Monitoring the Dynamic Mechanical Behavior of Polymers and Composites Using Mechanical Impedance Analysis (MIA)

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Synopsis

The techniques of mechanical impedance analysis (MIA) have been evaluated in terms of its potential for use as a reliable dynamic mechanical methodology. The method involves measuring the relation between a force input (excitation) and a motion output (response) between various points on the structure. In its most common and simple form, the force input and velocity output are measured at a single point of the structure and the values of the mechanical admittance (velocity/force) are plotted as a function of frequency. Rapid measurements and analysis of the dynamic mechanical properties of polymers and composites can be obtained by using a dual-channel FFT spectrum analyzer interfaced with a personal computer. It is demonstrated that the technique can be used to analyze polymer viscoelasticity, to study phase transformation of thermosetting resins, and to monitor cure of integrated composite structures. Also discussed are the potential advantages and limitations of the MIA techniques when applied in the fields of polymer science and engineering.

INTRODUCTION

A sizable number of techniques have been developed to study the viscoelastic behavior of polymers and composites. These include[1-6] (a) the transient measurements (creep and stress relaxation), (b) low-frequency vibrations (free oscillation methods such as the torsion pendulum), (c) high-frequency vibrations (resonant methods such as the vibrating reed technique), (d) forced vibration non-resonant methods (e.g., Rheovibron), and (e) wave propagation methods. Most of these techniques measure the deformation of a material in response to vibrational forces. The storage modulus (E'), loss modulus (E''), and mechanical damping or loss tangent ($\tan \delta = E''/E'$) are determined from these measurements. The study of these dynamic properties over a wide range of temperatures and frequencies has proven to be very useful in probing the structure of polymers and the variations of properties in relation to end-use performance.

Earlier models of dynamic mechanical apparatus were usually subject to one or more of the following drawbacks: (a) limited scope of applicability (e.g., some apparatus were only useful for studying liquids, gels, and solutions but not solids[7-11]), (b) only simple sample geometries were permitted, (c) data acquisition and reduction speed was in most cases too slow for

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the system to be used as a real-time monitoring tool for polymer processing,[12-14] and (d) the test methods were generally destructive in nature. During the past decade, the technology for investigating the dynamic mechanical properties of materials has advanced greatly due to new electronic circuits, improved transducer, better and faster signal receiving systems, and computerized controlling systems. New systems are generally faster and more convenient to use, although most of them are still subject to several limitations. For instance, Rheovibron (Toyo), DMTA (Polymer Lab.), DMA (DuPont), and Torsional Pendulum Apparatus cannot be readily used for studying viscous liquids (except for TBA[15] and TICA[18]) and useful frequency ranges of these systems are usually not very wide. Perhaps the most serious drawback to most of these systems is their destructive nature (e.g., limited flexibility in selecting sample shape and dimensions) that prevents them from being used as an in-process, real-time evaluation, or monitoring tool for material processing such as molding of thermoplastics and curing of adhesives and advanced composites.

Mechanical properties of thermosetting resins during cure through the various states of matter have been studied with different techniques[15-18] that all had one feature in common: The resins to be studied were supported by inert substrates. Torsional braid analysis (TBA)[15,16] is an extension of the torsion-pendulum method for examining the dynamic mechanical properties of materials. An advantage of using a supported-polymer technique is that specimen fabrication is generally simplified to deposition of the material from a melt or from solvent onto the substrate. The material can be investigated through various regions of the mechanical spectrum, including those in which the material may not be capable of supporting even its own weight. This provides an opportunity for studying resin-forming reaction, which may start in the liquid state and proceed through gel, rubbery, and glassy states. Torsion impregnated cloth analysis (TICA) is a variation of such techniques.[18]

Other major methods that have been developed for the cure study of thermosetting resins include dielectric techniques,[24-27,36,47] DC resistance measurements,[24,27] differential scanning calorimetry (DSC),[21-23] ultrasonic waves,[31,49-55] high-performance liquid chromatography (HPLC),[37] gel permeation chromatography (GPC),[37] Fourier transform infrared (FTIR), and others.[1,2,57,58] Although all these techniques can, in principle, be used to study the phase transitions in a thermosetting system, most of them (except for dielectric, DC resistance, and ultrasonic) cannot be employed as nondestructive techniques for the *in situ* cure monitoring and control of composite structures. This has been a subject of several recent reviews.[1,2,57,58] In the present report a relatively unexplored technique—the mechanical impedance analysis (MIA)—is presented and discussed in terms of its applicability as a general technique to measure the dynamic mechanical properties of polymers and as a process monitoring tool to follow the cure of thermosetting and composite materials and the molding of thermoplastics. The basic principles behind this technique will first be reviewed. Examples will then be given to illustrate the advantages of this technique and its limitations.

ANALYSIS

Vibration of A Simple Beam

For simplicity, let us consider only the damped vibration of a simple beam centrally loaded by a force $F = F_0 e^{jwt}$ with both ends free (Fig. 1). However, the analysis presented here may be extended to a wide range of structural configurations and boundary conditions. The mechanical impedance Z^* is defined to be the ratio between sinusoidal force and velocity (or acceleration) at a point of the beam. If the point where the force exerts and that where the velocity (or acceleration) is measured are the same point, the mechanical impedance measured is called a driving-point impedance; otherwise, it is called a transfer impedance. The reciprocal of Z^* is called mechanical admittance M^*. The driving point mechanical admittance of a free-free-ends beam with damping is[19]

$$M^* = -\frac{j}{dlw} + \frac{j}{dl}\sum_{i=1}^{\infty}\frac{x_i(1/2)}{w_{ni}[(1 - g_i^2) + jL_i]} \tag{1}$$

$$w_{ni}^2 = ki^4(E'I/d) \tag{2}$$

$$g_i = \frac{w}{w_{ni}} \tag{3}$$

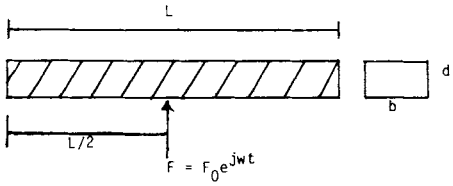
$$E^* = E' + jE'' = E'(1 + jL) \text{ and } L = \tan\delta = E''/E' \tag{4}$$

where $j = \sqrt{-1}$, d = linear density of the beam (mass/length), I = the moment of inertia, w = the angular frequency imposed on the system, l = the beam length, and the ki's are constants satisfying the following characteristic equation:

$$\cos kil \cosh kil = 1 \tag{5}$$

with their first several solutions being 0, 4.730, 7.853, 10.996, 14.137, 17.279,...,etc, and wni = the ith natural angular frequency for the ith normal mode of vibration

$$Xi(x) = (\sin kix + \sinh kix) + \frac{\cos kil - \cosh kil}{\sin kil + \sinh kil} * (\cos kix + \cosh kix) \tag{6}$$



A centrally-loaded beam with both ends free.

