TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Gelation of Epoxy - Glass Prepreg by Parallel Plate Rheometry

Number TA-126

Summary

Epoxy - glass prepregs are widely used in the electronic and aerospace industries in the fabrication of laminate products ranging from printed circuit boards to helicopter rotors. The properties of these products are a function of resin formula, resin/substrate ratios, degree of cure and processing conditions. One of the most important factors affecting the processing of prepreg materials is gel time. Gel time is the time from when the material begins to soften until gelation occurs, where gelation is the irreversible transformation from a viscous liquid to an "elastic gel" (1).

Thermomechanical Analysis (TMA), specifically parallel plate rheometry mode, provides a rapid, sensitive, reproducible means for monitoring gel time.

Introduction

Gel time is a critical processing time, since after the gel point the material is no longer able to flow and is therefore unprocessable. Traditional methods for the measurement of gel time are of questionable reliability because they tend to be very operator dependent. For example, in the electronics (printed circuit board) industry, a prepreg is evaluated by grinding it to a fine powder, passing the powder through a sieve to remove the glass reinforcement particles, placing the filtered resin powder on a preheated plate, and then stirring with a glass rod until the resin adheres to the stirring rod. The time from initial softening until adhesion occurs as monitored by a stop watch is considered to be the gel time.

Thermomechanical Analysis (TMA), which is a thermal analysis technique that measures material dimensional changes with temperature, provides a convenient and more reproducible, scientific approach to gel time measurement. Using a specific TMA probe configuration (parallel plate rheometer), TMA measured dimensional changes can be converted to gel time and viscosity values.

Experimental

Figure 1 shows the parallel plate rheometer configuration for TMA. The sample in disk form is placed between two stainless steel plates to form a "sandwich-type" arrangement which is kept together by a coaxial alignment cage. This whole arrangement is subsequently placed on the TMA quartz stage and a macroexpansion probe brought in contact with the top plate. As the sample flows due to applied force, or more typically increased temperature (or time at an isothermal temperature), probe displacement is measured. Although not used here because gel time is the parameter of interest, an equation is available which converts dimensional change into viscosity values. (2)

Conditions in the TMA (e.g. atmosphere, temperature, heating rate) can be varied to simulate actual process conditions. In this study, a circular wafer of appropriate size and thickness (the PPR accommodates samples 9.5mm in diameter and 0.25 - 1.00mm thick) was punched from a glass-epoxy prepreg sheet. After mounting in the TMA with 10 gram load on the probe, the sample was heated at 10°C/minute from ambient to 150°C under a 100 ml/minute nitrogen purge.

Results

Figure 2 demonstrates the dimensional change occurring as a result of the gelation process. The curve shows that as the prepreg is heated from room temperature, a slight expansion occurs. After approximately six minutes, the curve starts to drop, indicating softening of the resin. This continues until approximately eight minutes when the curve begins to flatten due to the cross-linking process of gelation.

The gel time of the prepreg is observed as the difference between time to the onset of gelation minus the time to the onset of softening (Figure 3). The total displacement during cure, 147.26 µm, in conjunction with a knowledge of the initial sample thickness, can be used to calculate the percent displacement during flow. Such data is useful for optimization of resin bleed versus void formation during laminate processing, as in a "scaled flow" test.

Additional information on resin performance can be gained by studying the derivative curve, or the rate of dimension change. The height of the derivative curve will vary in intensity with flow of the resin. The peak temperature, equivalent to the inflection temperature shown in Figure 4, represents the point of maximum flow rate. A close study of these parameters will thus yield further valuable information on the characteristics of prepreg, and lead to a more accurate definition of the analysis limits for the calculation of gel time.

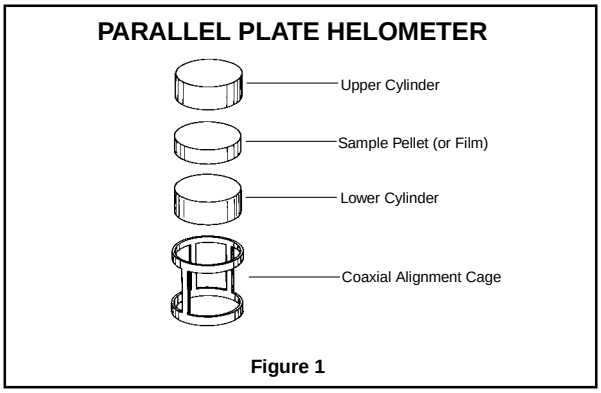

Figure 1

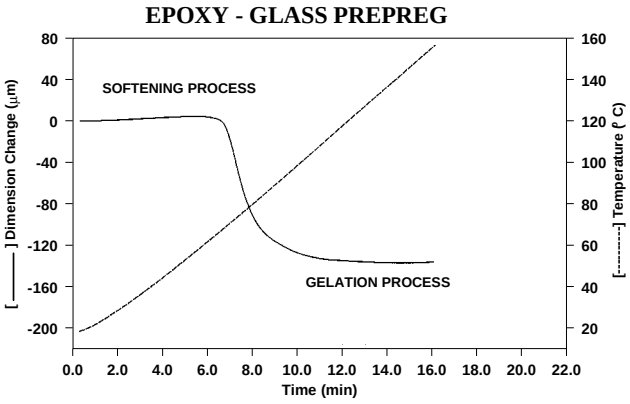

Figure 2

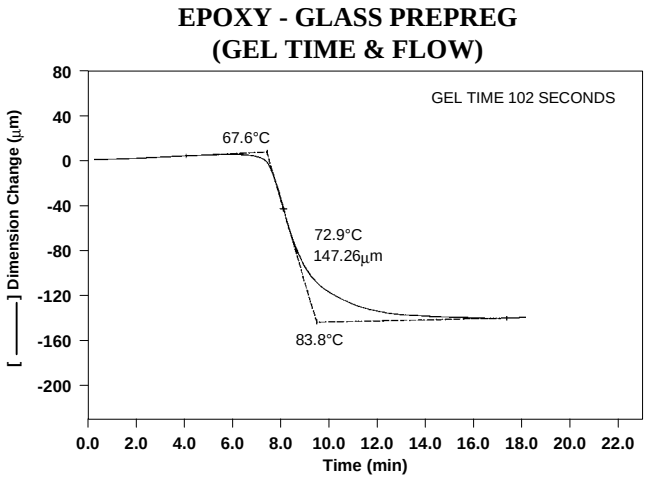

Figure 3

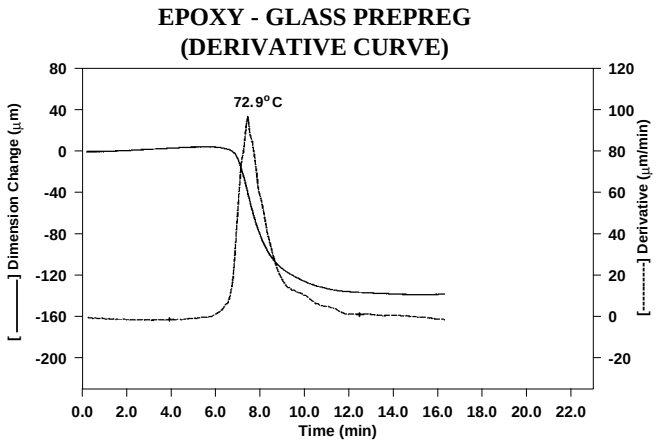

Figure 4

References

1. E. A. Turi, Thermal Characterization of Polymeric Materials, Academic Press, 1981, Orlando, FL

2. TA Instruments TMA 2940 Operators Manual.

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