

A dielectric loss investigation of moisture in epoxy-glass composites

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Dielectric loss has been used to study moisture absorption-desorption equilibria and kinetics in epoxy-glass composites. Measurements were made at a temperature of 90°C and a frequency of 200 Hz to maximize sensitivity to interfacial or Maxwell-Wagner polarization. Exposure to various partial pressures of moisture at that temperature permitted a kinetics analysis from which an estimate was made of the diffusivity of water in the composites. Values of dielectric loss at saturation were used to establish a moisture/dielectric loss calibration at 90°C which can be used to estimate the macroscopic internal moisture content of coupon samples and printed circuit cards and boards. Dielectric loss measurements offer promise as a screening technique for resin-glass coupler development.

Introduction

Field-induced polarization processes are often characterized by the frequency at which relaxation losses are maximized, i.e., when they are in phase with the applied alternating field. In heterogeneous materials, an additional type of polarization occurs—interfacial or Maxwell-Wagner

polarization [1]. Such polarization is viewed as the accumulation of charge at the interface between phases when the dielectric constant and the conductivity of each phase are unequal. Conceptually the polarization can be depicted in terms of the charging of a condenser. The charging current of an ideal condenser is 90 degrees out of phase with the alternating potential. With the introduction of absorptive polarization, the charging current acquires a resistive component in phase with the voltage. In vector notation, the angle between the total current vector and the vector for the charging current is the loss angle, and the tangent of this angle is the loss tangent, here termed $\tan \phi$.

One of the objectives of this study was to determine the suitability of dielectric loss measurements as a macroscopic monitor of the moisture content in epoxy-glass composites and printed circuit boards (PCBs).

Early work in the area of moisture-induced dielectric loss studies on glass-epoxy laminates was performed by Berry [2]. This work prompted additional efforts at the IBM Endicott laboratory to extend that work to the determination of moisture content in a circuit board. The work discussed here is a direct outgrowth of those efforts.

Experimental procedures

• Coupon experiments

Small, three-ply laminate coupons were used to facilitate the investigation of $\tan \phi$. All coupons were of the same configuration, consisting of three plies of resin-glass composite laminated between sheets of one-ounce copper. This laminate was cut into squares 16 mm on a side. To aid

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in moisture sorption/desorption, nine equispaced one-mm holes were drilled on four-mm centers. Pairs of coaxial leads of equal length were attached to the copper on each side of the laminate. Next the coupons were subjected to various levels of relative humidity (RH) ranging from 0 to 80%. Measurements of $\tan \phi$ were made by connecting the four coaxial leads from each coupon to an impedance bridge capable of measuring dielectric loss and capacitance at frequencies over the range 100 Hz to 100 kHz. $\tan \phi$ was measured at 90°C, and a low-humidity environment was maintained for at least one hour prior to the measurement to ensure that surface moisture did not contribute to the dielectric loss of all samples—coupons and PCBs. Results from these tests are presented and discussed later.

The coupon materials were of three types: epoxy-heat-cleaned glass (no coupler) and epoxy-glass coated with two types of coupler.

• Board experiments

Dielectric loss measurements were also performed on printed circuit boards. Measurements were made between internal signal lines and the nearest voltage plane. Usually a total of thirty nets were measured. Special fixtures were used to connect the board and the measuring cable/meter assembly. Connections to internal nets were established through plated through-holes (PTH) by means of these fixtures. Measurements reported here were made at a temperature of 90°C and a frequency of 200 Hz. The boards were subjected to the same range of humidity environments as the coupons. Data are reported for boards made with epoxy-glass coated with couplers exhibiting various amounts of resistance to moisture degradation.

Results

The kinetics of change in dielectric loss caused by moisture absorption-desorption were investigated at several humidity conditions on samples having a prior history ranging from dry (as-received or baked) to saturated following a previous exposure. For this reason, few samples had a comparable exposure history; i.e., the implicit assumption was that "equilibrium" was independent of the direction from which it was approached and that hysteresis effects were minimal.

Figures 1 to 4 illustrate typical kinetic responses to moisture absorption-desorption for coupons and printed circuit boards.

