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STRUCTURE-PROPERTY RELATIONSHIPS IN POLYMERIC MATRIX
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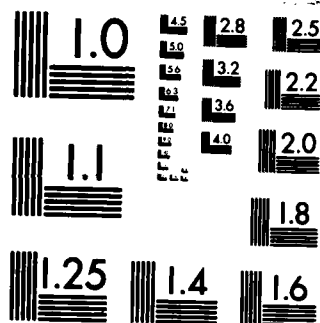
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Structure-Property Relationships
in Polymeric Matrix Materials

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The main results of the research program are summarized in terms of: I) development of a time-temperature-transformation (TTT) cure diagram for thermosetting systems; II) automation of a torsion pendulum using digital computers; III) characterization of novel polymers, composites and transitions, and IV) development and application of a pyrolysis system for characterizing the thermal degradation mechanisms of polymers. Originator supplied Keywords include:		

ACCOMPLISHMENTS

I. The principal accomplishment is the evolution of a generalized time-temperature-transformation (TTT) cure diagram for thermosetting systems which permits an understanding of the cure behavior, properties, and degradation of thermosetting systems.

Introduction

Research in this laboratory has shown that a time-temperature-transformation diagram (analogous to the TTT diagrams that have been employed for many years in metallurgical processing) may be used to provide an intellectual framework for understanding and comparing the cure and physical properties of thermosetting systems (1,12,15,18,22,25,26). Isothermal TTT diagrams such as that shown in Figure 1 can be obtained by measuring the times to events which occur during isothermal cure versus temperature (T_{cure}). The events include phase-separation (for example, in rubber-modified systems -- not shown in Figure 1), gelation, vitrification, and devitrification. Gelation is synonymous with the incipient formation of an infinite network. Vitrification occurs when the glass transition rises through the isothermal temperature of cure. Devitrification occurs when the glass transition temperature decreases through the isothermal temperature. The diagram displays the distinct states encountered due to chemical reactions. These states include liquid, gelled rubber, ungelled glass, gelled glass, and char. The diagram also displays the critical temperature T_{go} , $\text{gel}T_g$, and T_{go} which are, respectively, the glass transition temperature of the fully cured system, the temperature at which the times to gelation and to vitrification are the same, and the glass transition temperature of the unreacted reactants.

The TTT diagram shown in Figure 1 can be obtained conveniently using a technique (TBA) in which a resin-impregnated glass braid is the specimen in a torsion pendulum (1,6,12,15,23,24,25). Use of the composite specimen permits monitoring of the changes from liquid to rubber to glass, and to devitrification, which can occur on cure. Relationships between cure and properties (e.g., transitions) can be obtained from temperature scans of the cured specimens.

Discussion

Much of the behavior of thermosetting materials can be understood immediately in terms of the TTT diagram (Figure 1) through the influence of the gelation, vitrification, and devitrification events on properties: gelation limits macroscopic flow, and limits growth of a dispersed phase (as in rubber-modified systems); vitrification limits chemical conversion; devitrification marks the limit in time due to degradation for the material to support a load. Thus (in listed form):

- a) The ungelled glassy state is the basis of commercial molding and "prepreg" materials (21) since, on heating, the ungelled material will flow before gelling.
- b) Temperature $\text{gel}T_g$ is a critical temperature in determining the upper



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temperature for storing reactive materials to avoid gelation.

c) The morphology developed in a two-phase system (e.g., those in which rubber-rich domains precipitate as a dispersed phase) on isothermal cure depends on the gelation temperature. The reaction temperature determines the competition between thermodynamics and kinetic (transport) factors. For optimum mechanical properties, a two-phase system will be cured at one temperature to control the morphology, and subsequently cured above $T_{g\infty}$ to complete the reactions of the matrix (19,20).

d) Shrinkage stresses due to cure begin to develop at gelation. There is a need to develop polymerization systems involving low shrinkage on cure.

e) Isothermal cure at temperature T_{cure} below $T_{g\infty}$ will (in principle) lead to $T_g = T_{cure}$. There is a need to develop mechanisms of cure which yield $T_g \gg T_{cure}$.

f) Complete cure is obtained (in principle) only by curing above $T_{g\infty}$. Correlations between macroscopic behavior and molecular structure will then result only after curing above $T_{g\infty}$.

g) For high $T_{g\infty}$ systems there is a competition between cure and degradation.

h) The limiting viscosity in the fluid state is controlled by gelation above temperature $g_{el}T_g$, and by vitrification below temperature $g_{el}T_g$. At gelation the weight average molecular weight and zero shear rate viscosity become infinite. Viscosity in the vicinity of vitrification is described by the Williams-Landel-Ferry equation.

