Designing Cure Cycles for Matrix/Fiber Composite Parts

This methodology enables production of void-free laminates.

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A methodology has been devised for designing cure cycles to be used in the fabrication of matrix/fiber composite parts (including laminated parts). As used here, "cure cycles" signifies schedules of elevated temperature and pressure as functions of time, chosen to obtain desired rates of chemical conversion of initially chemically reactive matrix materials and to consolidate the matrix and fiber materials into dense solids. Heretofore, cure cycles have been designed following an empirical, trial-and-error approach, which cannot be relied upon to yield optimum results. In contrast, the present methodology makes it possible to design an optimum or nearly optimum cure cycle for a specific application.

Proper design of a cure cycle is critical for achieving consolidation of a reactive-matrix/fiber layup into a void-free laminate. A cure cycle for a composite containing a reactive resin matrix usually consists of a two-stage ramp-and-hold temperature profile. The temperature and the duration of the hold for each stage are unique for a given composite material. The first, lower-temperature ramp-and-hold stage is called the B stage in composite-fabrication terminology. At this stage, pressure is not applied, and volatiles (solvents and reaction by-products) are free to escape. The second, higher-temperature stage is for final forced consolidation.

The design of such a cure cycle is not trivial. The trial-and-error approach, still commonly used in industry, has several drawbacks:

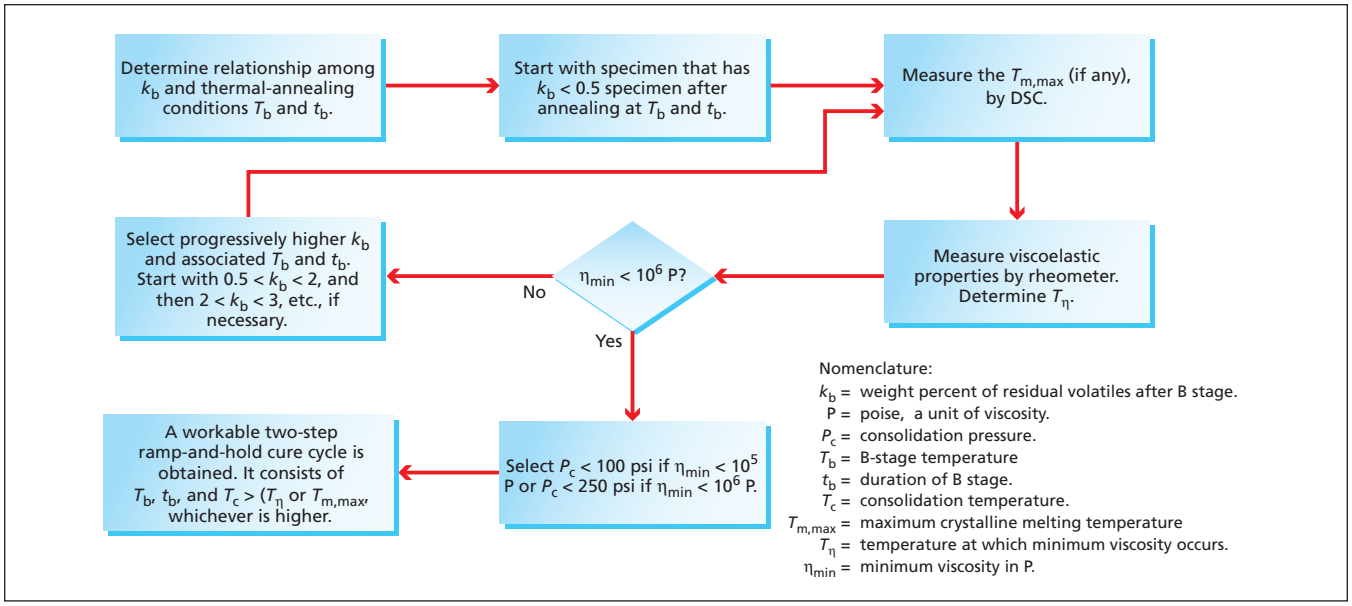
- Extensive experimentation is usually necessary for determining the proper cure cycle for a given material,
- A cure cycle found to be satisfactory for a given material under one set of conditions may not apply under a different set of conditions, and
- This approach does not ensure that the composite is cured completely under the optimal conditions and shortest amount of time.

Therefore, the trial-and-error approach is deemed costly, time-consuming, and inefficient as a means of designing cure cycles for the production of laminates of acceptable quality.

In order to make a void-free laminate, one must design the cure cycle to provide for depletion of a sufficient proportion of volatiles through the B stage, before consolidation. However, the viscosity of the resin increases during the B stage. Therefore, it is necessary to design the B stage so that the residual fluidity of the resin after the B stage is sufficient to enable infiltration of resin through fiber bundles during the subsequent pressure consolidation stage. The problem of balancing between the residual volatile content and the residual fluidity is very complex and unique for a given composite system.

The present methodology is founded on a universal "processing science" approach in which one uses available analytical equipment and techniques to effectively measure and logically analyze and design a workable cycle for any given unique resin/fiber composite. The methodology includes a protocol for:

- Measurements by a thermal gravimetric analyzer (TGA) to characterize mechanisms of depletion of volatiles,
- Differential scanning calorimetry (DSC) to characterize the degrees of imidization reactions and some aspects of the microstructures of partially cured resins, and
- Melt rheometry to characterize the residual fluidity and the temperature of onset of gelation of a partially cured resin.



This **Flow Diagram** represents the iteration scheme of the cure-cycle-design methodology.

On the basis of these measurements, a workable cure cycle for the subject composite system can be readily and logically designed.

This design methodology involves an iteration scheme (see figure) for satisfying several design criteria to arrive at the design cure cycle for any given thermoset-reactive-matrix-resin/fiber composite system. The number of iterations is based upon scientific judgments instead of empirical reasoning. A workable cure cycle can be established after only one iteration if the following criteria are satisfied:

- The residual volatile content after the B stage is < 0.5 weight percent;
- The forced-consolidation temperature (T_c) exceeds either the maximum crystalline melting temperature ($T_{m,max}$) or the temperature at which minimum viscosity occurs (T_η); and
- The residual minimum viscosity (η_{min}) is less than 10^6 poise.

In the event that η_{min} exceeds 10^6 poise, it is necessary to perform a second iteration utilizing a less severe B-stage condition. This condition results in greater fluidity and greater residual volatile content after the B stage. Consequently, η_{min} is reduced to < 10^6 poise, making it possible to use only moderate pressure for final consolidation. Optionally, during the second temperature ramp before final forced consolidation, the application of pressure can be delayed until the temperature reaches T_η in order to allow for additional depletion of volatiles.

The subject resin/fiber composite material is considered unprocessable (in that a laminated part made of this material cannot be made free of voids) under moderate pressures when the above-mentioned criteria cannot be satisfied concurrently. It is possible to refine the cure cycle by narrowing the B-stage pretreatment conditions in the TGA, DSC, and melt-rheometry analyses.

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