Dynamic Mechanical Analysis of Fiber-Reinforced Phenolics

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ABSTRACT: Dynamic mechanical analysis (DMA) was used to investigate the thermomechanical behavior and the effects of postcuring on a range of glass-reinforced phenolics. The materials examined were a pure resol (reinforced with S- and E-glass), a pure novolac (reinforced with S-glass), and three derivatives of the resol and/or novolac: a resol/novolac blend, a phenolic-furan graft copolymer, and a rubber-modified resol (all reinforced with S-glass). The blend and copolymer were prepared to obtain phenolic resins with improved impact strength, without degeneration of their high-temperature performance. They have a more loosely crosslinked structure compared to the pure resol or novolac. The rubber-modified resol was prepared with the intention of reducing the brittleness of the resin structure by incorporating an elastomeric phase within the resol resin matrix. It was found that the stiffness and glass transition temperature (T_{σ}) of the materials could be increased by postcuring, which also produced a decrease in their damping capacity. Knowing that the postcure process is a function of time and temperature, a master curve was constructed that allowed prediction of the T_g of the resol/novolac blend over a broad range of postcure times and temperatures. The effect of frequency on the storage modulus of the pure resol (S-glass), copolymer, and blend was also studied from 0.01 to 100 Hz. Master curves were constructed by timetemperature superpositioning that allowed prediction of the storage modulus at times and temperatures that are not experimentally accessible. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 73: 649-658, 1999

Key words: phenolic composites; dynamic mechanical analysis (DMA); postcure; crosslinking; storage modulus; tan δ ; glass transition temperature; frequency; time-temperature superpositioning

INTRODUCTION

Some of the properties of phenolics, including the glass transition temperature (T_g) , can be improved by postcuring, that is, heat-treating the material after it has already been cured. This is because phenolic resins are not completely cross-linked during the curing process and the degree of crosslinking increases with postcuring. Postcuring in the case of resols is just a continuation of

the polymerization reaction. The various dimers and trimers formed during the phenol-formaldehyde reaction crosslink with each other during the curing process to form a tightly crosslinked three-dimensional network structure. After postcuring, the material can still continue to crosslink due to the presence of some active sites in the molecular structure. A sense of the degree of crosslinking of a material can be obtained from a measure of the modulus, which increases with postcuring. Previous studies have indicated that there is no difference between a short postcure time at a high temperature.¹ This article discusses the thermomechanical behavior and effects of

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postcuring on a range of glass-reinforced phenolics and provides a relationship that allows prediction of the T_g over a broad range of postcure times and temperatures using dynamic mechanical analysis (DMA). The materials studied include pure resol (reinforced with S- and E-glass)and novolac (reinforced with S-glass)-based composites and three modified composites (reinforced with S-glass): a resol/novolac reactive blend, a phenolic–furan copolymer, and a rubber-modified resol.

DMA is a very useful technique for investigating the mechanical behavior of a material based on its viscoelasticity. Generally, DMA can provide more information about a given polymeric material than can other mechanical tests since it can cover a wide range of temperatures and frequencies, which are not possible with others.² A dynamic mechanical test measures the response of a material as it is deformed due to a sinusoidal or other periodic stress. Three important parameters that can be obtained during a dynamic mechanical test are (a) storage modulus, which is a measure of the maximum energy stored in the material during one cycle of oscillation and which gives an indication of the stiffness behavior of the sample; (b) loss modulus, which is directly proportional to the amount of energy that has been dissipated as heat by the sample; and (c) a mechanical damping term, tan δ , which is the ratio of the loss modulus to the storage modulus and is related to the degree of molecular mobility in the material. Some of the other useful quantities that can be characterized by DMA include storage and loss compliance, dynamic and complex viscosity, creep compliance, and the stress-relaxation modulus.