i) Isothermal reactions proceed slowly after vitrification. Below temperature $g_{el}T_g$ this leads eventually to gelation. At high temperatures this leads eventually to degradation. Degradation can result in devitrification as the glass transition temperature decreases through the isothermal temperature due to decrease in crosslinking, or formation of low molecular weight plasticizing material. Degradation can also result in re-vitrification (e.g., char formation) as the glass transition rises through the isothermal temperature due to increase in crosslinking or volatilization of low molecular weight plasticizing material.

j) The times to gelation and to vitrification each can be computed from the reaction mechanism (or the reaction rate dependence on the concentrations of the reactants) and the extent of reaction at gelation (which is constant according to Flory's theory of gelation) and the extent of reaction at vitrification, respectively. Since vitrification occurs when the glass transition temperature rises to the temperature of cure, computation of the time to vitrify involves knowledge of the relationship between T_g and conversion.

The following is an outline for computation of the times to gelation and to vitrification. The overall reaction rate is a function of the temperature (T), extent of reaction (X), reaction mechanism $f(X)$, and local microviscosity (η_L) which in turn is a function of the molecular weight and temperature.

$$\frac{dX}{dt} = A \exp(-E_A/RT) f(X) f(\eta_L) \quad (I)$$

Analysis of an epoxy system showed that diffusion influenced the reaction rate only at vitrification (25). The general kinetic equation describing the reaction is then

$$\frac{dX}{dt} = A \exp(-E_A/RT) f(X). \quad (II)$$

The time to reach gelation

$$t_{gel}^* = \exp(Ar/T^*) \int_0^{X_{gel}} \frac{dX}{f(X)} \quad (III)$$

and the time to reach vitrification

$$t_{vit}^* = \exp(Ar/T^*) \int_0^{X_g(T^*)} \frac{dX}{f(X)} \quad (IV)$$

where $t_{gel}^* = At_{gel}$ and $t_{vit}^* = At_{vit}$ are the dimensionless times to reach gelation and vitrification, respectively; $T^* = T/T_{go}$ is a dimensionless temperature, and $Ar = E_A/RT_{go}$ is the Arrhenius number. X_{gel} and X_g are the extent of conversion of reactants at gelation and vitrification, respectively. The times to gelation and to vitrification can be computed using these equations (III and IV), knowledge of X_{gel} (for gelation) and a relationship between X_g and T_g (for vitrification), and the reaction kinetics. k) The vitrification curve is "S"-shaped. At temperatures immediately above T_{go} the time to vitrification passes through a maximum in consequence of the opposing influences of the temperature dependence of the viscosity and reaction rate constant. At higher temperatures the time to vitrification passes through a minimum in consequence of the opposing influences of the reaction rate constant and the decreasing concentration of reactive sites at vitrification as T_{go} is approached.

l) For epoxies the reactions become diffusion controlled in the vicinity of vitrification.

m) The reaction mechanism of a particular thermosetting system in principle can be deduced from the shape of the experimentally measured "S"-shaped vitrification curve.

n) Increasing cure time leads at the curing temperature to increasing conversion, T_g , crosslinking density, and density. Prior to vitrification the modulus at the curing temperature also increases. However in the glass state at room temperature, the corresponding modulus and density can

decrease whereas absorption of water can increase with increasing extent of cure (26).

