



# Prepreg age monitoring via differential scanning calorimetry

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**Abstract:** Fabrication of composite parts from prepregs often requires layup and preparation times of days and even weeks, during which prepregs undergo room-temperature aging. The aging process can compromise compaction, tack, and overall quality of composite parts, and thus a need exists for an accurate and convenient method to monitor the extent of prepreg aging as a function of out-time. Here, we report a method to monitor prepreg age, which involves measurement of changes in glass transition temperature as a function of room-temperature aging time. Samples from three out-of-autoclave prepreg systems were aged in ambient conditions and tested periodically using modulated differential scanning calorimetry. A linear increase in glass transition temperature with prepreg age was noted. Results are discussed in the context of monitoring the chemical aging of epoxy resins that occurs at ambient temperature.

Key words: out time, ambient aging, differential scanning calorimetry, prepreg, polymer matrix composites

## 1. Introduction

A new generation of composite prepregs has been introduced for manufacturing out-of-autoclave. These vacuum-bag-only cured materials are attractive for use in aerospace applications, as vacuum bag curing eliminates the size constraints associated with autoclave processing.<sup>1,2</sup> Large parts,

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however, require long times for layup and placement of prepreg. Consequently, if vacuum-bag-only prepregs are to be used for the production of large composite parts, it will be essential to understand and monitor the chemical aging characteristics of out-of-autoclave epoxy resin systems.

The layup of prepreg is performed at ambient temperature. The epoxy resin in composite prepregs, however, undergoes aging at ambient temperature.<sup>3-11</sup> For this reason, prepregs are stored frozen to prevent advancement of cure, then thawed prior to layup.<sup>3-9</sup> Despite this precaution, ambient aging is inevitable for the production of large parts, as the lay-up of large-scale structures can take several days and even weeks, a period known as “out-time”.<sup>3-6</sup> Additionally, the chemical age of a given roll of prepreg is often unknown. With the push in the aerospace industry to manufacture large components such as airplane wings and fuselages with out-of-autoclave prepregs, an understanding of the out-life limits of these materials will be critical. Also of importance from a manufacturing standpoint is the development of a test method for monitoring prepreg age that is not only accurate but simple to carry out and suitable for production use.<sup>3,4,9</sup>

The degree of cure of epoxy resin in prepregs advances with room temperature aging time. The chemical changes that occur during ambient aging influence the mechanical properties of cured parts, as well as the practical attributes of prepregs, such as tack and drape.<sup>3,9-14</sup> The ability to assess prepreg age is important for the manufacturing of high-quality parts. Studies on ambient aging of traditional autoclave prepregs have been performed for a variety of resin systems.<sup>3-5,7,8,10-12,14-17</sup> These studies examined multiple methods for monitoring prepreg age, with the most success coming from infrared (IR) spectroscopy<sup>10,12</sup> and high-performance liquid chromatography (HPLC) techniques.<sup>15</sup> A more recent study demonstrated the use of photoacoustic spectroscopy to track prepreg age.<sup>3</sup> In this study, we examine a method for monitoring prepreg out-time via differential



scanning calorimetry (DSC) methods and verify the validity of this method for out-of-autoclave prepreg systems.

In the past studies of ambient aging, changes in IR spectra and in HPLC results were attributed to chemical aging effects. In the present study, these same effects resulted in a linear increase in the glass transition temperature ( $T_g$ ) of the B-stage epoxy. Results from the past aging studies, as well as the general glass transition theory, provide insight into the mechanism behind this relationship. The findings are of interest because  $T_g$  is readily and conveniently measured using conventional DSC. The results of this study, combined with past literature, suggest that DSC test methods could be implemented on the factory floor to monitor the age of a wide range of epoxy prepreg systems.

