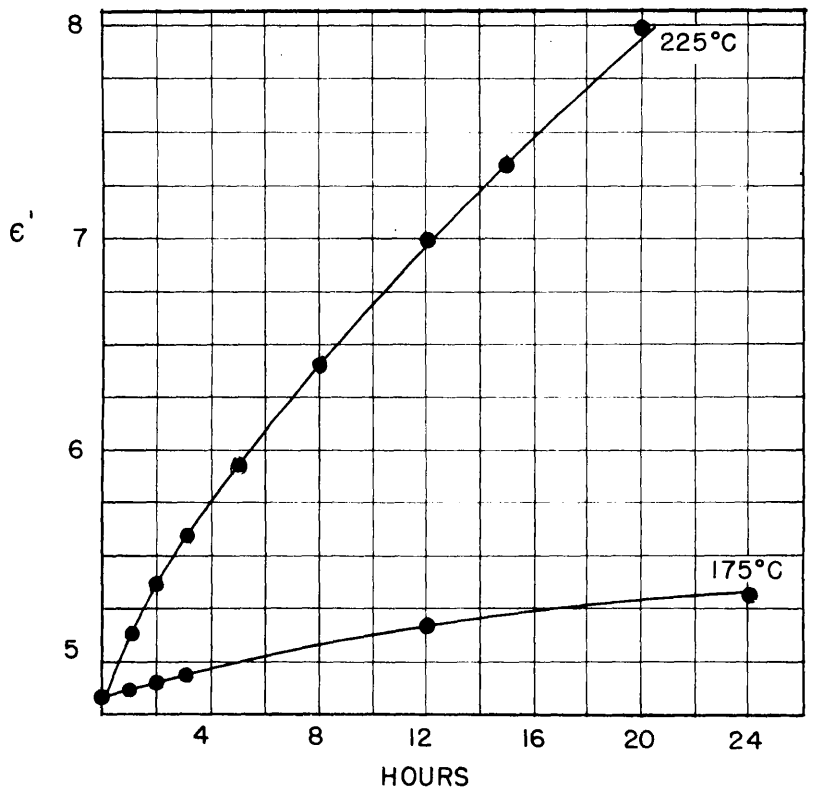

Fig. 11 - Effect of Thermal Aging on
G. E. Enamel 9825 at 225° C - 10,000~

film, whether or not it contains plasticizer or entrapped solvent has a flexibility not to be found in an aged coating. With aging, a film increases in tensile strength slightly and continues to possess this value until it shatters through embrittlement. The elongation of a dry film declines rapidly and indicates practically a zero value of stretch in spite of its flexibility remaining high.* Finally, it is known that the film cannot be bent through a large angle as time progresses, and in fact, after some time, the film shatters on being handled.

Theory

One explanation for these phenomena, which has the advantage of explaining all three at once, considers a freshly cast film as a very viscous liquid capable of being distorted according to the laws of plastic flow. In this case tensile strength is a measure of the adherence of the weakest points of contact of adjacent molecules. Elongation is a measure of the sliding or viscous character of the mass, and flexibility a measure of the random-orientation of the particles.

As time passes - and it may be assumed that no chemical change in the ordinary sense occurs - the plasticizers volatilize and some of the molecules tend to lie side by side. When this phenomenon obtains, the laws of plastic flow no longer act independently of Newtonian mechanics. The alignment of particles eventuates in shrinkage, hence in an increase in

* Mulvey, B., "Process Testing of Film Continuity on Formex Fine Wire," Gen. Elec. Rev., 49; 46 (Nov. 1947)

density and tensile strength; it results in a loss in elongation since there are a greater number of contact-points and hence an increase in adhesion. The flexibility is lessened simultaneously because rod-like or gelled structures are forming in the mass.

Electrical Properties

Although the qualities of tensile strength, elongation, and flexibility find no ready reference to the electrical properties of a plastic, the phenomena of shrinkage, change in contact angle, and formation of oriented sectors are suited for study in terms of classic electrostatics.

