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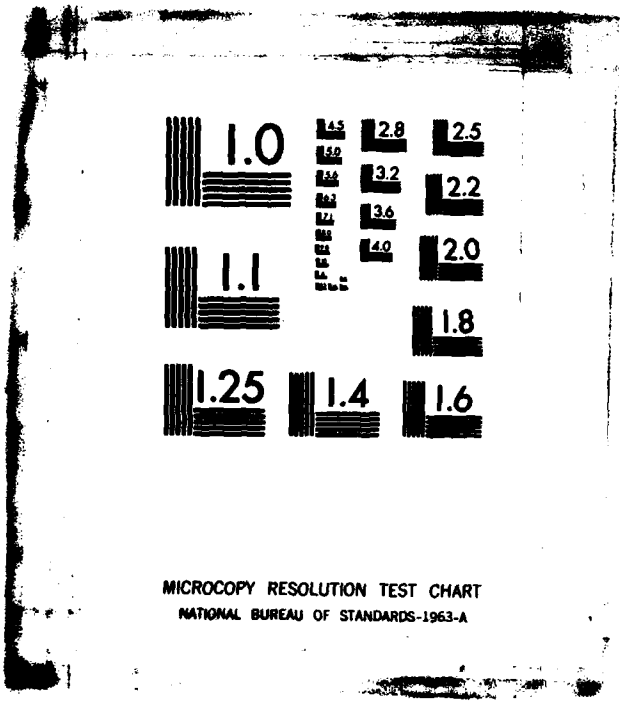

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A DIELECTRIC STUDY OF THE TIME-TEMPERATURE-TRANSFORMATION (TTT) DIAGRAM OF DGEBA EPOXY RESINS CURED WITH DDS

by

Norman F. Sheppard, Jr., Michael C. W. Coln
and Stephen D. Senturia

Article prepared for presentation at

The 1984 SAMPE Meeting, Reno, Nev April 1984

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
Department of Electrical Engineering and Computer Science
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December 14, 1983

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Microdielectrometry and differential scanning calorimetry have been used to study the isothermal cure of EPON 825 with diaminodiphenylsulfone (DDS). The results are compared with published torsional braid analysis (TBA) studies of the same system using a time-temperature-transformation diagram. The dielectric measurements were made at temperatures between 100 and 220 C using measurement frequencies between 0.1 Hz and 10,000 Hz. The DSC was operated in

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A DIELECTRIC STUDY OF THE TIME-TEMPERATURE-TRANSFORMATION (TTT) DIAGRAM OF DGEBA EPOXY RESINS CURED WITH DDS

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Abstract

Microdielectrometry and differential scanning calorimetry have been used to study the isothermal cure of EPON 825 with diaminodiphenylsulfone (DDS). The results are compared with published torsional braid analysis (TBA) studies of the same system using a time-temperature-transformation diagram. The dielectric measurements were made at temperatures between 100°C and 220°C using measurement frequencies between 0.1 Hz and 10,000 Hz. The DSC was operated in an isothermal mode to determine extent of conversion at temperatures between 137°C and 177°C. Two mechanisms of charge transport are responsible for the observed dielectric response; an ionic conductivity early in cure that decreases as the reaction proceeds, and a Debye type dipole relaxation later in cure. The time to reach 60% conversion determined by DSC correlates with the TBA gelation loss peak, as would be expected from the Flory theory of gelation, but there is no clear dielectric "event" at 60% conversion. The peak in dielectric loss factor identified with the dipole relaxation correlates with the TBA vitrification loss peak.

Keywords: Microdielectrometry, dielectric cure monitoring, time-temperature-transformation (TTT) diagram, DGEBA/DDS

1. INTRODUCTION

Dielectric methods for monitoring the cure of epoxy resins have been established for nearly 25 years, dating back to the pioneering work of Delmonte [1] and Warfield and Petree [2]. Dramatic changes in the dielectric properties of the material accompany the transformation of the resin from a viscous liquid to a brittle solid. The simplicity of dielectric measurements have led to wide use both in materials analysis and process control [3,4,5]. A recent development in the field has been the technique called Microdielectrometry, which utilizes a miniature integrated circuit sensor to perform the dielectric measurement [6,7]. The small size of the probe, the on-chip temperature sensor, and the wide measurement frequency range make Microdielectrometry a promising alternative to conventional parallel-plate dielectric measurements for process control applications.