## 2. Experimental

### 2.1 Materials

Three carbon fiber/epoxy prepreps were selected for this study, all formulated for vacuum-bag-only processing. The first (Prepreg 1) was a five harness satin (5HS) carbon fiber fabric with a toughened epoxy resin (CYCOM 5320, Cytec Engineered Materials, USA). The second (Prepreg 2) was an eight harness satin fabric (8HS) with a toughened epoxy resin formulated for long out-life (CYCOM 5320-1, Cytec Engineered Materials, USA). The third prepreg system, from a different manufacturer, was included in the study to assess chemical aging behavior for a prepreg formulation from a different source. This prepreg system (Prepreg 3) was composed of a 2 x 2 twill carbon fiber fabric and a toughened epoxy matrix (MTM44- 1/CF5804A, Advanced Composites Group, UK). The nominal resin content for all three prepreps was 35–40% by weight. The manufacturers' stated out-life for the three materials was 21, 30, and 21 days, respectively. The specific formulations of the epoxy resins examined in this study were proprietary.



## 2.2 DSC

Modulated DSC (MDSC) was used to track changes in the resin systems as a function of room-temperature aging time. Material was stored in unsealed plastic bags in ambient laboratory conditions ( $20\pm 2$  °C,  $50\pm 5\%$  relative humidity) and tested periodically by MDSC as aging progressed. DSC is used to measure the difference in the amount of heat required to change the temperature of a pan containing a material sample and an empty reference pan as a function of temperature. In modulated DSC, a sinusoidal temperature modulation is applied over the standard linear temperature ramp. This temperature modulation allows for the splitting of the total heat flow into reversing and non-reversing components. Reversing heat flow is a function of the heat capacity of the sample as well as the rate of temperature change, while non-reversing heat flow is a kinetic component dependent only on time and absolute temperature (not influenced by a sinusoidal temperature modulation).<sup>18,19</sup> Glass transition temperature is a reversing (heating rate dependent) event and was thus calculated from examination of the reversing heat flow signal.<sup>19</sup> In contrast, exothermic cure reactions are kinetic events, and thus degree of cure was determined by examination of the non-reversing heat flow signal. In this study, temperature modulation was used during DSC experiments to increase the accuracy of measurements. However, standard DSC methods (without temperature modulation) can also be used for the same purpose.

For prepreg characterization, MDSC tests were performed periodically as aging progressed. A DSC with temperature modulation capability and sub-ambient cooling was used (TA Instruments Q2000). A temperature ramp from  $-90$  to  $280$  °C was performed on each sample. A temperature modulation of  $\pm 1$  °C every 60 s was applied with a ramp rate of  $3$  °C/min. All tests were performed under a nitrogen purge.



Note that in this study, glass transition measurements were performed on prepreg samples that were not fully cured. The low-temperature  $T_g$  values measured in this study are characteristic of the B-stage resin, while the glass transition of the cured systems are much higher ( $\approx 200$  °C). To avoid confusion with the  $T_g$  of the cured resin, the glass transition temperatures reported in this study will be referred to as B-stage  $T_g$ .

### 3. Results and discussion

Past aging studies have shown that the degree of cure of thermosetting epoxy systems increases as a function of room temperature aging time.<sup>10,12,15</sup> Additionally, based on glass transition theory,  $T_g$  increases linearly with degree of cure.<sup>20</sup> This suggests that the B-stage  $T_g$  should depend linearly on ambient aging time. This trend has been validated for one autoclave resin system,<sup>7</sup> but proof of applicability to a range of resin formulations and an analysis of the accuracy of the trend as a predictor for prepreg age has not yet been demonstrated or reported.