The T_g is usually interpreted as the peak of the tan δ or the loss modulus curve that is obtained during a dynamic mechanical test conducted at a low frequency. The ability to measure large changes in the modulus and tan δ in the glass transition region using DMA improves the probability of making a correct assignment of the T_g of any material. It is well known that the T_g of polymeric materials is not a constant but varies with time. If DMA measurements are made at a rapid heating rate, then the apparent T_g is increased, whereas if they are made at a very low heating rate, the apparent T_g will be a lower value.³

Most commonly, DMA tests are conducted in a fixed frequency and constant amplitude (strain)

mode, in which the response of a material is studied as a function of temperature only. Other measurements that can be made using a dynamic mechanical analyzer include multifrequency and multistrain tests, creep, and stress-relaxation studies. By using multifrequency data at a variety of temperatures, the method of reduced variables or time-temperature superpositioning (TTS) can be used to make long-term time-dependent predictions of some of the properties of the material.⁴ The shifting is usually done using one of two well-established models, the Arrhenius model⁵ or the Williams–Landel–Ferry (WLF) model,⁶ depending on the reference temperature used during the master curve construction. This article examines the frequency-dependent behavior of the resol/novolac blend, the graft copolymer, and the rubber-modified resol and provides some explanation of their resulting performance. Also, long-term predictions of the storage modulus of these materials as a function of temperature and time (or frequency) will be made using TTS.

EXPERIMENTAL

All the composite sheets used in this study were supplied by the Plastics Engineering Co. (Plenco), Sheboygan, WI. The sheets fell into two broad categories: The first consisted of pure fiber-filled resol and novolac sheets, and the second consisted of sheets that were derivatives of the resol and/or the novolac which were prepared with the intention of improving their impact and thermal stability without degeneration of fire performance. All the sheets were compression-molded using 0/90 mats of S- or E-glass fibers.

Resins

The resol used in this study was a thermoset resin synthesized from formaldehyde and phenol in a ratio of 1.266: 1. It has a number-average molecular weight of 139 and a weight-average molecular weight of $173.^7$ The novolac resin was prepared from phenol and formaldehyde in the ratio 1.393: 1 and had a number-average molecular weight of about 1000 and a weight-average molecular weight of about 15,000. The novolac was initially a thermoplastic and was cured by addition of 10% hexamethylenetetramine (HMTA).⁷

A graft copolymer was prepared from a phenolic-furan novolac and the resol. The phenolicfuran novolac was first prepared by reacting furfural and phenol, and this polymer was then cured with the resol to produce a block-type graft copolymer having an irregular network structure consisting of resol and novolac segments. The copolymer has a number-average molecular weight of about 600 and a weight-average molecular weight of about 2000.⁷

A rubber-modified resin was prepared from the resol using 7% ABS rubber (Nipole DP 578) to form a dispersed elastomeric phase. The rubber-modified sheets were prepared with the intention of reducing the brittleness of the resin structure.

The resol and novolac resins were also mixed in a ratio of 70 : 30 to produce a reactive blend. Despite the presence of novolac resin in the blend, no external crosslinking agent was added to cure this modified resin since the novolac resin itself can partially crosslink with the resol resin during cure. This is possible because the *ortho* and *para* positions of the hydroxyl groups in the novolac resin are still active with respect to the remaining methylol groups of the resol resin which allows for crosslinking at these sites during cure of the blend. A loose network structure is obtained that has been found to retard microcrack formation at high temperatures.⁸ The absence of external curing agents means that less gas is released during the curing process, thereby reducing the formation of microcracks and the presence of molded-in stresses. Further details on the preparation methods for the modified resins is available in a previous article on glass-reinforced phenolics.⁷

Composites

The resins were combined with 10% by weight methanol and blended in a high-speed mixer. Then, 0/90 mats of glass fibers were laid out on tables and the resin mixture applied by hand layup. After uniform application of the mixture, the excess was squeezed out and the mats were then hung in an oven set at 90°C for 45 min. Seventeen of the impregnated fiber mats, cut to standard size, were stacked in a platen press, in order to obtain sheets of $\frac{1}{8}$ -in. thickness, and molded at 4.9 MPa and 145°C for 20 min. The glass loading for all the molded sheets was determined to be between 65 and 70%.