It was assumed that dielectric loss was proportional to the moisture concentration, and hence should track the moisture concentration profile established by moisture diffusion into and out of test samples. Accordingly, the loss data were fitted to an equation of the general form

$$c_Y = c_1 + (c_S - c_1) \operatorname{erf} B(t - t_0) \text{ on charging,} \quad (1a)$$

$$c_Y = c_S - (c_S - c_1) \operatorname{erf} B(t - t_0) \text{ on discharging,} \quad (1b)$$

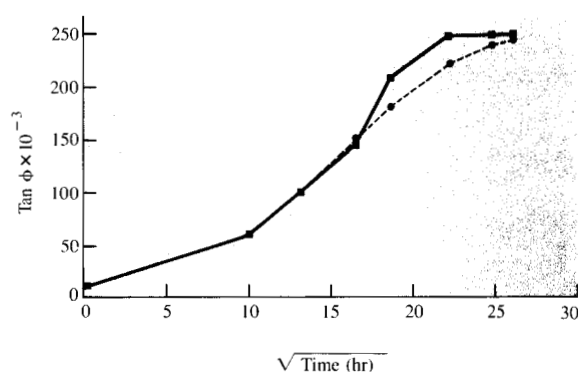


Figure 1

Illustrative change in dielectric loss of coupler A coupons upon exposure to moisture at 90°C. Measurements were made at an RH level of 80% at 200 Hz. The coupons were dried at 90°C to equilibrium, then exposed to an RH level of 35% at 90°C to saturation. Also shown is a calculated curve (dashed) obtained using Eq. (1) with the following parameter values: $c_S = 250$, $c_1 = 10$, $B = 0.0025 \text{ hr}^{-1}$, and $t_0 = 1 \text{ hr}$.

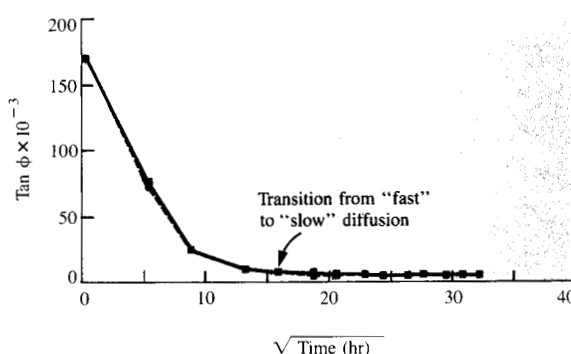


Figure 2

Illustrative change in dielectric loss of coupler A coupons upon desorption at 90°C following saturation at an RH level of 65% at 90°C. Here, the calculated curve (dashed) was obtained using (for the range preceding the transition from "fast" to "slow" diffusion) $c_S = 115$, $c_1 = 2$, $B = 0.022 \text{ hr}^{-1}$, $t_0 = -1 \text{ hr}$, and (for the remaining range) $c_S = 65$, $c_1 = 4.9$, $B = 0.00455 \text{ hr}^{-1}$, and $t_0 = -4.5 \text{ hr}$.

where erf is the error function and the following definitions apply:

- c_Y = the loss value after time t in seconds,
- c_S = the maximum loss value for a specified humidity,
- c_1 = the initial value of the dielectric loss at $t = 0$,
- t_0 = a time scale adjustment parameter,
- B = a parameter determined by sample dimensions and moisture diffusion kinetics for the material and test temperature; i.e., $[B(t - t_0)] = 4D(t - t_0)/x^2$, with x the transport distance (cm) and D the diffusion coefficient (cm^2/s).

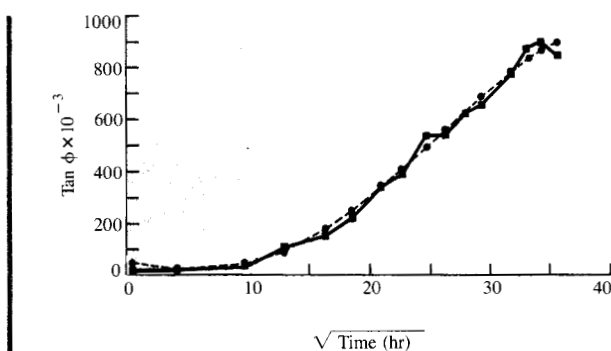


Figure 3

Illustrative change in dielectric loss of a PCB upon exposure to moisture at an RH level of 80% at 90°C. The calculated curve (dashed) was obtained using $c_s = 1000$, $c_l = 4$, $B = 0.0012 \text{ hr}^{-1}$, and $t_0 = 5 \text{ hr}$.