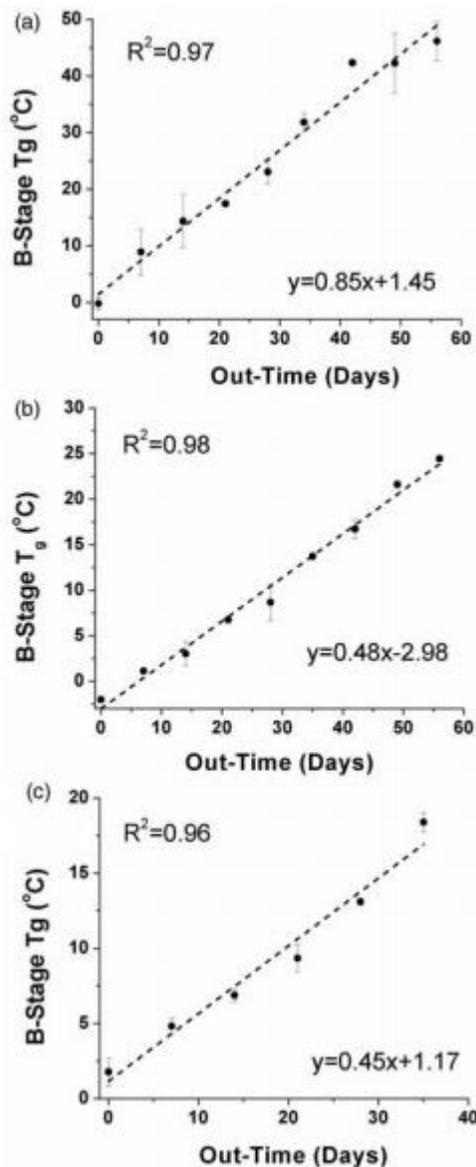
We have performed MDSC testing on three out-of-autoclave prepregs. To establish the linear trend in B-stage  $T_g$  as a function of out-time, two sets of samples taken from different regions of the prepreg roll were tested for each material system. Data were plotted and linear fits were applied. Results are presented in Figure 1, with linear fit equations and calculated correlation coefficients. One objective of this work was to investigate the reaction kinetics governing ambient aging and to develop a test method for monitoring prepreg age. To determine the accuracy of predicting age using glass transition temperature, a third set of samples was tested. This sample set, from still another area of the prepreg roll, was used as blind data to establish the accuracy of the linear fit. Using the B-stage glass transition temperatures obtained for each sample, a predicted prepreg age was calculated using the linear fit lines displayed in Figure 1. Predicted age was plotted against actual

known age for all three sample sets. Figure 2 displays predicted age for the prepregs, calculated

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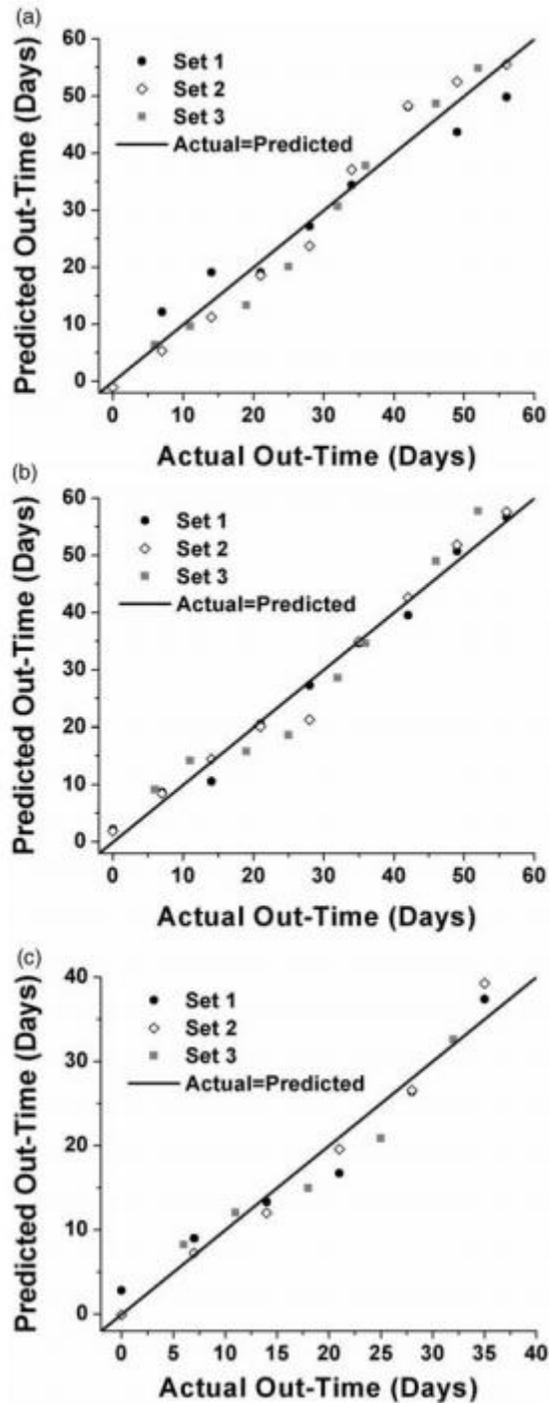