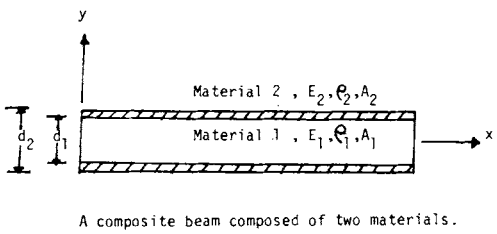
Fig. 1. A simple beam centrally loaded by a vibration force.

Equation (1) indicates that all the dynamic characteristics of a beam are related to M^*. The Z^* or M^* of a specimen can be measured by various methods, including sinusoidal sweeping, random excitation, and transient excitation test. This implies that it is possible to use some algorithmic approaches to identify the dynamic parameters such as E', E'', and tan δ from the admittance data. If the dynamic characteristics of a specimen varies with time and temperature, then so does the admittance. By studying mechanical admittance at regular time intervals, the variation of the dynamic characteristics of a thermosetting system can be obtained as a function of time and temperature.

Vibration of A Composite Beam

The curing of epoxy resins and similar materials is generally believed to be extremely difficult to follow.[38,39] The cure process starts from liquid components with liquid modulus and finishes with a highly crosslinked rigid glass of modulus above 10^9 Pa. So far it has been true that any single technique can only give absolute data over part of this curing range. In the early stages, dynamic viscosity measurements are required, replaced in the latter stages by dynamic modulus measurements. The compromised solution for measurement with a single specimen is to make the curing system part of a composite. The main techniques which have been used are impregnated braid,[15-17] impregnated cloth,[18] symmetrically supported coating on a substrate,[38] and one-sided coating.[38,39] In the latter two cases relavant equations have been given that permit the calculation of epoxy dynamic properties from the measurement of the properties of a steel "laminate" containing epoxy coating on one or both sides using the Polymer Lab Dynamic Mechanical Thermal Analyzer (PL DMTA).

In the present investigation, the specimens for cure study were prepared by either sandwiching the resin between two sheets of aluminum foil or filling the resin into a cylindrical tube with both ends sealed. The dynamic behavior of such a composite system therefore needs to be understood. Consider the transverse vibration of a beam with circular cross-section, as shown in Figure 2, which is loaded at the center with both ends free. Elementary elasticity indicates that it is possible to calculate the modulus and loss tangent of the material interested given the dimensions of both components, the modulus of material 2 (container), and the linear mass densities. Equations (1)–(6) and simple rules-of-mixture equations (similar to those used in Refs. 38–41) may be employed to obtain the absolute prop-



A composite beam composed of two materials.

Fig. 2. A composite beam with a circular cross section. Material 2 represents the container while material 1 the resin or prepreg being cured.

erties of a thermosetting material. For cross sections other than circular, similar equations can be readily obtained.

EXPERIMENTAL

The principal object of the present investigation was to demonstrate the scientific feasibility of and the advantages to be gained from using the mechanical impedance analysis (MIA) in studying the dynamic mechanical behavior of polymers and composites. The first set of experiments were therefore designed to determine if the MIA apparatus established in our laboratory could be readily used for studying polymer viscoelasticity; in particular, the dynamic properties (e.g., tan δ) as a function of temperature and frequency. The second group of experiments were undertaken to qualitatively determine the feasibility of using MIA as a means of monitoring cure of composite materials. The third task represented the effort to quantitatively monitor the curing process of simple composite beams using our MIA setup as well as PL DMTA. Task 4 was to utilize a simplified model composite system to simulate the actual molding process of a complex composite structure and to quantitatively follow its curing behavior.

Materials

The E-glass fiber-epoxy prepreg tapes were supplied by 3M Co. (commerical name Scotchply 1003) and used as received. Both isotropic and crossply tapes were studied. For the cure monitoring study using PL DMTA, one layer of isotropic tape was cut into rectangular shape and sealed in an aluminum enclosure to prevent epoxy flash while in the liquid state. For the samples tested by MIA technique either 12 layers of crossplied or 6 layers of isotropic tapes were utilized. The temperature dependence of dynamic properties of a rubber-modified Nylon (DuPont Zytel 801ST) was also investigated along with several grades of high impact polystyrene (HIPS).

Apparatus

The MIA setup may be schematically shown in Figure 3 where the sample, a free-free beam loaded at the middle (other structural configurations and boundary conditions are also feasible), is excited with its force and velocity response measured and analyzed real-time through a fast Fourier transform (FFT) analyzer and a personal computer. The signal analyzer used here is a dual-channel FFT (B&K Model 2032), although other instruments with similar functional capability could be used equally well. The mechanical admittance data after a selected number of scans can be readily displayed in the FFT screen or the screen of a personal computer and plotted out using a printer or a plotter. The impedance head, connected to the sample through an extension rod, has two built-in transducers to measure the force and velocity, respectively. The signals detected are amplified by two charge amplifiers and then analyzed by the FFT. The driving force (excitation) comes from the shaker (exciter) driven by a separate signal generator or directly by the FFT with an internal signal generating device. The result of a normal FFT analysis shows a distribution of frequency from zero up to the Nyquist frequency fN, while the frequency resolution is determined