Postcuring of Composite Specimens

Postcuring of the specimen coupons was accomplished by heating them in an oven for specific periods of time at the set postcure temperature of 180°C. This temperature was chosen initially since it is a standard postcure temperature used in the automotive industry. Although previous studies have indicated that the interruption of a postcuring process has no effect on the resultant behavior of a material,¹ care was taken to see that there was no break during the postcure. Coupons $(2.5 \times 0.5 \text{ in.})$ of $\frac{1}{8}$ in. thickness from all the materials were cut at a rate of 1.0 cm/min to minimize any frictional effects on the material.

Dynamic Mechanical Analysis

The dynamic mechanical tests were performed using a TA Instruments DMA 2980 dynamic mechanical analyzer. The data were collected and analyzed using TA Instruments Thermal Solutions software. The DMA 2980 provides a good operating temperature range of -145 to 600°C. The use of a high-resolution optical encoder displacement sensor provides excellent modulus accuracy ($\pm 1\%$) and tan δ sensitivity (up to 0.0001).⁹ Some other relevant features of the DMA 2980 include allowable heating rates of 0.1–50°C/min, cooling rates of 0.1–10°C/min, and an experimental frequency range of 0.01–200 Hz.

The DMA 2980 has a variety of clamping configurations that allows for experimentation with a wide spectrum of specimens, within specific dimensions, such as rigid thermosets, thermoplastics, elastomers, thin films, fibers, and viscous liquids. Depending on the experimental sample and the type of clamping configuration, testing can be done under flexure, compression, tension, or shear mode of deformation.

In this study, the specimens were deformed under flexure using a single cantilever clamp arrangement. The DMA tests were carried out in the following two modes: (a) a fixed-frequency mode in which the frequency was held constant at 2 Hz and the temperature was ramped from ambient to 350 or 400°C (depending on the material) at a heating rate of 5°C/min, and (b) a multifrequency isothermal hold mode in which the amplitude of deformation was held constant and experiments were performed on samples at constant temperature and varying frequency. The samples were subjected to frequencies between 0.01 and 100 Hz at a variety of temperatures for use in TTS, which was carried out using the TA Instruments Rheology Solutions TTS software.

RESULTS AND DISCUSSION

The importance of the dynamic storage modulus in many structural applications is well known. A clear understanding of the storage modulus-temperature curve obtained during a dynamic mechanical test provides valuable insight into the stiffness of a material as a function of temperature. Figure 1 shows the storage modulus-temperature curves of the six un-postcured phenolic materials. All the materials exhibit different modulus-temperature behavior depending on their structures and processing conditions. The decrease in modulus around the glass transition region is more accentuated for the novolac, copolymer, and, to a lesser degree, the blend, due to their initially more loosely crosslinked structures. The modulus-temperature curve is useful in assessing the molecular basis of the mechanical properties of materials since it is very sensitive to structural changes such as molecular weight, degree of crosslinking, and fiber-matrix interfacial bonding.

The initial storage modulus is defined as the first storage modulus recorded in the DMA modulus-temperature plot and the final storage modulus is defined as the last storage modulus recorded in the same plot. On comparing the initial storage modulus values of the un-postcured specimens, it can be seen that the novolac had the highest value followed by the copolymer, rubber-



Figure 1 Storage modulus-temperature curves of the un-postcured phenolic materials.



Figure 2 Initial storage modulus values of the unpostcured phenolic materials.

modified resol, and resol/novolac blend, respectively (Fig. 2). The pure resol samples reinforced with S- and E-glass had lower initial storage modulus values compared to the others.

The high initial storage modulus value of the novolac could have two possible explanations: (1) high initial molecular weight and/or (2) a high degree of crosslinking in the un-postcured state. It is well established that the modulus of polymeric materials improves with the degree of crosslinking and molecular weight.² But, on observing the tan δ curve for the uncured novolac sample, it was seen that it had a greater area under the curve than most of the other materials (Fig. 3). This indicates greater molecular mobility within the material and means that it cannot be in a highly crosslinked state. Also, the presence of a secondary transition at a lower temperature than T_{σ} means that there are some molecular motions going on even before the glass transition region. Therefore, it can be argued that the most significant reason for the high modulus values of the uncured novolac is its high initial molecular weight. Since it is essentially thermoplastic before the curing stage, it develops its mechanical properties at an earlier stage than does any of the other materials.

