

light, whether visible or ultraviolet, to elicit fluorescent emissions from the dyes described herein.

If desired, more than one dye can be used in the method of the invention. This is advantageous when different dyes react and bond to the resin at different reaction sites and at different stages in the curing cycle. This enables more accurate analysis of the rate and extent of cure of the resin system to be made. For example, a first dye can be used which exhibits maximum fluorescence during the first part of the reaction, and a second dye can be used which exhibits maximum fluorescence during the latter stages of the reaction. By proper calibration, total monitor of the cure can be realized.

The method of the invention can also be used to track extent and rate of cure in resin systems when the resin is incorporated into a composite material. This is exhibited in FIG. 1, which shows a fiber optic determination of cure in a fluorescein, carbon fiber composite.

The present invention may be embodied in other specific forms without departing from the spirit and scope thereof. These and other modifications of the will occur to those skilled in the art and are intended to fall within the scope of the appended claims.

What is claimed is:

1. A method for measuring the rate and extent of cure of a resin system undergoing polymerization which comprises:

A. adding at least one multifunctional fluorescent dye, in an amount sufficient to produce a measurable change in fluorescence, to the resin system comprising a resin undergoing polymerization, said dye being one which initially complexes with itself or with other components of the system, and subsequently chemically reacts with the resin and which degree of fluorescence of said dye varies in accordance with the degree of reaction with the resin;

B. polymerizing said resin to cure said resin system;

C. simultaneously measuring the fluorescence of the resin system using a probe and monitoring system; and

D. comparing the measured fluorescence at one or more wavelengths with pre-existing baseline values which are correlated with the extent of cure, whereby the rate and extent of cure of the system being evaluated is determined, wherein said multifunctional fluorescent dye is selected from the group consisting of (a) fluorescein, fluorescein diacetate, halo-substituted fluorescein, and nitro-substituted fluorescein, and (b) acridine, amino-substituted acridine, and lower alkyl-substituted acridine.

2. The method of claim 1 wherein said dye is selected from the group consisting of fluorescein, dibromodinitrofluorescein, tetraiodotetrachlorofluorescein, tetrabromofluorescein, 4',5'-dibromofluorescein, 2',7'-dichlorofluorescein, and 4',5'-diiodofluorescein.

3. The method of claim 1 wherein said dye is selected from the group consisting of acridine, amino-substituted acridine, and lower alkyl substituted acridine.

4. The method of claim 1 wherein said resin is an epoxy resin.

5. The method of claim 1 wherein said resin is a urethane resin.

6. The method of claim 1 wherein said measurement of fluorescence of said resin system is accomplished using a fiber optic probe inserted into said system.

7. The method of claim 1 wherein said measurement of fluorescence of said resin system is accomplished using a fluorometer.

8. The method of claim 1 wherein said resin system is heated to a temperature of from about 80° to about 200° C. while undergoing polymerization.

9. The method of claim 1 wherein the rate and extent of cure in the last 10 percent of the cure reaction is determined from said fluorescence measurements of the reaction system during polymerization.

10. The method of claim 1 wherein the amount of dye in said resin system is between about 1 to about 100,000 parts per million by weight.

11. The method of claim 1 wherein said resin is selected from the group consisting of polyethylene and copolymers, vinyls, polystyrene and copolymers, phenolics, polypropylene and copolymers, ureas and melamines, alkyds, polyesters, polyurethanes, polyacetals, polycarbonates, styrene-butadiene elastomers, acrylonitrile-butadiene elastomers, and acrylics.

12. The method of claim 3 wherein said dye is acridine yellow.

13. The method of claim 4 wherein said epoxy resin is a diglycidal ether of bisphenol A.

14. The method of claim 6 wherein said measurement of fluorescence of said resin system is accomplished using the fiber optic probe using a light wavelength of from about 400 nm to about 600 nm.

15. A method of measuring the rate and extent of cure of a resin system undergoing polymerization wherein the resin system comprises a thermosetting plastic resin and a curing agent therefore which comprises:

A. adding at least one multifunctional fluorescent dye in an amount sufficient to produce a measurable change in fluorescence, to the curing agent, said dye being one which initially complexes with itself or with other components of the system and subsequently chemically reacts with the resin and which degree of fluorescence varies in accordance with the degree of reaction with the resin;

B. mixing said curing agent with said thermosetting plastic resin;

C. polymerizing said thermosetting plastic resin to cure said resin system; and

D. simultaneously measuring the fluorescent emissions of said resin system;

E. comparing the measured fluorescent emissions at one or more wavelengths with pre-existing values which are correlated with the extent of cure, whereby the rate and extent of cure of the system being evaluated is determined;

wherein said multifunctional fluorescent dye is selected from the group consisting of (a) fluorescein, fluorescein diacetate, halo-substituted fluorescein, and nitro-substituted fluorescein, and (b) acridine, amino-substituted acridine, and lower alkyl-substituted acridine.

16. The method of claim 15 wherein said thermosetting plastic resin is an epoxy resin.

17. The method of claim 15 wherein said thermosetting plastic resin is a polyurethane.

18. The method of claim 16 wherein the amount of dye in said resin system is between about 10 to about 30 parts per million by weight.

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