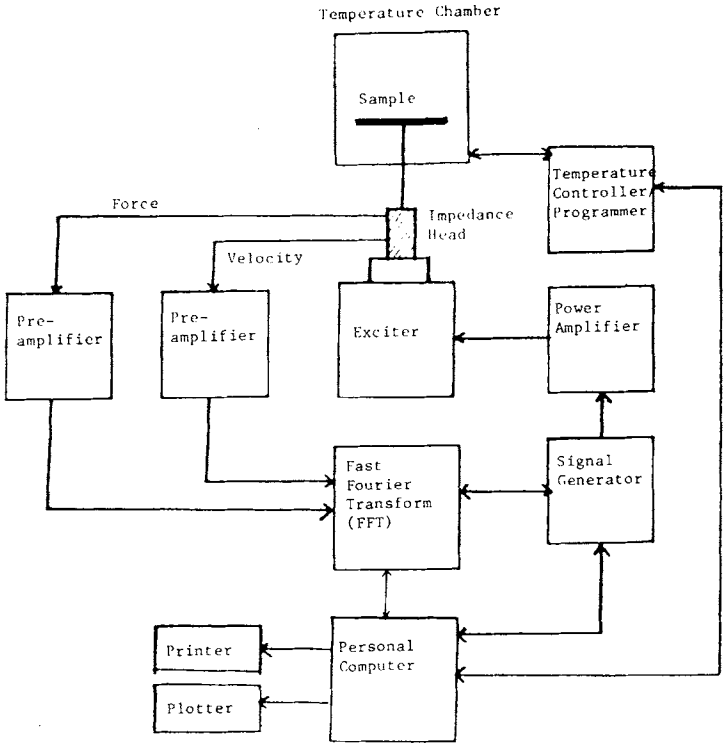


Fig. 3. Schematic of the mechanical impedance analysis apparatus.

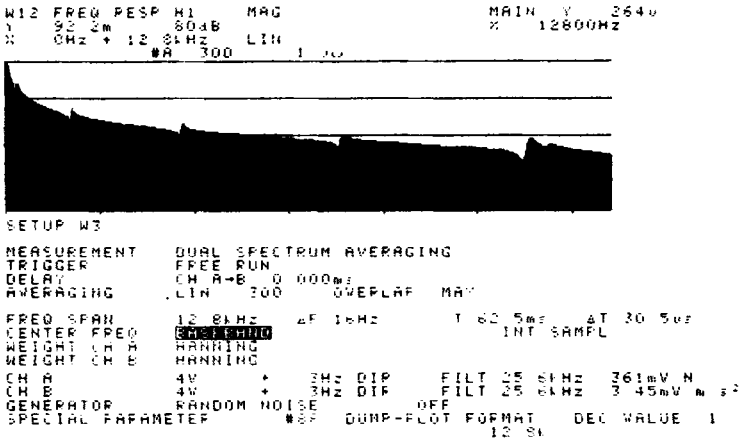
by the number of frequency lines up to fN. The advent of Zoom-FFT provides a method by which increased resolution can be obtained within a smaller part of the frequency range.

RESULTS AND DISCUSSION

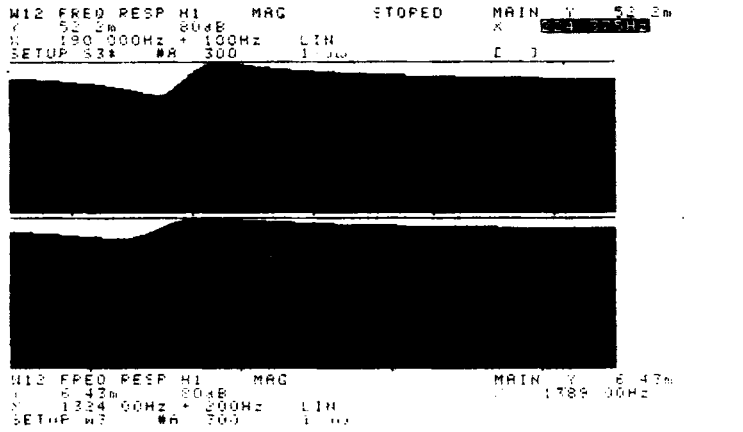
Dynamic Mechanical Behavior of Polymers

A study was undertaken to assess the dynamic response of thermoplastics using the technique of MIA. Eight different grades of high impact polystyrene (HIPS) were investigated. Typical outputs of the mechanical admittance (velocity/force) data that can be viewed on the screen of an FFT or personal computer and printed out through either instrument are shown in Figure 4, where the ordinate represents the amplitude of the admittance while the abscissa the frequency spectrum. As shown in Figure 4(a), except for the largest peak at the far left end ($w = 0$), which is a measure of the rigid body mation, there are five peaks representing five normal modes of vibration. The location, shape, and size of each peak can be used to determine the dynamic parameters. The storage modulus E' can be calculated from eq. (2) if the natural frequency w_{ni} is identified from the location of each peak. The values of loss tangent can be read off directly from each peak of the graph using the half-power points method:

$$\tan \delta = \frac{E''}{E'} = \frac{w_{i2} - w_{i1}}{w_{ni}} \tag{7}$$



(A)



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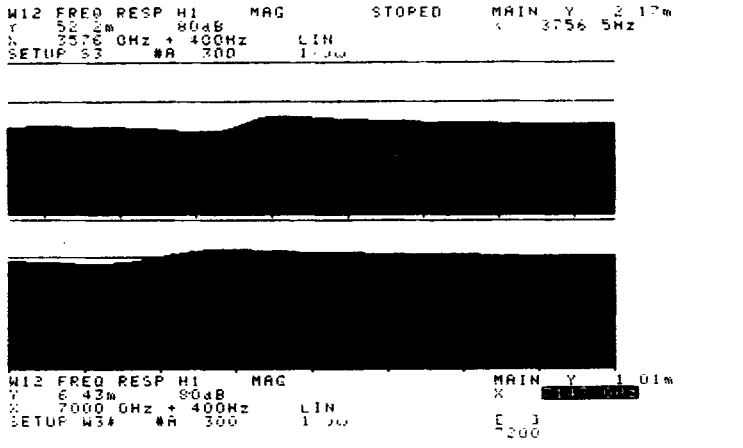
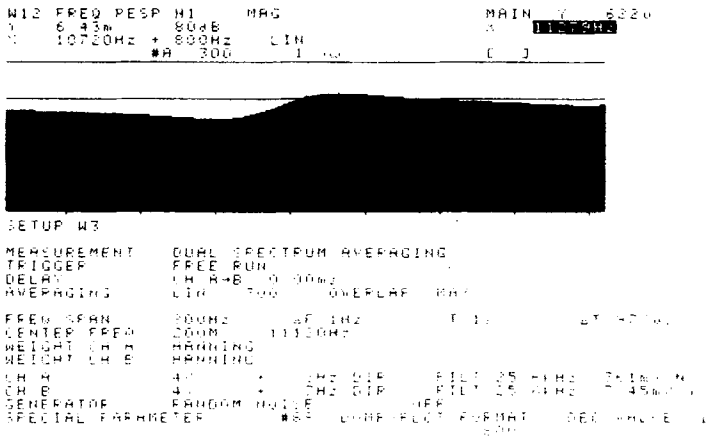


(C)

Fig. 4. Typical mechanical admittance output obtained from the vibration of a simple beam of high impact polystyrene.