using measured B-stage T<sub>g</sub> values and the linear fit equations (shown in Figure 1), plotted against actual prepreg age. The solid lines in each plot are reference lines for which the actual and predicted age values are equal. The accuracy of using glass transition temperature as a measure of prepreg age was evaluated by examining the difference between actual age and age predicted using MDSC results. Table 1 displays the minimum, maximum, and average difference (in units of days) for all three prepreg systems. The method was accurate, on average, to within 3 days (Table 1).





**Figure 1:** Linear trends between B-stage Tg and prepreg out-time for (a) Prepreg 1, (b) Prepreg 2, and (c) Prepreg 3.



**Figure 2:** Age predicted using linear fit equations plotted against actual known age for (a) Prepreg 1, (b) Prepreg 2, and (c) Prepreg 3.

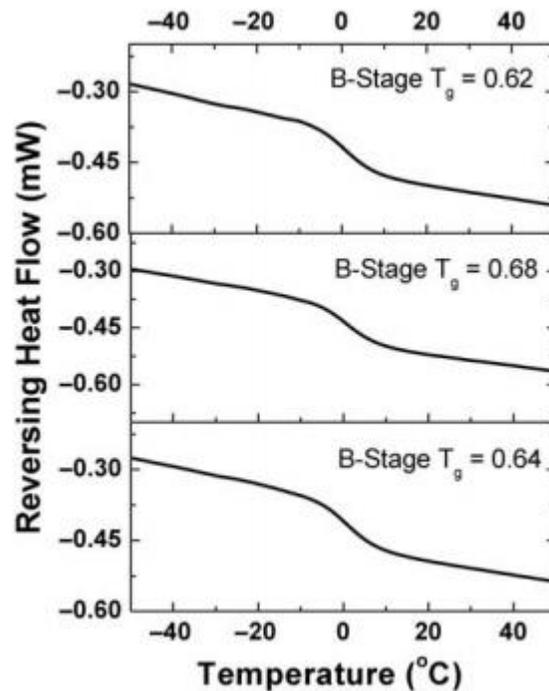


**Table 1:** Minimum, maximum, and average difference in predicted age and actual age (in units of days) for the three prepreg systems examined.

	Min	Max	Average
Prepreg 1	0.36	6.26	3.08
Prepreg 2	0.04	6.71	2.33
Prepreg 3	0.11	4.28	1.87

### 3.1 Validity of the method

To verify the precision of the experimental testing and to evaluate the error inherent in the MDSC test method, a validity test was performed. Three samples were prepared from adjacent regions of a single prepreg ply (Prepreg 1). One sample was tested each day for 3 consecutive days. All samples were stored frozen until tested. Because samples were taken from the same region of the prepreg roll, the resin content in each sample was assumed to be constant. Additionally, because the samples were stored frozen, the resin was not exposed to environmental factors such as moisture and temperature variations. The results of the validation experiment, presented in Figure 3, demonstrate that the error inherent in the test method is negligible. The standard deviation in glass transition temperatures measured was 0.03 °C. Thus, the scatter in the measurements reported in Figure 1 is assumed to arise from differences in temperature and humidity exposure during aging, and variations in resin content and specific thermal history in different regions of the prepreg roll. No variance is attributed to experimental test errors.



**Figure 3:** Reversing heat flow signals, showing B-stage glass transition temperature results for three samples of fresh prepreg (Prepreg 1), used to validate the accuracy of the modulated differential scanning calorimetry (MDSC) method.