This paper addresses the interpretation of dielectric data obtained during the cure of epoxy resins in terms of the physical changes taking place during cure. The nature of the changes can be described with the aid of the time-temperature-transformation (or TTT) diagram for thermosets, developed by Gillham [8]. The TTT diagram describes the state of a resin undergoing isothermal cure as a function of cure time and the isothermal cure temperature. The generalized TTT diagram in Figure 1 shows four identifiable states of the curing material: liquid, gelled rubber, ungelled glass, and gelled glass. At cure temperatures between the glass transition temperature at gelation, T_{gel}, and the ultimate glass transition temperature of the fully cured material, $T_{g\infty}$, curing proceeds from the liquid state to a gelled rubber, and finally to a gelled glass. Gelation is the transformation of the liquid to a rubbery gel. Further curing in the rubbery gel state increases the crosslink density of the network until the glass transition temperature of the gelled rubber reaches the curing temperature, at which point vitrification, i.e., the formation of a gelled glass, occurs.

The objective of this work is to examine dielectric measurements in the context of the TTT diagram with the goal of obtaining a better understanding of the physical mechanisms underlying the dielectric response. The system chosen for study was a low molecular weight DGEBA resin, Shell EPON 825, cured with diaminodiphenylsulfone (DDS). The TTT diagram for this system has been published by Enns and Gillham [9] based on torsional braid analysis (TBA). The isothermal cure was studied using Microdielectrometry over the temperature range 100°C to 220°C, and the kinetics of the chemical reaction were studied using differential scanning calorimetry (DSC) over the range 137°C to 177°C.

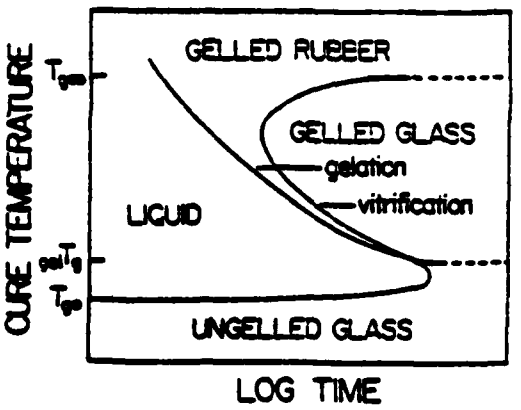

Fig. 1. Generalized Time-Temperature-Transformation diagram showing states of thermosetting resins as a function of cure time at isothermal cure temperatures. After Gillham [8].

2. EXPERIMENTAL

2.1 Materials

The EPON 825 resin was obtained from New England Resins and Pigments, the diaminodiphenylsulfone curing agent from Dr. Gary Hagnauer at the Army Materials and Mechanics Research Center in Watertown, MA. The resin mixture was prepared in a 5 gram batch by melting together the resin and the curing agent until the curing agent was dissolved. The mixture was kept refrigerated until use. No evidence of reprecipitation of the DDS was observed.

2.2 Microdielectrometry

Two types of Microdielectrometry sensors were used in this work. Most of the results were obtained using the sensor described in Reference 7, which incorporates an on-chip semiconductor diode thermometer for combined dielectric/temperature measurements. Some measurements were made with a modified sensor in a flat Kapton® "ribbon" package. A photomicrograph of the modified sensor mounted in its package is shown in Figure 2. The packaged sensor is 0.5 mm thick, 5 mm wide and 50 cm