(D) Fig. 4. (*Continued from previous page.*)

where $M^*(w_{i2}) = M^*(w_{i1}) = 0.707M^*(w_{ni})$ defines the locations of frequency spectrum where the admittance value has dropped from a peak to 0.707 of this maximum value. Other techniques such as curve-fitting can also be used. The values can be obtained in real time with the help of a personal computer. Once the values of E' and loss tangent are determined, then so is that of E''.

To increase the resolution of analysis, the zooming mode of FFT analyzer is particularly useful. In Figures 4(b)–(d) are shown the zoom spectrum of each peak within a smaller part of the frequency range. The zoom spectra are of high resolution so that the resonant frequencies and damping ratios may be obtained directly and clearly. It is clear that essentially all the dynamic mechanical parameters of a polymer can be obtained by the techniques of MIA. The dynamic behavior can only be followed at a few (practically seven) discrete frequencies for a typical small rectangular bar (e.g., dimensions 5 × 1/2 × 1/8 in.). However, the maximum frequency that a specimen can be tested is only limited by the working frequency range of an instrument. A frequency of 20,000 Hz is the limit imposed on the B&K Model 2032.

A useful dynamic mechanical method should be sensitive enough to detect molecular relaxations such as those occuring at the glass transition T_g and other secondary relaxations. To testify the sensitivity of the MIA technique in studying polymer viscoelasticity, a number of materials were tested as a function of temperatures. Figure 5 indicates the temperature dependency of tan δ of Zytel 801ST, a rubber-modified Nylon from DuPont. A large relaxation peak can be clearly identified which is presumably due to the glass transition of the polyamide phase. A couple of peaks appeared in the tan δ vs. w curve for materials like HIPS, which have been tested over a wider temperature range.

Qualitative Observations of A Composite Curing Process

A group of glass fiber–epoxy prepreg tapes were cured at an oven with a preset temperature and then removed for testing at T = 23°C intermittently after varying periods of time. Several different curing temperatures,

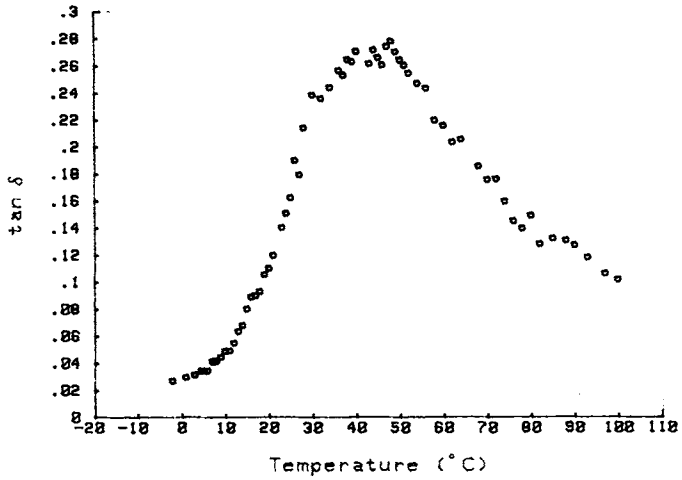


Fig. 5. The temperature dependency of loss tangent of a rubber-modified polyamide.

ranging from 110 to 180°C, were utilized. Twelve layers of crossply tapes were stacked to form a rectangular specimen and then sealed with aluminum foil.

Typical outputs of the admittance data are shown in Figure 6, where the prepreg specimen has been cured intermittently at 140°C for a series of lengths of time. At time $t = 0$ (before curing), the curve hardly exhibits any peak where maximum admittance exists. This is characteristic of any viscoelastic liquids with very small rigidity and great damping capacity.

As curing proceeds the rigidity increases while the damping ability decreases, as reflected in the following observations:

1. A monotonically increasing number of peaks shape up with time. For instance, at time $t = 5$ min, the first peak can be clearly identified while the second one can barely be seen. After 15 min of curing at least three peaks can be recognized and, at $t = 25$ min, five peaks clearly stand. Such peaks, from left to right with increasing frequency, correspond to the 1st, 3rd, 5th, 7th, and 9th normal modes of vibration, respectively.

2. As cure time increases, the peak values of the lower-order modes ascend while those of the higher-order ones descend. The peaks generally become narrower and sharper with cure time. However, we noticed that overcuring of an epoxy resin often results in broadening of a peak.

3. The zeroth order peak located at the far-left end corresponds to the rigid-body motion of the beam and is a measure of the material rigidity. The peak increases in amplitude very rapidly as the crosslinking level rises and material becomes more rigid.