The rubber-modified resol sample showed a surprisingly high initial storage modulus for the un-postcured specimen. This was not expected since it was thought that the presence of the rubber phase would give the material more flexibility, resulting in a decrease in the stiffness of the material. The high modulus could be due to the fact that the material had very good initial fiber/matrix interfacial bonding. This is due to the preferential concentration of the rubber molecules at the glass/resin interface, where the rubber phase forms crosslinks with the fibers, leading to stronger bonds at the interface. The high initial storage modulus of the un-postcured copolymer specimen was again due to its comparatively high molecular weight. This argument can be made since the copolymer showed the same behavior as that of the un-postcured novolac with respect to its modulus and tan δ .

Tan δ is a damping term that can be related to the impact resistance of a material.¹⁰ Since the damping peak occurs in the region of the glass transition where the material changes from a rigid to a more plastic state, it is associated with the movement of small groups and chains of molecules within the polymer structure, all of which are initially frozen in. Obviously, the higher the peak tan δ value, the greater the degree of molecular mobility. On comparing the peak tan δ values of the un-postcured specimens in Figure 4, it can be seen that the blend has the highest value, followed by the novolac and the copolymer.

The reason for the high tan δ peak value of the blend is its irregular loose network structure. The blend was prepared by using the resol to cure the



Figure 3 Peak tan δ values of the un-postcured phenolic materials.



Figure 4 Tan δ as a function of temperature for the un-postcured phenolic materials.

novolac instead of using a crosslinking agent such as HMTA. When HMTA is used, the linear novolac prepolymers are tightly crosslinked into a highly developed network.⁷ However, when a resol is used, the *ortho* and *para* hydroxyl groups of the novolac are active with respect to the methylol groups of the resol. Crosslinking at these sites leads to the cure of the blend. The irregular size of the resol prepolymers, mainly dimers and trimers, results in an irregularly crosslinked network compared to the regular novolac. This gives the blend greater flexibility, leading to greater degrees of molecular motion and, hence, better damping characteristics.

The peak tan δ value for the rubber-modified resol was very low and this was unexpected since the reason behind modifying the resol with rubber was to provide more plastic properties to the material, which, in turn, would lead to higher energy dissipation. This fact combined with the high modulus value for the material indicates that the rubber phase is being excessively crosslinked and is producing a rigid interface rather than a ductile one.

Effect of Postcuring on Phenolic Composites

Previous studies have shown that the glass transition temperatures of reinforced phenolic materials can be pushed to higher temperatures by postcuring.^{11,12} In addition, postcuring results in a stabilization of some of the mechanical proper-



Figure 5 Effect of postcuring on the storage modulus of the novolac material.

ties of phenolics. The effect of postcuring on the phenolic composites will be discussed briefly in terms of their storage modulus, $\tan \delta$, and T_{σ} .

As mentioned previously, upon postcuring, phenolic materials continue to crosslink due to the presence of active sites available even after they are cured. Postcuring also results in the formation of stronger interfacial bonds between the fibers and the resin matrix. These result in an increase in their storage modulus values. An increase in crosslinking produces two important effects on the storage modulus of a material. First, the sudden decrease in the storage modulus in the glass transition region is shifted to higher temperatures and, second, the storage modulus decreases at a lower rate through the glass transition and plateaus at a higher level. Figure 5 indicates that there is a marked stabilization of the stiffness properties of phenolic materials (a novolac in this case) over a wide temperature range for postcuring times of up to 24 h.

A quantitative indication of the modulus stabilization with postcuring can be obtained from looking at the difference between the initial and final storage moduli ("difference-in-moduli") of the un-postcured and postcured specimens of the novolac material. In Table I, each "difference-inmoduli" value for a postcured specimen is normalized with respect to the "difference-in-moduli" value of the uncured specimen in order to get an indication of the modulus behavior as the length of postcuring increases. A decrease in the difference between the initial and final storage moduli with increasing postcure times indicates an increase in the degree of crosslinking in the material and, therefore, greater storage modulus stabilization.