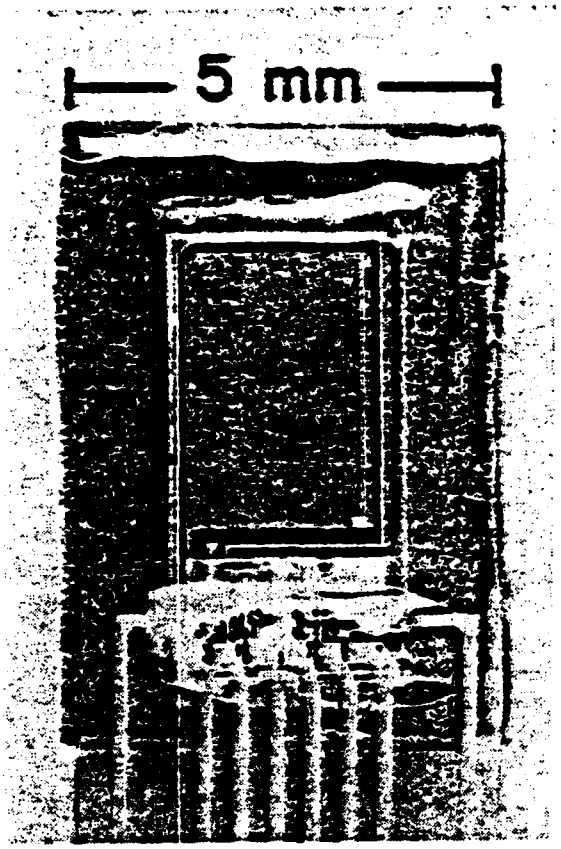

Fig. 2. Photomicrograph of microdielectrometer chip showing closeup of sensor chip in flexible Kapton® "ribbon" package.

long. This new sensor/package combination has been used for neat resin studies and for cure monitoring in laminates. The sensors were prepared by heating them on a hot plate to approximately 50°C and placing a small sample of the resin mixture (typically 20 mg) on the sensor electrodes. Rapid melting and flow produced good contact between the resin mixture and the electrodes.

The instrumentation used for the Microdielectrometry measurements has been significantly improved over that described previously [6,7]. The Hewlett-Packard 3575A Gain/Phase Meter used for analyzing the sensor response has been replaced by a Fourier Transform Digital Correlator of our own design. With the correlator, the accessible frequency range of the measurement is extended to 0.005 - 10,000 Hz and the sensitivity is significantly improved compared to that achievable with the HP3575A. Loss factors as low as 0.01 can now be measured, corresponding in typical cured resins to tan delta values on the order of 0.003.

In the isothermal cure experiments reported here, the sensor and socket assembly were placed into an oven preheated to the cure temperature. The sample temperature, as measured by the on-chip temperature sensor, came to equilibrium in approximately 3 minutes. The dielectric permittivity, loss factor, and temperature were then monitored for the duration of the cure. An inert atmosphere was maintained by flowing dry nitrogen through the socket assembly.

2.3 Differential Scanning Calorimetry

A Perkin-Elmer DSC-II differential scanning calorimeter (DSC) was operated in an isothermal mode to measure extent of conversion versus cure time at cure temperatures in the range 137°C to 177°C. The heat flow output signal was measured by a digital voltmeter interfaced to an HP85 calculator, which stored the data on cassette for later processing. The temperature scale was calibrated using the melting points of indium and lead, and the enthalpy was calibrated using an indium standard. Hermetically sealed aluminum sample pans were used. The sample sizes were in the range of 5 to 15 mg. The sample was placed in the cell at 77°C and then heated to the desired temperature at 160°C/min. The reaction exotherm was followed to apparent completion. The area under the exotherm was taken to be the heat of conversion at that temperature.

The curing of an epoxy resin at temperatures below the ultimate T_g does not insure complete reaction. To obtain extent of conversion from an isothermal cure below the ultimate T_g, the total heat of reaction of the fully cured resin must be known. This is obtained by dynamically scanning the temperature at a slow rate to insure complete conversion. The total heat

of reaction was obtained from a ramped cure at a rate of 2.5°C/min from 80°C to 300°C. This experiment yielded a total heat of reaction of 99.5 cal/g, which is 99.2 kJ/mol (23.7 kcal/mol), consistent with results reported in the literature for similar epoxy systems [11]. Degree of conversion as a function of time at each isothermal cure temperature was obtained from partial integration of the reaction exotherm, normalized by the total heat of reaction.