The purpose of this phase of study was to assess the sensitivity of MIA technique when applied as a tool to monitor the cure process of a thermosetting material system. The results of such an extensive (a few different material grades were investigated and several different temperatures involved) and detailed study, albeit qualitative, appear to be very encouraging. At this stage of study no attempt was made to calculate the absolute value of the storage modulus of the prepreg tape from the measured E' of the sandwich composite system (upper and lower layers being a thin Al foil

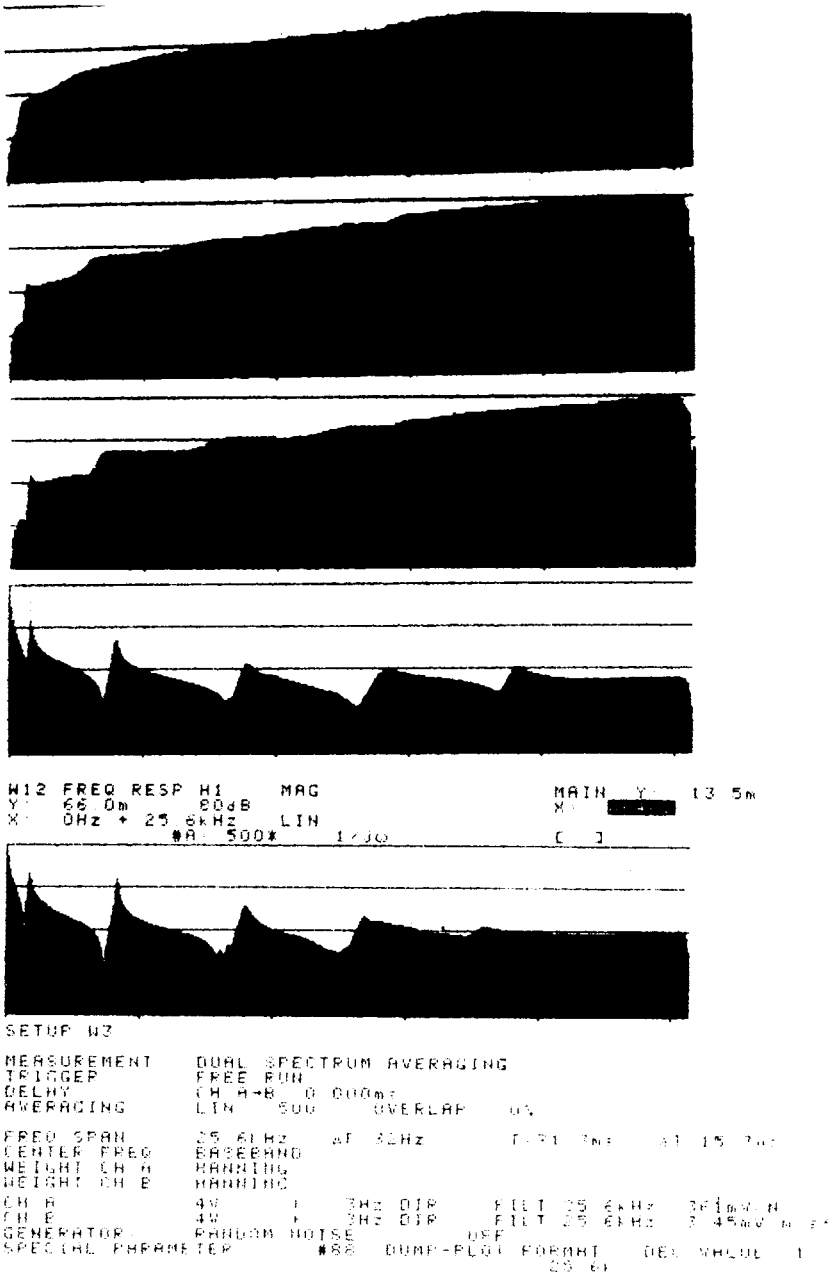


Fig. 6. The admittance data of epoxy–glass fiber prepreg stacks cured at 140°C and intermittently tested at 23°C.

while the middle layer the prepreg). Since the damping characteristics of the Al foil do not change with time, any variation in this property is believed to be due to the crosslinking process of the epoxy resin.

Quantitative Study of the Curing Process

To put the cure monitoring technique on a more quantitative perspective a temperature chamber that affords an accuracy of ± 0.5°C was constructed. The cooling medium for the low temperature study is liquid nitrogen while the heat is supplied by four built-in medium-power heating elements. The system was driven by an Omega proportional-band type of temperature controller. The first specimen tested was a rectangular laminate of crossply prepreg tapes with dimensions $5 \times 1/2 \times 1/8$ in. The bar was sealed in Al foil and mounted when still cold on the extension rod connected to the impedance head. The specimen was slowly heated from 25 to 135°C and then held at 135°C thereafter for isothermal curing. It took approximately 30 min for the specimen to reach 135°C. The moment when the temperature read 135°C was assigned to be time = 0 and the damping characteristics of this specimen were followed subsequently. The results of this vibration test are shown in Figure 7, where tan δ as calculated from the mechanical admittance curves using the half-power-point method is plotted against the cure time. It is clear that tan δ is a sensitive indicator of the cure status of the epoxy resin involved. By following the values of tan δ and other dynamic parameters, the maturity of the network formation can be gauged.

In order to understand the possible effects of the slow heating process upon the curing of epoxy resin both tan δ and E' were measured during heat-up. One measurement each for tan δ and E' was taken per °C of temperature increment. As exhibited in Figure 8, the storage modulus E' decreases monotonically until about 145°C where it begins to increase. It appears that pronounced crosslinking or gelation has taken place ahead of

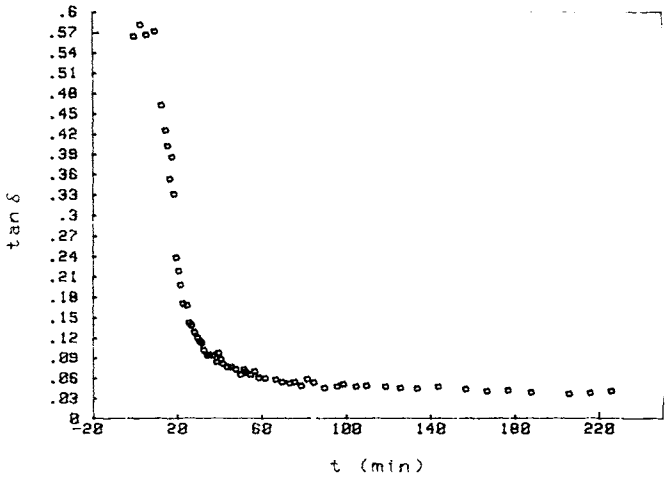


Fig. 7. The loss tangent of a glass fiber–epoxy prepreg specimen as a function of cure time (cure temperature = 135°C).