The area under the tan δ curve gives an indication of the total amount of energy that can be absorbed by the material during an experiment. A large area under the tan δ curve indicates a great degree of molecular mobility, which translates into better damping properties, meaning that the material can better absorb and dissipate energy. The areas under the tan δ curves for the blend decrease with increase in the length of postcuring as can be seen in Figure 6. This is to be expected since the degree of crosslinking increases with postcuring and, simultaneously, molecular motions become more and more restricted, resulting in a decrease in the amount of energy that can be dissipated throughout the specimen. Also, scanning electron microscopy has shown that there is stronger fiber-matrix interfacial bonding with increased postcuring.⁷ This also restricts the movement of molecular segments.

Figure 7 shows the increase in T_g for the blend and the copolymer materials as the time of postcuring increases. This increase is due to the increase in crosslinking of the materials with post-

Time of Postcure (h)	Initial Storage Modulus (GPa)	Final Storage Modulus (GPa)	Difference-in- moduli (GPa)	Normalized (%)
0	28.4	7.5	20.9	100
4	24.7	12.7	12.0	57.5
8	24.6	14.7	9.9	47.5
16	26.2	17.5	8.8	40.2
24	26.7	18.6	8.1	38.7

Table I Difference-in-Moduli Values of the Novolac Material Before and After Postcure



Figure 6 Effect of postcuring on the tan δ values of the blend material.

curing, which results in the transition region (from a rigid to a more flexible state) being pushed to higher temperatures. Also, the stronger fiber/matrix interfacial bonding produced on postcuring restricts the mobility of molecular chains and segments, resulting in the transition region being pushed to higher limits. Since the dimensional stability of a material is directly related to



Figure 7 Effect of postcuring on the T_g of the blend and copolymer materials.



Figure 8 T_g of the blend as a function of postcure time and temperature.

its T_g , the results seen in Figure 7 indicate that the dimensional tolerance of phenolic materials can be preserved, even at elevated temperatures, by appropriate postcuring.

All the above dynamic mechanical tests were conducted on specimens that were postcured for various lengths of time at a single postcure temperature of 180°C. However, previous studies have shown that the effect of postcuring on phenolics is a function of postcure temperature as well as of time.^{1,11} The principle of time-temperature correspondence or TTS can be used to construct master curves that allow such predictions and has been used by many authors to study the long-term behavior of other polymeric materials.^{6,10,13}

Prediction of T_g

Dynamic mechanical tests were conducted at a fixed frequency of 2 Hz on samples of the resol/ novolac blend that were postcured at five different temperatures and five different times of postcure at each temperature. The T_g 's (assigned to the peaks of the tan δ curves) obtained at each time and temperature of postcure are given in Figure 8. Now, by arbitrarily choosing the curve at a postcure temperature of 150°C as the reference curve, the master curve was constructed by TTS of the data (Fig. 9). The time-temperature correspondence principle states that this master



Figure 9 T_g master curve for the blend material.

curve is identical to the T_g values which would have been obtained at shorter or longer postcure times at a postcure temperature of 150°C.

The amounts shifted by each curve are functions of the respective postcure temperatures and are called shift factors (a_T) . The actual times of postcure were multiplied by the respective shift factors for each postcure temperature in order to obtain the master curve for a postcure temperature of 150°C. A plot of $\ln a_T$ versus the reciprocal of the postcure temperatures was found to be well modeled by the Arrhenius relationship (Fig. 10). The activation energy was calculated to be 68.25 kJ/mol and compares well with values reported in the literature.¹¹ Use of this master curve along with the relationship between the shift factors and postcure temperature allows prediction of the T_{σ} at any time and temperature of postcure. This would make it possible to set a postcure protocol for a particular phenolic material depending on the T_g required for the application.