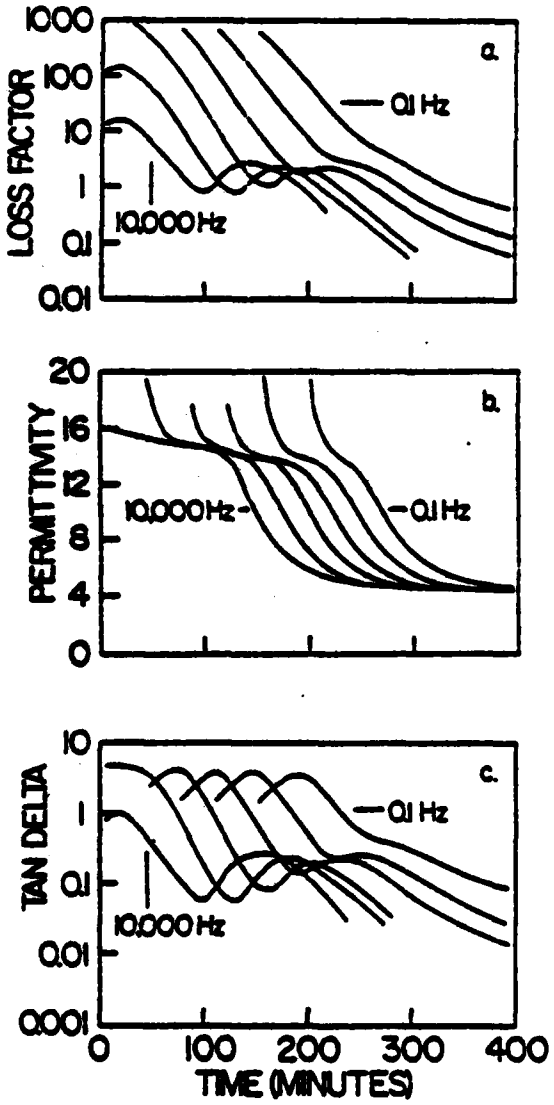

Fig. 3. Microdielectrometry results of cure of EPON 825/DDS at 100°C. Measurement frequencies 0.1, 1, 10, 100, 1000, 10000 Hz.
a) loss factor, ε" b) permittivity, ε' c) tan delta ε"/ε'

3. RESULTS

3.1 Microdielectrometry

Figures 3a, 3b and 3c show the dielectric loss factor, ε", the dielectric permittivity, ε', and the loss tangent (or tan delta), which is the ratio ε"/ε', for the cure of EPON 825/DDS at 100°C measured at frequencies of 0.1, 1, 10, 100, 1000 and 10,000 Hz.

The frequency dependence of the loss factor versus time indicates the superposition of two components, an ionic conductivity and a dipole relaxation [7]. Early in cure, ε" is inversely proportional to frequency, indicating a conductivity. The conductivity decreases approximately exponentially with cure time, due to the increasing viscosity of the resin as cure proceeds. This decrease is followed by a peak in ε" with an amplitude of about 2, which occurs earliest at the highest frequency. This loss peak is due to the restriction of molecular dipoles in the crosslinking resin.

The two processes of ionic conduction and dipole relaxation are also evident in the permittivity versus time plot of figure 3b. Early in cure, when the loss factor is large (>10), the apparent permittivity is extraordinarily large (>20) due to charge accumulation at the blocking sensor electrodes. As the loss factor drops, the blocking electrodes are no longer important and the apparent permittivity levels off at about 14, a value consistent with the polarizability of the resin. As curing proceeds, the permittivity then drops to a final value of approximately 4. Associated with this drop in ε' is the peak in ε", clearly evident in Figure 3a.

The data of Figures 3a and 3b can be combined to yield tan delta versus time, as in Figure 3c. The characteristic features of the tan delta versus time are a first peak early in the cure, related to the

ionic conductivity and the blocking electrodes, and a second peak later in cure due to dipole relaxation.