### 3.2 Mechanism of ambient aging

A clear linear dependence of B-stage  $T_g$  on ambient aging time was observed for all three resin systems examined. This linear dependence is attributed to chemical reactions occurring in the resin during ambient aging. During room-temperature chemical aging, the number of unreacted epoxide groups in the resin decreases. These reactions are similar in nature to those reported in previous studies using IR spectroscopy<sup>10,12</sup> and HPLC15 and cause the gradual increase in B-stage glass transition temperature reported here. Though we have examined out-of-autoclave resin systems in this work, the general aspects of epoxy aging are common to all prepreg systems. In previous ambient aging studies, IR spectroscopy results were used to define an “epoxide index,” which was tracked as a function of age. The epoxide index, a measure of the number of unreacted epoxide groups, was calculated as the ratio of the area of the aromatic ring band of the IR spectra to that of



the epoxide band.<sup>10,12</sup> The reduction in epoxide index was reported to be a linear function of prepreg age.<sup>10</sup> A reduction in the primary epoxy component of prepreg resin during ambient aging has also been reported in HPLC results.<sup>15</sup> These studies showed that a reduction in free amine molecules (as measured by reverse phase liquid chromatography) occurs during the aging process.<sup>10</sup> The reduction in unreacted epoxide groups, resulting from an epoxy-amine reaction, is known as precuring. This precuring reaction shifts the glass transition temperature of the uncured system to a higher temperature. Glass transition marks a change in the resin system from a glassy state to a rubbery state. As the number of unreacted end groups is reduced during precuring, segment mobility decreases, leading to an increase in glass transition temperature. Such a phenomenon, though never examined as a means of monitoring ambient aging, has been discussed in terms of the general properties of the glass transition temperature of thermosetting resins.<sup>20</sup> The classical theory of glass transition in cross-linking polymers is complicated in the temperature region beyond the gel point of the resin, where molecular weight becomes undefined.<sup>21</sup> For the B-stage glass transition temperature examined in this study, however, the resin is well below the gel point, and has a defined molecular weight. A simplified theory of glass transition temperature can thus be employed to describe the behavior observed. As reported in past IR studies of ambient aging, the concentration of unreacted epoxide groups decreases linearly with aging time.<sup>10</sup> This process can also be discussed in terms of the degree of cure (DoC) of the resin. In reference to an ideal backbone glass transition temperature ( $T_{1g}$ ), glass transition as a function of degree-of-cure can be expressed as<sup>20</sup>

$$T_g = T_g^\infty - K_1(1 - \rho) \quad (1)$$

where  $\rho$  is the DoC of the system (or end group conversion) and  $K_1$  is a constant for a given resin system. Here, DoC is defined as the ratio of the number of reacted sites to the total number of reactive sites available. The equation shows that a linear increase in glass transition temperature with end



group concentration is expected at low conversions (small  $p$ ).<sup>20</sup> This simple relationship holds in the low-conversion region because the effect of cross-linking (the linking of polymer chains) is negligible. The observed change in glass transition temperature  $\Delta T_g$  is simply a function of the consumption of end groups (the reduction in the number of unreacted epoxide groups in the resin) that occurs during the epoxy-amine reaction as the cure progresses.

Thermoset resins such as the ones examined in this study are synthesized by condensation reactions. The resins begin as low molecular weight oligomers, with a high initial concentration of end groups.<sup>20</sup> These end groups are consumed during the cure process, which influences the glass transition temperature of the material. Past investigations of the effect of DoC on glass transition temperature for thermoset resins have reported trends similar to the behavior predicted in equation (1).<sup>20</sup> Characterization of these materials through a broad temperature range shows a linear relationship between glass transition temperature and degree-of-cure up to a conversion of approximately 0.30.4 (DoC of 30-40%).<sup>20</sup> The relationship becomes non-linear only when branching and cross-linking reactions become dominant (far beyond the useful out-life of prepreg materials). These results are consistent with the trend observed here for B-stage  $T_g$  as a function of aging time. Because the number of unreacted epoxy groups decreases in proportion to aging time, and because the general theory predicts that  $T_g$  will increase in proportion to DoC,  $T_g$  is also expected to increase linearly with ambient aging time. This trend, reported for a single resin system formulated for autoclave curing,<sup>7</sup> is shown here to hold for a variety of out-of autoclave prepreps as well.