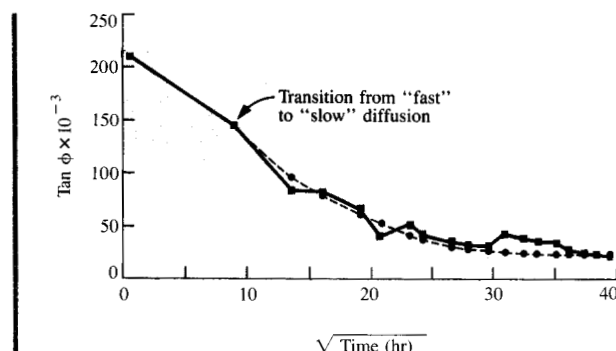


Figure 4

Illustrative drying curve of a PCB saturated at an RH level of 65% at 90°C and dried at an RH level of 35% at 90°C. The calculated curve (dashed) was obtained using (for the range preceding the transition) $c_s = 35$, $c_l = 12$, $B = 0.0073 \text{ hr}^{-1}$, $t_0 = -2.7 \text{ hr}$, and (for the remaining range) $c_s = 200$, $c_l = 23$, $B = 0.001 \text{ hr}^{-1}$, and $t_0 = -11 \text{ hr}$.

For coupons, the x -distance is taken to be a radial (or unidirectional) distance; z -direction transport is barred by the copper surface cladding. For PCBs, the size negates planar (edge) transport as a significant contributor; transport normal to the plane of the board through clearance holes etched in internal ground or voltage planes is the major contributor.

An iterative approach was used to determine the best set of parameters for a given sample. In the process of this curve fitting, it was observed that a satisfactory fit to the data could not always be obtained with a single set of parameters, particularly on discharging. In effect, a satisfactory fit required the use of two diffusion coefficients. This argument is developed in the next section.

Discussion of results

General observations

Some general observations can be made concerning the relationship between $\tan \phi$ values and moisture exposure before a more detailed analysis is discussed. From an examination of **Tables 1** and **2**, a prior history dependence on the dielectric loss at saturation is apparent for RH levels in excess of approximately 35%. The equilibrium value reached for a given condition depends on the conditions previously experienced by that sample. In summarizing the data, we have chosen to average those values for a given condition which were arrived at from the same direction. This does not strictly limit the data because of history considerations but it does take into account the history immediately preceding the condition under consideration.

The saturation or equilibrium value of $\tan \phi$ increases with exposure to increasing levels of RH at 90°C. This is illustrated in **Figure 5** for epoxy-glass coupons in which the adhesion-promoting coupler either has been removed (heat-cleaned) or is one of two types, designated here as A and B. As can be seen, the use of coupler B leads to more resistance at higher humidities to polarization effects resulting from added moisture than does the use of coupler A. The behavior of coupler A samples is similar to that of the heat-cleaned samples at higher humidities. If the $\tan \phi$ measurement is sensitive to the glass/epoxy interface, it appears that coupler A becomes increasingly ineffective at an RH level above 50%. Such behavior suggests moisture interaction with the coupler-epoxy bond or the glass-coupler bond. This interaction apparently does not result in permanent interfacial damage, but it is probably the cause of the hysteresis observed on desorption to a saturation value corresponding to a reduced humidity, i.e., the source of the history dependence.

The moisture-induced loss response in printed circuit boards is illustrated in **Figure 6**, and for comparative purposes in **Figure 7**, where coupon and board data are compared. The dramatic increase in loss at RH levels greater than 65% for boards constructed from the epoxy/A-composite and exposed to the process chemistry is apparent. This large increase for the same interfacial area is attributed to residual ionic contamination. Notice that the loss values for the board containing coupler B track those of the coupler A board, although at a reduced level. For example, the ratio of loss for coupon samples (coupler A:B) at an RH level of 80% is about that observed for boards, implying that both board types contain approximately the same amounts of ionic contamination.

Dielectric loss: Electrical analog

In order to lay the foundation for an interpretation of the dielectric behavior of epoxy and epoxy-glass composites, it is useful to recall that, aside from geometrical factors, the

Table 1 Tan ϕ charging data summary.