The use of a wide range of measurement frequencies helps illustrate the extent to which the orientational mobility of polar groups in the resin is being restricted as cure proceeds. At each successive ε" peak, the reciprocal of the frequency for that peak gives a measure of the characteristic time required for a dipole to overcome the viscous drag of the surrounding molecules and orient with the electric field. From the time at which the peak is first observed at 10kHz to the time the peak is observed at 0.1 Hz, this characteristic time increases by five orders of magnitude, from hundreds of microseconds to tens of seconds.

Curing experiments similar to those illustrated in Figure 3 were performed over a wide range of cure temperatures. Figures 4a, 4b and 4c show the cure temperature dependences of the loss factor, permittivity and tan delta versus cure time at a measurement frequency of 10 Hz. The characteristic features resulting from the ionic conductivity and the dipole relaxation mechanisms discussed above are apparent at all cure temperatures. As expected, the ionic conductivity decreases more rapidly with time and the dipole relaxation occurs sooner at higher cure temperatures, due to the thermal activation of the curing reaction.

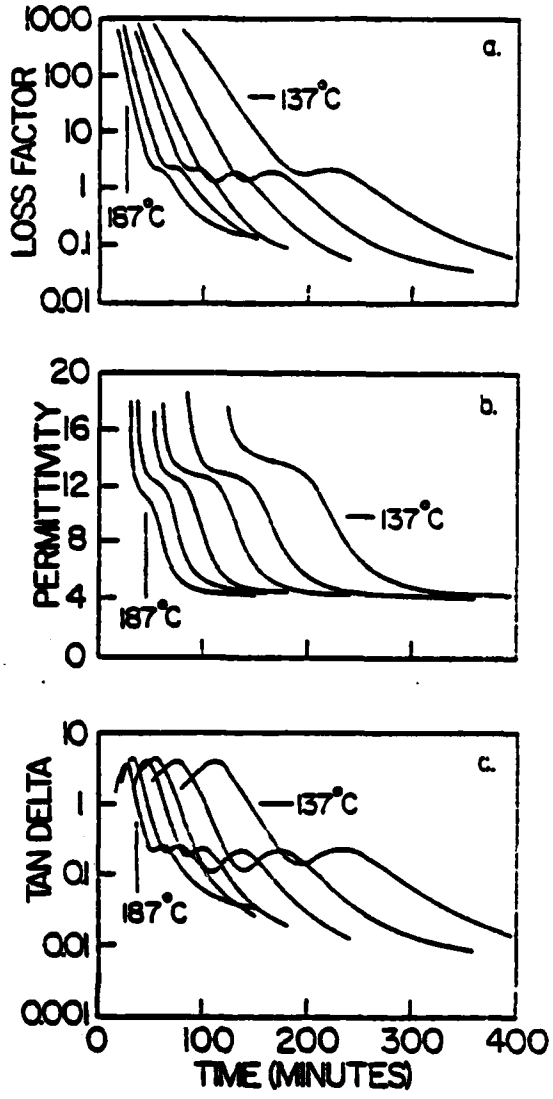

Fig. 4. Microdielectrometry results of cure of EPON 825/DDS at 10 Hz. Cure temperatures of 137, 147, 157, 167, 177, 187°C.
a) loss factor, ε" b) permittivity, ε' c) tan delta ε"/ε'

3.2 Differential Scanning Calorimetry

The extent of conversion versus time results obtained from DSC are shown in Figure 5 for cure temperatures ranging from 137°C to 177°C. There are three things to note about these curves: (1) As expected, the rate of reaction increases with increasing cure temperature, due to the thermal activation of the curing reaction. (2) There is an inflection in the extent of conversion versus time curves reflecting the autocatalytic