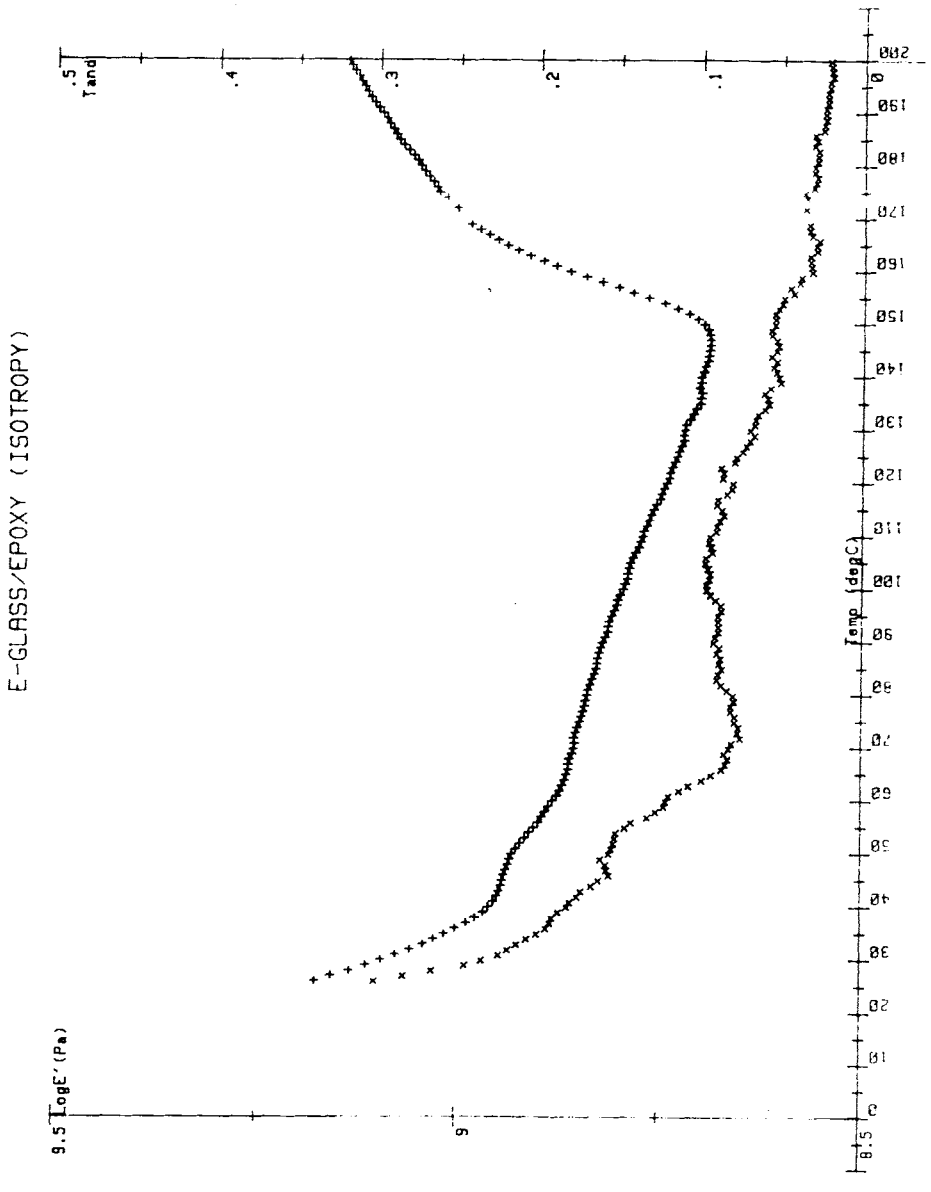


Fig. 8. Monitoring of cure status of epoxy–glass fiber prepreg specimen subjected to a cure program of incremental temperature.

this temperature. It is expected that there exist two competing effects during heat-up from 25 to 145°C. On the one hand, the rising temperature promotes molecular mobility, reducing the viscosity and rigidity of the liquid resin and resulting in the trend of decreasing viscosity. On the other, an increase in temperature should lead to a higher reaction rate. Once the crosslinking process takes place, the viscosity would rise dramatically, and this phenomenon predominates at higher temperatures. A combination of tan δ and E' data provides an effective and accurate tool to monitor and control the cure of thermosetting and composite materials. The three apparent transitions, located at about 47, 105, and 148°C, seem to be due to the softening, gelation, and vitrification, respectively, of the epoxy resin.

Gillham[16] has been successful in utilizing an automated torsion pendulum (TBA) for monitoring the curing reaction of epoxy resin impregnated in glass braids. The experimental results at a series of curing temperatures were used to obtain estimates of the gelation time and the vitrification time vs. cure temperature. These transformation times were measured using peaks in the mechanical damping curves and summarized in a phase diagram called "TTT diagram." The results of Gillham's phase transformation study indicate that there are three types of behavior depending on the curing temperature. At high temperatures the liquid gels but does not vitrify. At low temperatures the liquid vitrifies and need not gel if the chemical reactions are quenched by vitrification. At intermediate temperatures the liquid first gels and then vitrifies. These observations suggest that competition exists between the increased rate constants for reaction and the increased degree of reaction required to overcome the thermal motions for vitrification at higher temperatures. The curing process should be well designed and monitored if the structure, and therefore properties, of the resin are to be controlled.

Effort was also made to study the phase transformation behavior of thermosetting resins using the technique of MIA.[42] A similar study was also conducted using the PL DMTA. Shown in Figure 9 are the data of tan δ and E' of crossply prepreg specimens isothermally cured at four different temperatures. Data points symbolized by the "cross" and "dagger" were the results of curing at 160°C while the solid and dotted lines were carefully traced from the data points of curing at 130, 140, and 150°C, respectively. The DMTA was operated in such a way that the specimen chamber was preheated to the desired cure temperature. Approximately 10 min were allowed for the specimen to reach the thermal equilibrium before the instrument was put on the isothermal mode at time = 0. To meet this need, the commercial software package provided by Polymer Lab., Inc. was suitably modified.