Although the B-stage  $T_g$  exhibited a linear dependence on aging time for the range of out-times considered here, deviation from linearity is expected to occur at longer aging times, as predicted by equation (1). In fact, the dependence of  $T_g$  on out-time should become non-linear when the B-stage  $T_g$  exceeds the cure temperature (here  $\approx 22^\circ\text{C}$ ), because vitrification of a polymer network occurs



when the glass transition temperature reaches the cure temperature.<sup>22</sup> Of the prepreg systems studied here, only Prepreg 1 reached T<sub>g</sub> values significantly greater than ambient temperature through the length of this study. Despite the fact that T<sub>g</sub> values exceeded 22°C for Prepreg 1, no significant deviation from linearity was observed. Similar behavior has been reported for an autoclave prepreg system, where measurements of B-stage T<sub>g</sub> as a function of out-time, showed little deviation from linearity until T<sub>g</sub> reached 45°C at an exposure time of 100 days.<sup>7</sup> For greatest accuracy in predictions of prepreg age, the linear relationship between B-stage T<sub>g</sub> and ambient aging time should be defined for B-stage T<sub>g</sub> values less than 22°C. This is a reasonable limit to impose for practical purposes, because the B-stage T<sub>g</sub> of an epoxy system will not exceed the cure temperature (vitrify) within the useful out-life of the material.

While the chemical aging process described in this work is well understood, a means of implementing this understanding to monitor prepreg age in-service is new. Past studies have shown IR techniques and chromatography methods can be used to monitor prepreg age. However, these methods are not suitable for production floor use. IR measurements require dissolving resin from the prepreg with acetone to produce resin films, a labor-intensive sample preparation task.<sup>3,10</sup> Other proposed age-monitoring methods (IR, HPLC, and photoacoustic spectroscopy) require post-processing and analysis that is time-consuming and skill intensive. DSC methods, on the other hand, require minimal sample preparation, simple analysis/interpretation of data, and can be applied to a wide variety of sample types.<sup>18</sup> Only small samples of prepreg (6-mm disks) are needed for DSC methods and are easily cut and no separation of resin from the carbon fibers is required. Lastly, data analysis is straightforward, and due to the linear nature of the trend observed in glass transition temperature, complicated statistical methods are not necessary. These factors highlight the potential



for DSC testing to provide a quality control method for monitoring prepreg age suitable for production floor use.

### 3.3 Degree of cure

As described in the preceding discussion, the degree of cure of the epoxy in the prepreg systems is expected to increase as a function of ambient aging time. To support this supposition, DoC was calculated and plotted as a function of both aging time and B-Stage Tg.

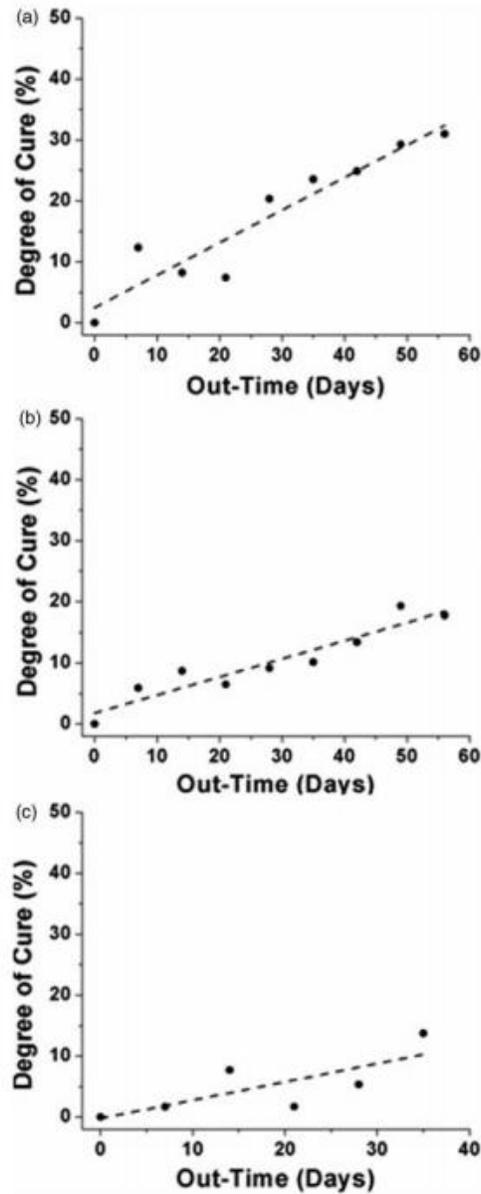
The degree of cure was calculated using the following equation:

$$\%DoC = 100 \times \frac{\# \text{ reacted sites}}{\text{total } \# \text{ reactive sites}} \quad (2)$$

Here, values for the number of reacted sites and total number of reactive sites were determined by integration of exothermic cure reaction peaks. Calculating the area of the exothermic cure reaction peak for unaged prepreg yielded the heat of reaction corresponding to full cure (DoC of 100%). As the cure progressed, the number of unreacted sites decreased, lowering the heat of reaction required to complete the cure. In other words, the exothermic cure peak decreased in intensity as a function of aging time. Degree of cure was thus determined by comparing the heat of reaction of the aged material ( $H_{r,aged}$ ) to that of the unaged sample ( $H_{r,total}$ ). This calculation is shown in equation (3).

$$\%DoC = 100 \times \frac{H_{r,total}(J/g) - H_{r,aged}(J/g)}{H_{r,total}(J/g)} \quad (3)$$

Results for percent DoC as a function of aging time and B-stage Tg are presented in Figures 4 and 5.



**Figure 4:** Degree of cure as a function of ambient aging time for (a) Prepreg 1, (b) Prepreg 2, and (c) Prepreg 3.

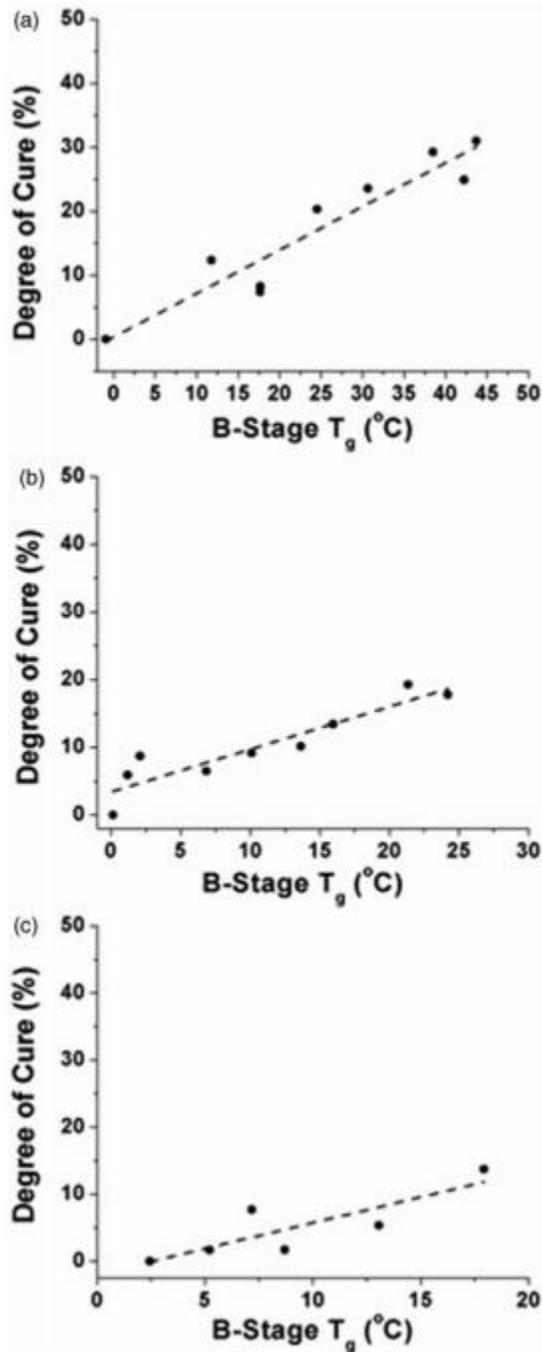


Figure 5: Degree of cure as a function of B-stage  $T_g$  for (a) Prepreg 1, (b) Prepreg 2, and (c) Prepreg 3.

## 4. Conclusions



In this study, we considered measurements of B-stage glass transition temperature as a means of tracking out-time and monitoring prepreg age. Measured values of B-stage T<sub>g</sub> showed a linear dependence on prepreg out-time for three out-of-autoclave prepreg systems from different manufacturers. This linear dependence resulted from the precuring reaction that occurs during room-temperature aging of epoxies. Analysis of degree of cure corroborated this mechanism of ambient aging. Comparisons with other aging studies, as well as consideration of general glass transition theory, supported the conclusion that precuring of epoxy prepregs causes a linear increase in B-stage T<sub>g</sub> with ambient aging time.

The T<sub>g</sub> of uncured epoxy at low temperatures was measured using MDSC. The test method was precise, and relatively quick and simple, introducing minimal error into the results. Because of these attributes, measurement of T<sub>g</sub> by MDSC affords a convenient means for monitoring prepreg age. The method also is appealing because it involves virtually no sample preparation, requires only small sample sizes, and the data analysis is trivial. For these reasons, the method is well suited to a production floor setting, where it can be readily employed to accurately determine the remaining useful life of prepreg materials.