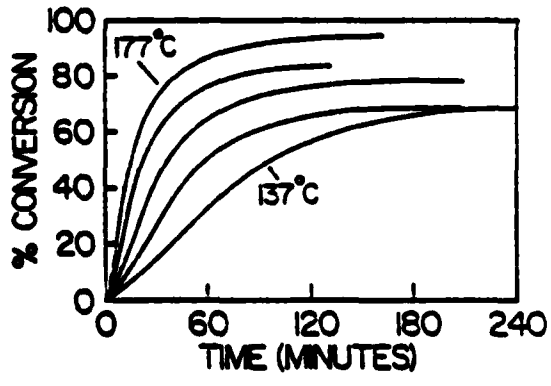

Fig. 5. Extent of conversion based on heat of reaction versus cure time determined using isothermal DSC measurement. Cure temperatures 137 - 177°C.

nature of the epoxy-amine curing reaction [11]. (3) The ultimate extent of conversion reached decreases with decreasing cure temperature. These results are related to the TTT diagram and to the dielectric data in the following section.

4. DISCUSSION

The objective of this study was to relate dielectric measurements to the time-temperature-transformation (TTT) diagram, so as to obtain a better understanding of the physical processes underlying the dielectric measurement. The TTT diagram for the EPON 825/DDS system determined using torsional braid analysis (TBA) was reported by Enns and Gillham [9]. TBA measures the free oscillatory decay of a resin-impregnated glass-fiber braid in a torsion pendulum. Maxima in the damping (logarithmic decrement) of the braid are interpreted to indicate the liquid to rubber transition associated with gelation and the rubber to glass transition associated with vitrification of the resin. Because this is a dynamic measurement, the time of occurence of the loss peak is frequency dependent. The TTT diagram indicates the time to reach the gelation "event" and the time to reach the vitrification "event" as a function of the isothermal cure temperature.

The DSC results provide a way of verifying that a valid comparison can be made between the dielectric measurements made in our lab to the TBA measurements of Enns and Gillham, which were done on nominally the same material. According to the Flory theory of gelation [12], the gel point of a stoichiometric system of difunctional molecules reacting with tetrafunctional crosslinkers will gel at an extent of reaction of 57.7%. If one assumes that the heat liberated by the epoxy curing is due only to the reaction of epoxides with amines, then the time to reach a DSC conversion of 57.7% should coincide with the time to reach gelation. Figure 6 shows a TTT diagram of the time to reach fractional conversions of 20%, 40%, 60% and 80%, taken from Figure 5, superimposed on the gelation and vitrification curves determined by Enns and Gillham [9]. The time to reach 60% conversion coincides with the time to reach gelation. The agreement is exceptionally good considering the assumptions made, and confirms that the systems are nominally identical and valid comparisons can be made to the TBA measurements.

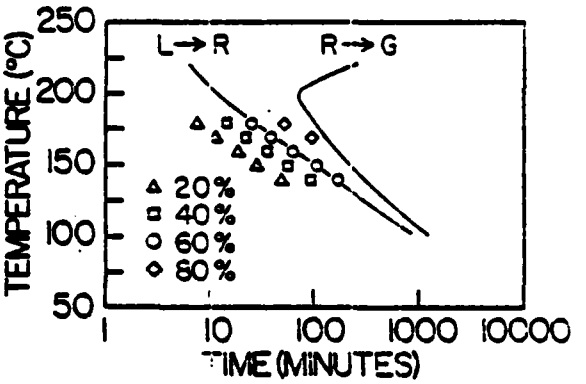

Fig. 6. Time-Temperature-Transformation diagram for EPON 825/DDS system showing time to reach conversions of 20%, 40%, 60% and 80%. Liquid to Rubber (gelation) and Rubber to Glass (vitrification) transformations from Torsional Braid Analysis results of Enns and Gillham [9].