It may be noted that the values of tan δ and E' obtained from the present study are those of the Al-Prepreg-Al laminate. However, it is straightforward to calculate the absolute material properties as discussed in the Analysis section. Wetton et al.[38] have provided equations for conveniently estimating the absolute properties for a symmetrically coated or one-side coated beam measured in a single cantilever bending mode. In order to prevent the liquid flash problem these workers[38] had to turn the sample head 90° to an upright position so that the specimen lies horizontal. With

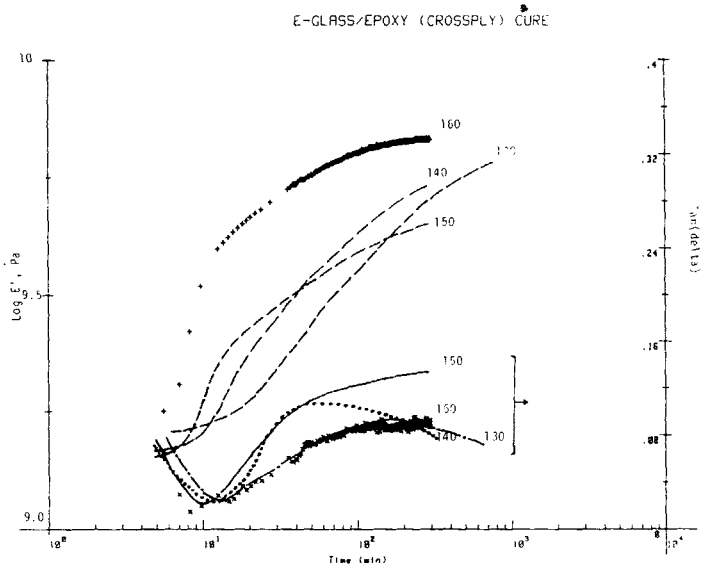


Fig. 9. Dynamic characteristics of prepreg samples as a function of isothermal cure time and temperature.

the specimen lying horizontal, however, one still has to worry about the problem of liquid flow and buildup at the clamping locations even with one-side coated specimen. We have found it much more convenient to seal the material in a thin aluminum enclosure. A rectangular container was used although any other geometry with a regular cross-section (e.g., circular) could also be used. The same equations used by Wetton et al.[38] may be adapted with the modification that the top and bottom layers are now aluminum with the material of interest sandwiched inbetween.

Cure Monitoring of a Model Composite Structure

In the previous phases of study only the dynamic properties of isolated, simple rectangular bars were investigated. It has been proved that, with this simple geometry, the MIA technique is a useful tool for studying polymer viscoelasticity and for following the phase transformation processes of thermosetting material systems. As far as practical applications are concerned, it remains to demonstrate that the MIA technique is appropriate for use in monitoring the cure of a real composite structure. Bearing this objective in mind, we designed a simple compression mold for fabricating composite laminates. As shown in Figure 10, the mold is composed of two mating pieces of aluminum that can be bolted together to form a cavity to accommodate the laminate. The bottom piece was screwed down to the wall of the temperature chamber at the four corners. An extension rod with both ends threaded was connected to the geometric center of the bottom piece at one end and to the impedance head at the other.

The study began with the measurements of mechanical admittance of the system containing only the bottom piece of mold. This was followed by a study of the system consisting of both pieces in place but still with the mold cavity being empty. The two control experiments were conducted at

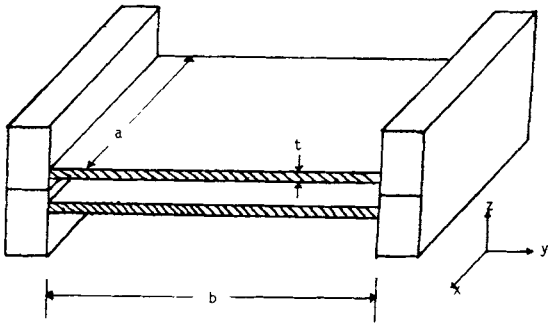


Fig. 10. The compression mold used in the model study of composite structure cure process.

the desired isothermal cure temperatures. As shown in the first graph of Figure 11 there exist two major peaks (peaks #1 and #3) within the frequency range scanned (0–6.4 KHz). It can be shown from simplified calculations[42] that these two peaks correspond to the resonant frequencies for two of the normal modes of vibration ($N = 1$, $M = 1$ and $N = 1$, $M = 3$) of a rectangular Al plate with two sides fixed and the other two free. The damping coefficients of both peaks are relatively low. Attachment of the mating piece to the first one appears to increase the resonant frequency, and thus the storage modulus, as well as the damping capacity. The second graph of Figure 11 indicates the lowering and broadening of these peaks due to the second plate.

Addition of a prepreg sample in the cavity further enhanced damping, as shown in the third graph of Figure 11. The measurement was taken at 100°C during the heat-up period. Increasing the temperature to 128°C almost forced peak #3 to disappear while in the meantime another peak (#2) began to shape up. Peak #2 became to dominate as curing proceeded. This peak seemed to be associated with the prepreg material in the cavity; this has been confirmed by a theoretical calculation.[42] A series of spectra of the mechanical admittance for the system after different periods of cure time were collected in Figure 12. The bottom graph represents a zoomed version of the graph after a cure time of 203 min at 140°C. Spectra of this nature are a reliable index of the curing status. The values of tan δ obtained from peak #2 for cure temperatures 130 and 140°C are shown in Figure 12.

For a given composite curing configuration, either a compression molding press or an autoclave, the mechanical admittance spectra of the system without material of interest can be obtained as the reference data against which the future spectra can be compared. With the fiber–resin material in place for curing, any new features in the spectra should be directly or indirectly ascribed to the material of interest. The cure of the material can therefore be monitored by following the variations in the dynamic characteristics.

The Advantages of MIA

The mechanical impedance techniques are not new. For instance, Smith, Ferry, and Schremp[10] have used the impedance concept to study the mechanical properties of polymer solutions and gels while Fitzgerald and

Ferry[11] used an essentially identical technique to determine the dynamic mechanical behavior of solids at audiofrequencies. The advent of new digital electronic instrumentation has made it possible to achieve rapid and accurate data acquisition and reduction in the vibration tests. Complex computation and analysis can now be executed almost instantaneously using equipment such as the FFT and a digital computer. Therefore, it should be technically feasible to utilize the MIA technique to monitor and control the fabrication process of any viscoelastic material system. In the previous studies (e.g., Refs. 10 and 11) the possibility of using MIA for following the processing of materials was not recognized. The techniques of MIA utilized in our laboratory appear to have the following advantages:

1. Automatic frequency sweeping over a wide range of frequencies and temperatures can be readily performed. Therefore, the dynamic mechanical properties of polymers can be conveniently studied. Since this is a stand-alone apparatus, the system can be easily modified to suit the needs of other vibration tests.