Relative humidity (%)	Identification	c_s	c_i	B (hr ⁻¹)	t_0 (hr)	Mean value of c_s
35	A	10	2.5	0.002	5.5	10
	H-C	12	2.5	0.0017	3.5	12
	B	12	3.5	0.0025	5.5	12
	PCB-A	16	5	0.0005	5	16
50	A	48	4	0.002	3.5	
	A	65	10	0.002	-5	
	A	45	5	0.00275	2	
	A	18	3	0.002	3	44
	PCB-A	50	16	0.001	-2	
	PCB-A	40	4.5	0.0005	-2.5	45
	H-C	40	5	0.005	-2	
	H-C	150	12	0.0045	-6	
	H-C	72	4	0.002	4	87.3
	B	32	4	0.0006	4.5	32
	A	230	1	0.0015	2	
	A	215	2	0.002	2.5	
65	A	225	2.5	0.0015	5	223.3
	PCB-A	300	40	0.0005	1.5	
	PCB-A	210	30	0.00075	-3	255
	H-C	230	1	0.0035	2	
	H-C	225	1	0.0022	3.5	227.5
	B	50	4	0.002	-2	50
	A	280	210	0.0025	7.5	
	A	250	10	0.0025	-1	
80	A	300	10	0.00175	6.5	276.7
	PCB-A	1000	4	0.0012	-5	
	PCB-A	1500	30	0.0015	-12	1250
	H-C	295	210	0.005	2.5	295
	B	160	45	0.0018	-5	160
	PCB-B	500	5	0.0005	-8.5	
	PCB-B	700	10	0.0005	-8	600

Table 2 Tan ϕ parameters for discharging.

Relative humidity (%)	Identification	c_{s1}	c_{i1}	B_1 (hr ⁻¹)	t_{01} (hr)	c_{s2}	c_{i2}	B_2 (hr ⁻¹)	t_{02} (hr)
0	A	60	2	0.030	-2.2	35	5.6	0.0033	4.6
	A	175	2	0.014	1.3	45	4.2	0.0044	4
	A	240	2	0.0085	1.9	50	5	0.0035	7.5
	PCB-A	8.9	4.45	0.0025	-4	—	—	—	—
	PCB-A	7.0	0.2	0.007	1	6.8	4.3	0.0006	-7.7
	PCB-A	9.6	4.5	0.0037	1.3	—	—	—	—
	H-C	270	5	0.016	0.15	95	5.5	0.0037	6
	H-C	160	2	0.018	-1.2	60	4.5	0.0042	4.8
	H-C	235	1.5	0.023	-2.5	50	5.5	0.0033	3.5
	B	19	1	0.009	2	12	4.5	0.001	4
	A	28	1.5	0.009	2.5	18	14	0.0045	1.5
35	PCB-A	148	55	0.00195	1.3	—	—	—	—
	PCB-A	35	12	0.0073	2.7	200	23	0.001	1
	H-C	108	5	0.0095	2	20	15	0.0045	1.5
	PCB-A	75	20	0.0037	-0.4	825	150	0.0007	12

Note: The subscripts 1 and 2 refer, respectively, to values of the indicated parameters in the "fast" and "slow" diffusion ranges.

capacitance of a sample is proportional to the ratio of the permittivity to the dielectric constant. Both of these material constants are traceable to the polarizability (number of

dipoles per unit volume) of the dielectric. Thus, the permeation of moisture can result only in a change in capacitance if the dielectric constant or the permittivity is

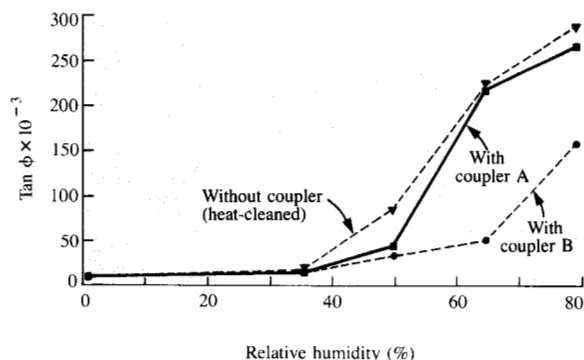


Figure 5

Saturated values of dielectric loss for coupons at various RH levels. Samples without coupler (heat-cleaned) become increasingly identical to those with coupler A at RH > 50%. Behavior for samples with coupler B indicates less interfacial interaction with moisture.