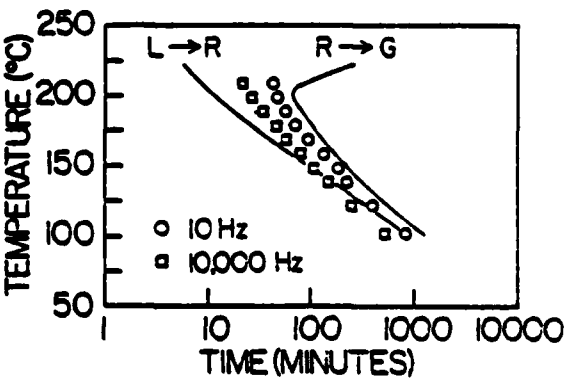

Fig. 7. Time-Temperature-Transformation diagram for EPON 825/DDS system showing time to reach 10,000 Hz and 10 Hz dipole relaxation loss peaks. Liquid to Rubber (gelation) and Rubber to Glass (vitrification) transformations from Torsional Braid Analysis results of Enns and Gillham [9].

As discussed earlier, the peak in the dielectric loss factor at a fixed frequency indicates that the average dipole relaxation time has reached a value equal to the reciprocal of the frequency. As curing proceeds, the peak is observed at successively lower frequencies, so the dipole relaxation time is increasing. We can plot these "events", the times to reach given dipole relaxation time, on the TTT diagram. Figure 7 shows a TTT diagram of the time to reach the dipole loss peak at 10,000 Hz and 10 Hz, again superimposed on the torsional braid gelation and vitrification events [9]. The countours of constant dipole relaxation time appear to parallel the vitrification curve of the TBA measurement. The vitrification of the resin occurs when the mobility of the reactive groups in the resin becomes so small that the reaction essentially stops. The dipole relaxation time is a measure of the mobility of the polar groups of the matrix. It is not surprising then that the vitrification event correlates with the rapid increase in the dipole relaxation time.

In contrast with the correspondance between the dipole peak and TBA vitrification, there is no clear "event" in the dielectric response corresponding to TBA gelation. In a previous paper [10] studying the cure of DGEBA with m-phenylene diamine (mPDA), we reported that a pre-gelation dielectric relaxation time approached "infinite" values at gelation, i.e. values beyond the measurement capability of the system. In Reference 10, that dielectric relaxation time was attributed to dipole orientation. We now understand that it arises from the ionic conduction and blocking electrodes. Furthermore, our improved instrumentation now permits even longer relaxation times to be observed. The data in this paper do not support the assignment of a divergence in the pre-gelation relaxation time to gelation. The crosslinking leading to the formation of a rubbery gel does decrease the conductivity, as evidenced in the ε'' values of Figures 3a and 4a. However, the crossing of the 60% conversion point, corresponding to divergence of the macroscopic shear viscosity at gelation, does not produce a singularity in conduction properties. The macroscopic shear viscosity reflects the ability of the molecules to undergo rearrangement over macroscopic distances, while the transport of ions through the material requires molecular rearrangement only on the atomic scale. While the ionic conductivity decreases with increasing crosslink density because of an increase in local or atomic-scale "microviscosity", the conductivity does not cease at gelation.

5. CONCLUSIONS

This study has examined the curing of a typical epoxy/amine system with dielectric measurements made over the frequency range 0.1 Hz to 10,000 Hz using Microdielectrometry. Two mechanisms of charge transport are evident in the measured dielectric loss factor, ε'', and permittivity, ε'. An ε'' inversely proportional to frequency and an abnormally large ε' suggest that an ionic conductivity dominates the response early in cure. Later in cure, a peak in ε'' concurrent with a fall in ε' characteristic of a Debye type relaxation indicates that the mobility of polar groups in the matrix is rapidly decreasing. The dielectric measurements were compared with the mechanical properties of the resin determined by torsional braid analysis [9], by plotting the time to reach the dipole relaxation loss peaks as a function of cure temperature on a time-temperature-transformation diagram. The cure temperature dependence of the time to peak correlates with the "vitrification event" observed by TBA, because both events depend on the mobility of the resin matrix. There is no evidence of a feature in the dielectric response which correlates with the "gelation event", because at that stage of

cure the dominant mode of charge transport is ionic conduction, which is a measure of "microviscosity" and not the macroscopic shear viscosity sensed by TBA.

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8. BIOGRAPHIES

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