2. The MIA method affords a great deal of flexibility in selecting sample configuration and loading mode.

3. A wide scope of materials and physical states may be tested. These include thermoplastics and thermosetting polymers, neat resins and com-

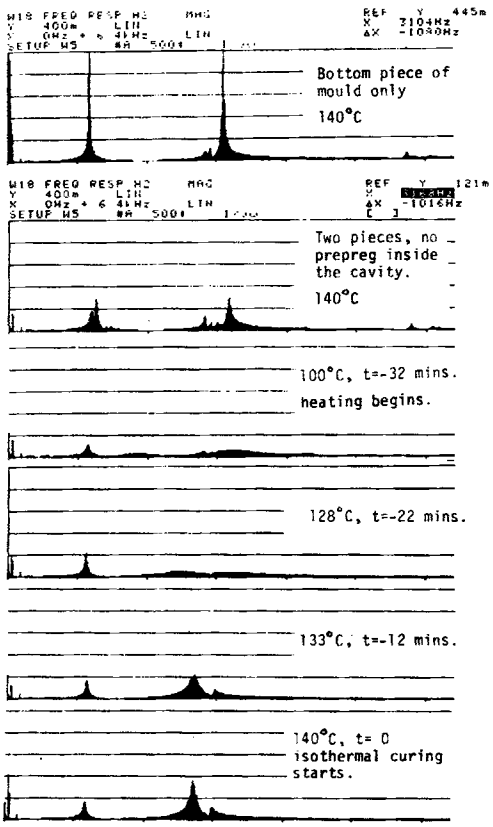


Fig. 11. Cure status of a composite structure as monitored by the MIA techniques.

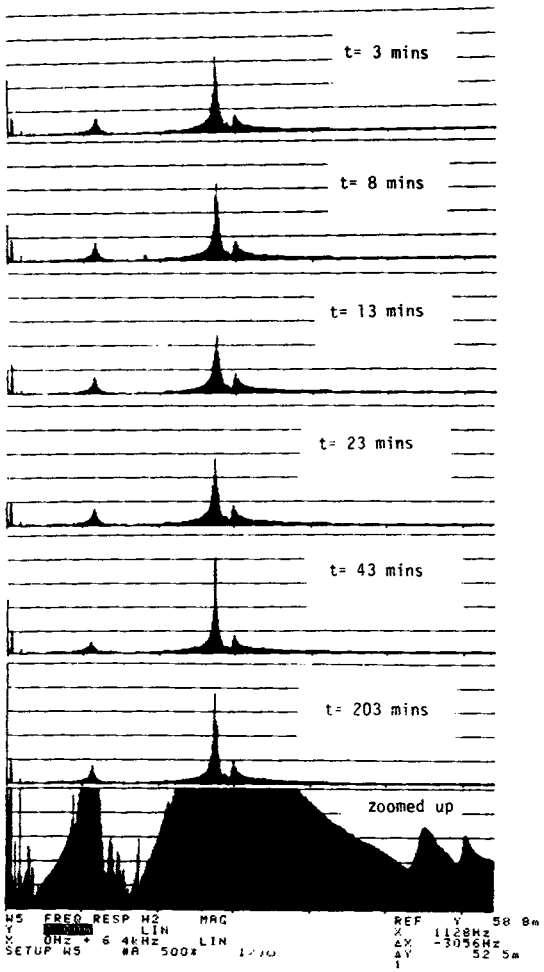


Fig. 11. (*Continued from previous page.*)

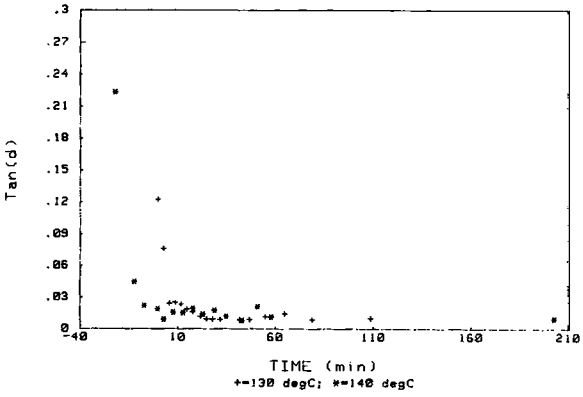


Fig. 12. The loss tangent of prepreg material in a model composite structure as a function of cure time, time and temperature.

posites, and states of materials such as liquid, rubber, gel, glass, and crystalline. An inert substrate or container may be used when studying a high-flow liquid which cannot support its own weight,[9] as suggested by the concepts of TBA[3-5] and TICA.[6]

4. With appropriate modification to the apparatus and suitable selection of the oscillation mode, nondestructive evaluation (NDE) of material is possible.

5. Since the data acquisition and reduction can be handled very rapidly and accurately, the technique may serve as an effective, real-time monitoring tool for polymer and composite processing.

CONCLUSION

The scientific feasibility of and the advantages to be gained from using MIA techniques for studying the dynamic mechanical behavior of polymers and composites have been explored. This investigation leads to the following conclusions:

1. The mechanical impedance or admittance spectrum obtained from the simultaneous measurements of the force and velocity of a beam subjected to a cyclic loading contains the information concerning the dynamic properties of the material. The storage modulus E', loss modulus E'', and damping (tan δ) of the material system can be calculated near a series of natural frequencies in the transfer functions.

2. Polymer viscoelasticity can be studied using MIA since the dynamic mechanical properties of polymers can be conveniently measured as a function of temperature and, to a lesser extent, of frequency. The maximum frequency that can be imposed upon the material is only limited by the highest available frequency from the instrument. A frequency as high as 20 kHz is readily available.

3. The MIA techniques can be used to investigate the phase transformation behavior of a thermosetting resin. The absolute values of the dynamic properties as a function of the degree of cure can be obtained by testing the material in a simple configuration.

4. The MIA techniques could be employed as a real-time, nondestructive evaluation (NDE) tool for monitoring the cure of a complex composite structure.

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