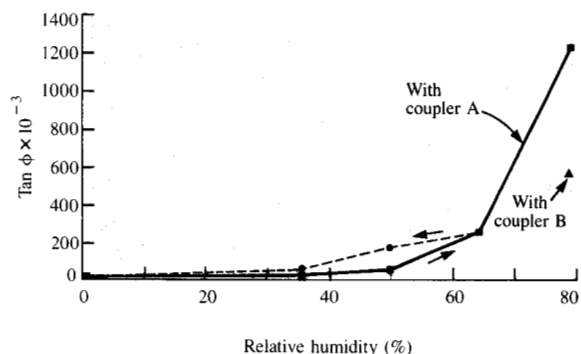


Figure 6

Saturated values of dielectric loss for PCBs with coupler A. Note hysteresis between charging and discharging values. Only one data point exists for PCBs with coupler B, and that value is considerably less than the corresponding value for PCBs with coupler A.

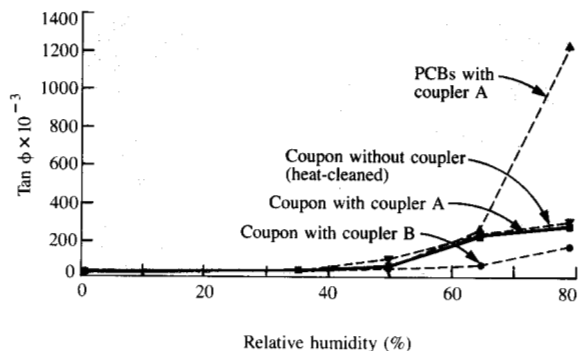


Figure 7

Comparison of dielectric loss for coupons and PCBs. Response at RH > 65% may be ionic (bulk plus interfacial)—from chemical processing.

altered, i.e.,

$$C = Q/V = (\epsilon/\mu)A/\ell; \quad \epsilon = \epsilon_0 + \chi,$$

the dipole density is changed [3]. For a parallel RC network analog model for the dielectric loss, the resistive impedance is given by

$$Z_R = R/\ell + (\omega RC)^2,$$

while the capacitive impedance is given by

$$Z_C = -\omega R^2 C/\ell + (\omega RC)^2,$$

with $\tan \phi = Z_R/Z_C = -1/\omega RC$ and $\omega = 2\pi f$.

Since C increases with moisture absorption (Figure 8) for a typical example, an increase in $\tan \phi$ must be attributed to a decrease in the ohmic resistance of the epoxy (including the composite). This behavior is also exhibited in the resin itself, although to a much lesser degree; i.e., the effect is not exclusively an interfacial effect. Also, as shown in Figs. 5 and 6, extensive departure from a linear dependence on RH is not evident until RH levels in excess of 35% are experienced. The mechanism (process) causing this decrease in resistance is presumably free ion transport or a charge transfer process between chain sites affected by interaction with water. At this point it can be speculated that interchain bonding is altered by moisture. These altered sites establish a conductive network through the polymeric structure. It is not clear how the volume ahead of the diffusion front contributes to this conduction process. However, if the "dry" value of $\tan \phi$ is assumed to be 0.003, and the capacitance is assumed to be 20 pF at 200 Hz, the calculated value of bulk resistance (insulation resistance) is found to be approximately 10 000 megohms, a reasonable value at 90°C. When the dielectric loss increases to values of approximately 0.3, the bulk resistance has decreased to approximately 100 megohms. This would represent the combined series resistance of a region with moisture coupled to a region without moisture; i.e.,

$$R = (1 - f)R(\text{without water}) + fR(\text{with water}), \quad (2)$$

where f is the fraction of the sample diffusion path that the diffusion profile has penetrated. When this calculation is performed, the f -value required for a given change in loss is inconsistent with the moisture penetration depth based on values of bulk diffusion. A short-circuiting path is required, a high-moisture-content region which quickly distributes moisture for diffusion perpendicular to the path. Such a path may well be the interchain network in the case of the resin itself, supplemented by the resin/glass interfacial path for resin-glass composites. In any case it seems clear that the loss-time behavior requires a transport process involving a "slow" process to reach absorption equilibrium, accompanied by a "fast" process for the observed changes in the conduction process (dc-resistance). Such speculation is supported by some limited data. Figure 9 illustrates the

variation of loss with time at an RH level of 65% at 90°C for samples composed of a number of laminate plies ranging from 2 to 10. As can be noted, the response is immediate, with approximately the same slope and essentially the same time to saturation. The limited data seem to suggest an ordering according to thickness at early exposures and an inverse ordering with respect to thickness at saturation. Clearly, additional experimentation would be required to fully clarify the transport processes involved and the structural changes induced by the absorption of moisture.