Frequency Dependence of Phenolics

All the previous tests on the different un-postcured and postcured samples were conducted in a fixed frequency mode (2 Hz), and their behavior was studied as a function of temperature only. However, the mechanical behavior of viscoelastic materials subjected to dynamic mechanical testing is dependent on time (or frequency) as well as



Figure 10 Variations of the shift factors with postcure temperatures fitted to the Arrhenius model.

on temperature. By operating the DMA in a multifrequency isothermal hold mode, the time-dependent aspects of the material properties were studied. In this mode, the sample was allowed to equilibrate at different temperatures and then subjected to a series of frequencies ranging between 0.01 and 100 Hz. The resulting behavior of the material was recorded as a function of frequency (or time scale). Figures 11–13 show the effect of frequency on the storage modulus of the resol/novolac blend, graft copolymer, and pure resol (S-glass reinforced) materials at different temperatures in the region of their glass transitions. It can be clearly seen that the storage moduli of all three materials increase with increasing frequency. Also, the decrease in the storage moduli of the blend and copolymer materials as a function of frequency is more significant as the temperature exceeds their respective T_g 's.

Using the multifrequency data at the different temperatures, TTS was used to construct master curves that allow long-term predictions of the storage modulus of the three materials (Fig. 14). The T_g of each material was chosen as the reference temperature during construction of the master curves. The master curves allow prediction of the storage modulus over 15 decades of frequency for the materials, whereas the experimental data covered only four decades.

The projection of the storage modulus as a function of frequency (or time) is dependent on



Figure 11 Storage modulus as a function of frequency and temperature for the blend.

the temperature and frequency ranges used in the experiment, and with the use of more temperature steps, the master curve can be extended further in time. The WLF model was used to fit the variations of the shift factors with temperature, since the T_g was used as the reference curve. Use of the master curve and the variations of the shift factors with temperature allows prediction of the



Figure 12 Storage modulus as a function of frequency and temperature for the copolymer.



Figure 13 Storage modulus as a function of frequency and temperature for the resol (S).

storage modulus at frequencies and temperatures that are not experimentally accessible.

CONCLUSIONS

DMA was found to be a very useful technique for interpreting some of the thermomechanical be-



Figure 14 Storage modulus master curves for the blend, copolymer, and resol (S).

havior of phenolic composites based on their viscoelasticity. Three useful quantities obtained by dynamic testing, namely, storage modulus, tan δ , and T_g , were used to interpret the behavior and performance of the materials.

On comparing the initial storage moduli of the un-postcured specimens, the novolac had the highest value followed by the copolymer, rubber-modified resol, and blend, respectively. The high modulus values of the un-postcured novolac and copolymer material were due to their higher initial molecular weights. The surprisingly high modulus value of the rubber-modified resol was due to strong initial fiber/matrix interfacial bonding.

On comparing the peak tan δ values, the blend had the highest value followed by the copolymer and novolac. The tan δ value of the rubber-modified resol was the lowest of all the materials and this was probably due to the excessive crosslinking of the rubber phase, resulting in a rigid interface that does not allow high-energy dissipation.

It was seen that the stiffness and dimensional tolerance of phenolics could be increased by postcuring, with a simultaneous decrease in the damping capacity of the material. The exact time and temperature at which the materials should be postcured will have to be a compromise among these three important parameters and will depend on the application of the finished product and the environment to which it will be exposed. The increase in modulus with postcuring is due to the increase in crosslinking of the polymer resin and the formation of stronger fiber/matrix interfacial bonds.

The peak tan δ values and the area under the tan δ curves for the blend material were found to decrease with the time of postcuring. This was expected since molecular motions become more restricted because of crosslinking and formation of stronger interfacial bonds, resulting in a decrease in the energy that can be dissipated throughout the sample.

The T_g 's of the blend and copolymer were found to increase with increase in postcure time. Since the effect of postcuring on phenolics is a function of the postcure temperature as well as time, the principle of time-temperature correspondence was used to obtain a T_g master curve for the blend. Use of the master curve and the relationship of the shift factors with temperature, fitted to the Arrhenius model, allowed prediction of the T_g of the material over a broad range of times and temperatures of postcure.

The effect of frequency on the storage modulus of the blend, copolymer, and resol (S) materials was studied. TTS was also used to predict the retention of their storage modulus over several decades of frequency. The shifting of the curves during the storage modulus master curve construction was modeled successfully using the WLF model.

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