TIME-TEMPERATURE-TRANSFORMATION (TTT) CURE DIAGRAM

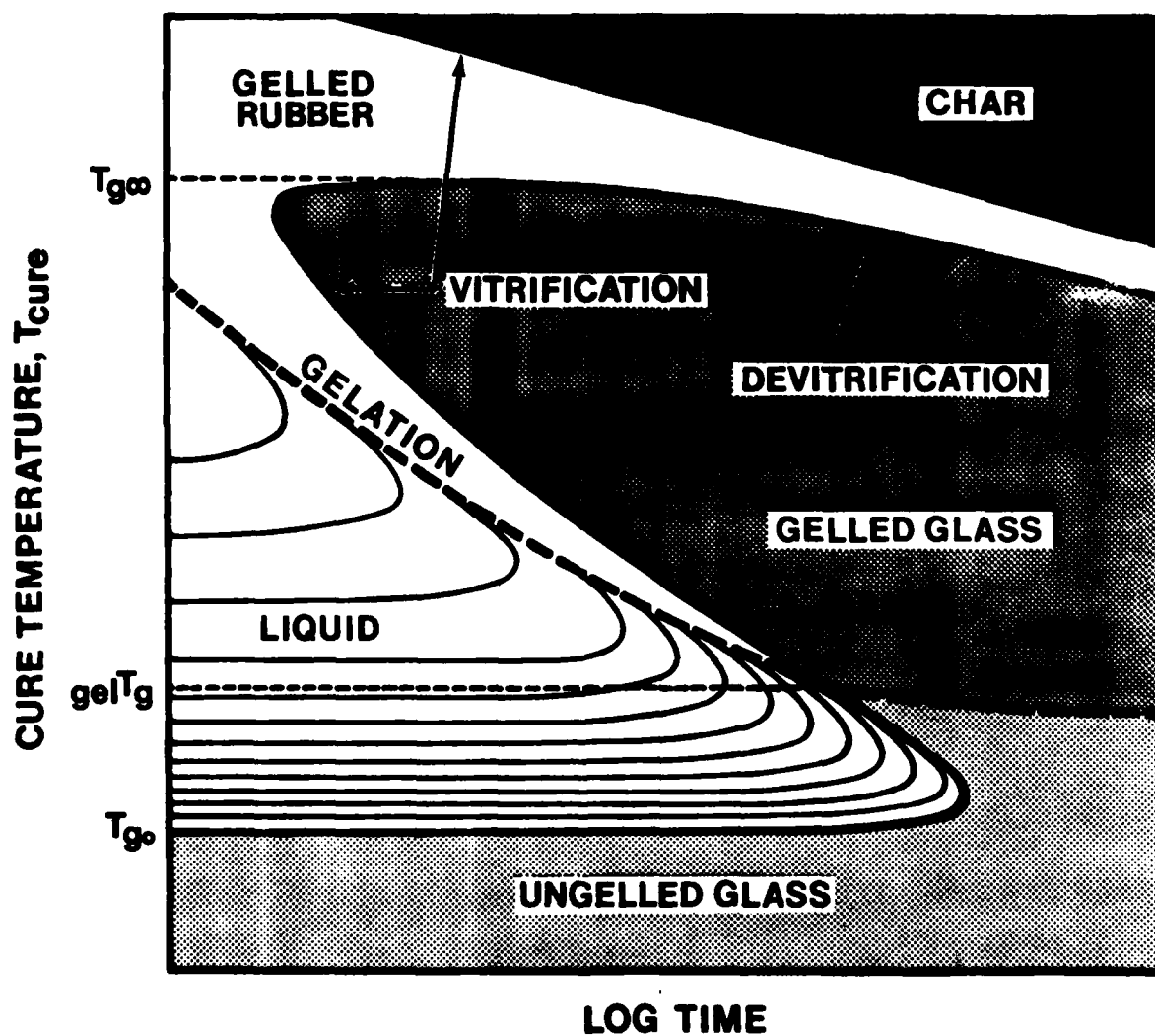


Figure 1

II. Much of the research has employed an adaptation of the freely oscillating torsion pendulum, "Torsional Braid Analysis (TBA)". Throughout this program the technique has been upgraded to include digital control and data reduction using hierarchical and desk-top computer systems (6,24).

III. The TBA technique (see II, above) has been central to developing the TTT cure diagram using epoxies (see I, above). It has also been used to characterize novel polymers such as bibenzoxazoles (5), tactic polymethylmethacrylates (6), and polyimides (14). It has also been employed to characterize high performance composite systems such as prepregs (21) and coated optical fibers (23). The technique was applied to difficult-to-investigate region above T_g of amorphous polymers where a relaxation, the T_{β} "transition", was investigated using homopolymers (2); blends of homopolymers (3), and block copolymers (13) of styrene.

IV. A new technique for investigating the thermal degradation of polymers was developed (4,7,8,10,16). The system uses a pyrolyzer in series with a thermal conductivity cell, a mass chromatograph, and an on-the-fly infrared spectrometer. The thermal conductivity cell gives the flexibility for selective trapping of volatile decomposition products, and also provides data that are complementary to thermogravimetric and differential thermal analyses. The mass chromatograph is composed of two gas chromatographs that are run parallel from a common injection port. Each chromatograph uses a different carrier gas and is equipped with a gas density detector. The instrument simplifies identification of mixtures of unknowns indirectly providing molecular weights, absolute quantities, and gas chromatographic retention times of the constituents. In this respect, the system is analogous to pyrolysis-gas chromatography-mass spectrometry. The infrared spectra together with the mass numbers provide a basis for assignment of molecular structure. The technique was used to investigate the degradation mechanism of polyolefins (7), polysulfones and polystyrene (8), and the influence of cis/trans ratio of 1,4-polybutadienes.

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AWARDS

- 1978 - American Chemical Society Award in the Chemistry of Plastics & Coatings (Borden Award).
- 1978 - North American Thermal Analysis Society Award in Thermal Analysis (Mettler Award)
- 1980 - American Chemical Society, Division of Organic Coatings and Plastics Chemistry; Best Technical Paper of Year (Doolittle Award)
- 1983 - Federation of Coatings Technologies; Best Technical Paper of Year (1st Prize Roan Award Competition)

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