Dielectric loss and moisture solubility and diffusion

• Moisture solubility

Dielectric loss values in composites are presumed to arise from contributions from the bulk resin and the resin/glass interface. A hypothesis for the bulk and interfacial contributions has already been proposed. Basically, dielectric polarization arises from reorientation of a polar group in response to an applied field. The incorporation of moisture into the chain structure results in a moisture-induced dipole contribution measurable at approximately -40°C at 200 Hz, as well as a change in polymer chain segment length free to vibrate in response to the field frequency. The latter component of dielectric loss may well be the prime effect of moisture absorption at temperatures above 25°C. It is speculated that entry of moisture into the interstices of the epoxy chain structure results in local expansion, formation of hydrogen-type bonds, and loss of interchain bonds. This process of plasticization provides two opportunities for polarization relaxation: relaxation of a moisture-chain complex and relaxation from an increase in chain segment mobility. Thus, as moisture penetrates the host structure, the volume of resin responding to the applied electric field increases, with a proportionate increase in $\tan \phi$. It is argued that this is the reason the kinetics of loss parallels the kinetics of moisture diffusion and provides a means to estimate moisture diffusivity.

From a structural viewpoint, the process is viewed as adsorption followed by transport into the resin structure along paths defined by the polymer chain network. Since moisture solubility in epoxy is linearly dependent on partial pressure of water at constant temperature [4], one would expect the loss values also to be linearly dependent. From Fig. 6 this is clearly not the case, at least for humidities greater than some yet-to-be-determined threshold value. This nonlinear behavior would seem to require two components: a bulk contribution and an interfacial component. It is postulated that initially moisture is absorbed into the resin at sites favoring "reversible" transport. At a transition moisture content, the partitioning of moisture between bulk and interfacial occupation is shifted to favor the latter. At this point the dominant loss mechanism is shifted to a Maxwell-

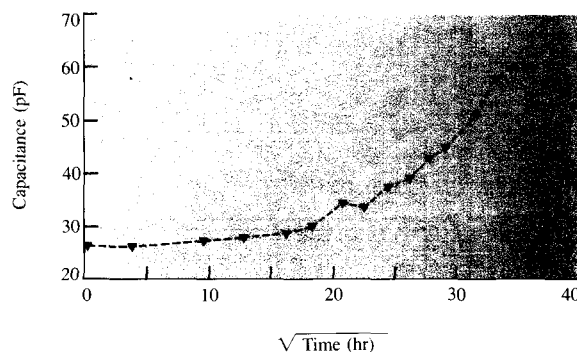


Figure 8

Illustrative change in capacitance of a PCB with moisture exposure at an RH level of 80% at 90°C.

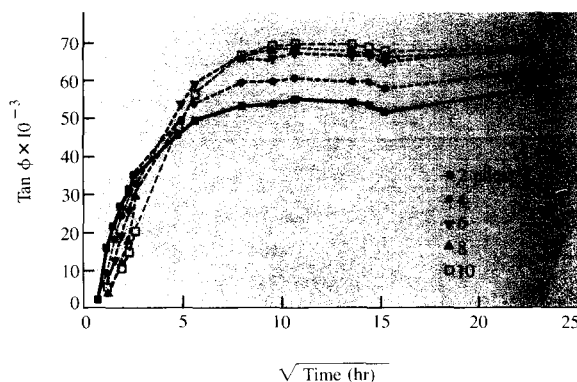


Figure 9

Illustrative loss behavior for coupons composed of from 2 to 10 plies. As can be seen, sample thickness does not play a part in initial kinetic response.

Wagner type of polarization, primarily the result of moisture-induced dipole mobility. The nature of this dipole mobility is not known.

• Diffusion

From Fick's second law, the moisture concentration profile at any (x, t) in a semi-infinite medium for which the initial concentration is γ_i throughout and the surface is maintained at γ_s is given by [5]

$$\gamma = \gamma_s + (\gamma_i - \gamma_s) \operatorname{erf} [(x/2(Dt))^{0.5}], \quad (3)$$

whereas the total quantity (M) of moisture absorbed in a sample of thickness s is [6]