The increase in density which occurs when a freshly cast film shrinks, results in an increase in polarization according to the well-known relationship of Clausius and Mosotti. This may also be described by saying that an increase in the number of dipoles in a unit volume effectively increases the static dielectric coefficient of a material in an alternating electromagnetic field. Finally, the presence of particles of gelled structure gives rise to absorption phenomena classed as Maxwell-Wagner polarization.*†

Shrinkage accounts for an increase of 18 percent in the value of the dielectric coefficient. This increase is rapid since the material reaches its final limits within 5 hours at 175° C.

This factor does not explain fully the relatively large increase in dielectric coefficient which polyvinyl formal suffers on aging. An increase in polarization by the formation of new dipoles having greater Debye values, changes in the relaxation time of the polymer and a change in the molecular weight of the material must also be considered. The presence of the unreacted hydroxyl (-OH) group in the polyvinyl chain offers a point for oxidative attack to a carbonyl or carboxyl group. The Debye values for these complexes are actually lower than that for (-OH). Rupture of the formal-ring structure to yield carbonyl and carboxyl groups would eventuate in an increase in over-all dielectric coefficient. That aldehydes are split from polyvinyl formal has been reported and utilized in the manufacture of commercial Formex.‡ The limiting dielectric coefficient would then be that of the original polyvinyl alcohol. Another reason for the increase in dielectric coefficient with aging relates to the degradation of the vinyl chain eventuating in shorter lengths and hence ones with shorter periods of relaxation. This would result in more chains being capable of following the filed if a distribution of the Wagner-Yager† type be supposed. This latter concept is substantiated by the gradually changing position of the primary absorption peak to higher temperatures.

Limitations

The basic limitations of these correlations lie in the inter-dependence of the change in dielectric constant with age and the change in brittle point with age. Two equations

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* Murnaghan, F. D., "Maxwell's Theory of the Layer Dielectric," Trans. Am. Inst. Elec. Engrs., 46; 259 (1947)

† Yager, W. A., "Distribution of Relaxation Times in Typical Dielectrics," Physics 7; 434 (1936)

‡ Patnode, W., et al. "Synthetic Materials as Wire Insulation," Ind. Eng. Chem. (Ind. Ed.) 31; 1063 (1939)

have been derived, viz,

$$\epsilon'^2 = 0.01\,t + 0.08\ K$$

$$B.P. = 108 + 3.75\,t - 0.185\,t^2 + 0.003\,t^3$$

The first equation is a measure of the over-all changes which occur in G. E. Enamel 9825 and includes:

1. change in Number of Dipoles per unit volume attributable to (a) densification and to (b) chemical change.

2. change in relaxation time of the molecule attributable to changes in (a) molecular weight and (b) intrinsic viscosity.

On the other hand the brittle point is a measure of:

1. point at which the electrical losses contribute least to the dielectric coefficient (losses at a minimum).

2. the point at which the intrinsic viscosity changes abruptly.

Other limitations include the effect of age on non-polar materials. The brittle point and dielectric constant are related in that,

$$\epsilon'_{B.P.} = \epsilon + j\,\epsilon''$$

where $\epsilon'_{B.P.}$ is the dielectric coefficient at the temperature at which ϵ'' reaches a minimum value, ϵ is the complex dielectric constant and j is $\sqrt{-1}$. It is the fact that both dielectric coefficient and loss factor are related to intrinsic viscosity that the former may be employed as a measure of the life expectancy of the insulation.

Recommendations

1. It is recommended that evaluations of dielectric coefficients of polyvinyl acetate, polyvinyl alcohol, and polyvinyl formal (cp) be made after aging in nitrogen and in steam at lower temperatures and less drastic conditions.

2. It is also recommended that the changes which occur in a disc prepared from a melt rather than cast from a solution be studied. This would eliminate entrapped solvent.

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PRNC-4364-1-23-48-100