The need for a method for accurately determining prepreg chemical age is growing, particularly as manufacturers undertake fabrication of larger and more complex parts that require longer preparation times. Composite parts fabricated with prepreg that has exceeded the recommended out-life limits exhibit defects and degraded mechanical properties that are often unacceptable. Implementing a simple method to determine prepreg age would result in more efficient production of high-quality parts.

Execution of the method presented here requires generating an initial calibration curve governing the chemical aging kinetics of the particular prepreg. The linear fit equations will be specific to each



prepreg system because of differences in resin formulations. For example, although the specified out-lives for Prepregs 1 and 3 were the same, the more rapid increase in DoC for Prepreg 1 (and the higher values of B-stage T<sub>g</sub> measured) indicates that room-temperature chemical aging proceeds more rapidly in this material system. Prepreg 2, which was formulated for extended out-life, and Prepreg 3, show similar dependences of B-stage T<sub>g</sub> values on aging time. Thus, chemical aging occurs more gradually in these systems, indicating a longer useful out-life. Calibration curves are therefore required to characterize each material system, although these calibrations can be performed in a few weeks and require only grams of material.

The method described here could be deployed on the production floor in straightforward fashion. An allowable limit for ambient aging time is first established by conventional means, such as mechanical testing of cured laminates, or simply by following the manufacturer's recommended out-life for a given material. By either method, manufacturers set a B-stage T<sub>g</sub> limit over which prepreg will be discarded. Prepreg age is then determined by measuring the B-stage T<sub>g</sub> and comparing to a pre-measured calibration curve specific to the system used.

Note that the allowable out-time will depend on storage conditions, including temperature and humidity. Similarly, the variation in age prediction reported here (Table 1) is assumed to arise due to variations in temperature and humidity during aging. Prepreg stored in a controlled environment, such as a clean room with controlled humidity and temperature, would display less variability, allowing for increased accuracy in age prediction. Nevertheless, the results reported here for out-of-autoclave prepreps, combined with past results for an autoclave curing system,<sup>7</sup> indicate that the technique will be applicable to a wide range of composite prepreps.

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