$$M = M_i + (M_s - M_i) \{1 - \exp [-7.3(Dt/s^2)^{0.75}]\}. \quad (4)$$

Since the dielectric loss-time analysis, i.e., Eq. (1), correlates

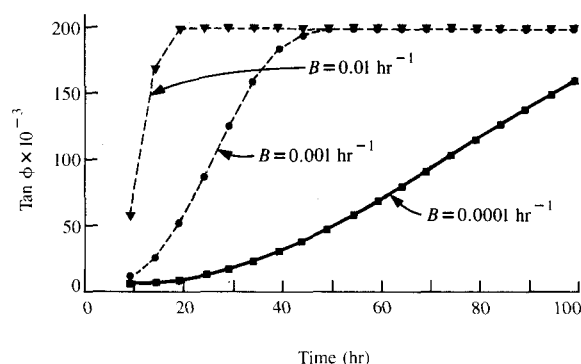


Figure 10

Effect of the parameter B on response kinetics characterized by Eq. (1) for a constant value of t_0 . Here, t_0 is assumed to be 5 hr and c_s and c_i are assumed to be, respectively, 200 and 5.

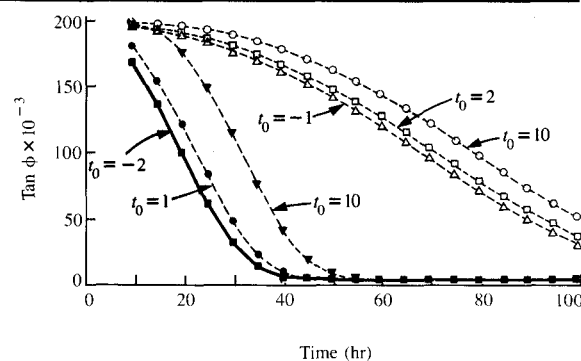


Figure 11

Effect of the parameter t_0 (in hr) on response kinetics characterized by Eq. (1) for constant parameter B values of 0.001 (solid) and 0.0001 (empty) hr^{-1} . The parameters c_s and c_i are assumed to be, respectively, 200 and 5.

with Eq. (3) rather than Eq. (4), it seems implied that the moisture-induced loss is determined by concentration-dependent equilibria and controlled by the diffusion process. This presents a dilemma. How is the observation of measurable change in loss for short exposure times to be reconciled with small diffusion zones (depths of penetration) forecast by Fickian diffusion with "bulk" values of the diffusion coefficient? To put this another way: The parameter B in Eq. (1) contains a transport distance term (x) whose nature is puzzling. If the value of x is set at the sample thickness, then, as mentioned, the response is inconsistent with Fickian diffusion. Alternatively, x can be interpreted to be the leading edge of the advancing front representing epoxy saturated with moisture in the case of resin only, or saturation of the resin/glass interface for the case of the composite. Hence, the value of B represents a mean estimate of transport kinetics for the absorption process to saturation. Similarly, the physical significance of the parameter t_0 in Eq. (1) remains unresolved, although the influence of B and t_0 on the resultant kinetics is shown in Figures 10 and 11. In these figures the effect of B at constant t_0 and the effect of t_0 at constant B on the kinetics of loss change are illustrated.

A possible interpretation of t_0 can be extracted from Eq. (3) once the credibility for Eq. (3) has been established. From independent data the temperature dependence of the diffusion coefficient for moisture is given by [4]:

$$D = D_0 \exp(-Q/RT),$$

where

$$\begin{aligned} D_0 &= 0.023 \text{ cm}^2/\text{s}, \\ Q &= 9500 \text{ cal/mol}, \\ R &= \text{gas constant} = 1.98, \\ T &= \text{temperature, K}. \end{aligned}$$

Thus the value for D at 90°C is $4.18 \times 10^{-8} \text{ cm}^2/\text{s}$ in the z -

Table 3 Diffusion coefficients for charging and discharging.

Charging						
Identification	$B \text{ (hr}^{-1}\text{)}$			$D \text{ (cm}^2\text{/s)}$		
	min.	mean	max.	min.	mean	max.
A	1.63×10^{-3}	2.05×10^{-3}	2.47×10^{-3}	0.34×10^{-8}	0.43×10^{-8}	0.52×10^{-8}
H-C	1.95×10^{-3}	3.4×10^{-3}	4.85×10^{-3}	0.417×10^{-8}	0.725×10^{-8}	1.04×10^{-8}
B	0.9×10^{-3}	1.7×10^{-3}	2.5×10^{-3}	1.96×10^{-9}	0.36×10^{-8}	0.535×10^{-8}
Discharging						
Identification	"Slow" $D \text{ (cm}^2\text{/s)}$			"Fast" $D \text{ (cm}^2\text{/s)}$		
	min.	mean	max.	min.	mean	max.
A	7×10^{-9}	8.29×10^{-9}	9.57×10^{-9}	1.13×10^{-8}	3.25×10^{-8}	5.38×10^{-8}
H-C	4.89×10^{-9}	8.29×10^{-9}	1.17×10^{-8}	2.38×10^{-8}	3.55×10^{-8}	4.72×10^{-8}
B		2.12×10^{-9}			1.9×10^{-8}	

direction (perpendicular to the plane of the composite).

Table 3 summarizes the results obtained when the value for D is extracted from the parameter B using sample (PCB or coupon) thickness or width as appropriate for x . The agreement with the expected value is reasonable and encourages consideration of the parameter t_0 .

That parameter appears to be an adjustable time scale shift which allows enough time for water molecules to permeate to a depth sufficient to cause a change in the bulk and/or interfacial loss. Those cases exhibiting an "immediate" response, i.e., values of t_0 which are small or negative, tend to correlate with samples with a prior exposure history to levels of RH in excess of 35%. This shift in the time scale to earlier times may be the result of prior moisture interaction with the epoxy resin or the resin/glass interface.

• Dielectric loss/moisture (humidity) calibration

Figures 6 and 7 attest to the fact that the loss response to moisture at saturation is not linear with partial pressure, at least beyond some threshold value. Can an argument be constructed which supports a threshold value and is plausible?

The loss responses of epoxy-glass samples to moisture with couplers A, B or without coupler (heat-cleaned) are essentially linear and indistinguishable from one another at low absorption levels of moisture. Therefore, the initial slope may indeed be linear with partial pressure. Data regression yields the following equation to describe loss as a function of partial pressure:

$$y, \text{ units of } 0.001 \text{ (linear)} = 5.5 + 18.57 RH \text{ (decimal)}.$$

Deviation from linearity starts at some threshold RH level and contributes exponentially. The equation describing such behavior is

$$y, \text{ units of } 0.001 \text{ (exp)}$$

$$= \gamma_s - (\gamma_s - \gamma_0) \exp \left[- \left(\frac{RH - RH_c}{1 - RH} \right)^2 \right].$$

Figure 12 illustrates the resulting fit to the data for samples representing epoxy-glass with couplers A and B and without coupler. Each material required a different critical level RH_c of RH and those values order as would be expected if the exponential contribution arose from the resin/glass interface. The RH_c levels were 25, 35, and 48% for heat-cleaned and A- and B-coupled epoxy-glass composites, respectively. If additional experiments support this proposed model, dielectric loss as a measurement technique becomes, in addition to a moisture monitor, a method to screen the moisture resistance of candidate couplers.

Conclusions

The measurement of dielectric loss has been determined to be a reproducible predictor of moisture content in epoxy-glass composites and printed circuit boards at equilibrium

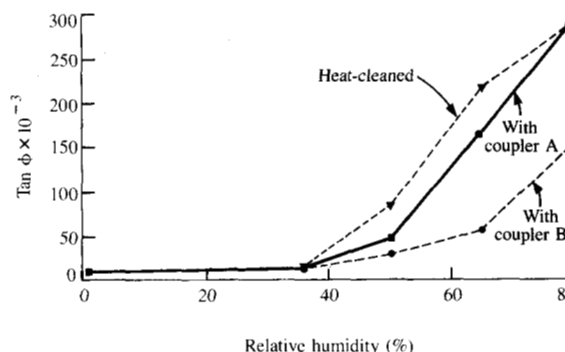


Figure 12

Fitted curves for saturated values of $\tan \delta$ for coupons at 90°C. Regression analysis indicates start of nonlinear behavior at 25, 35, and 48%, respectively, for heat-cleaned samples and those with couplers A and B.

with the external environment. The kinetics of the approach to this equilibrium were found to be consistent with the kinetics of moisture diffusion determined by more traditional methods. The change in dielectric loss with moisture absorption/desorption could be attributed to a change in dc resistance because the capacitance was found to be essentially independent of moisture content and frequency.

The elevated-temperature, low-frequency loss behavior which was observed has been interpreted in terms of interfacial or Maxwell-Wagner polarization applied to the resin and the resin/glass interface. Moisture-induced effects at interfaces suggest that the loss behavior can provide a means to screen the resistance of potential coupler candidates to reversible moisture degradation.

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