

GENERATION OF RESIDUAL STRESSES AND DISTORTION DURING THE CURE OF POLYMER MATRIX COMPOSITES

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ABSTRACT

Experimental results are presented on the development of curvature in unsymmetric laminates during the cure. Measurements of spring-in of curved sections during the cure are also presented, which show quite a different trend. The results illustrate the relative importance of differential thermal contraction and cure shrinkage, and show how these mechanisms contribute to development of stresses and distortion in components during the cure.

1. INTRODUCTION

Residual stresses during the cure can lead to cracking. They also produce distortion that result in difficulties in assembly leading to increased costs. Although these problems are well known, the way in which stresses and distortion build up during the cure is still not fully understood. Previous experimental work using interrupted cures has concentrated on distortion of unsymmetric laminates [e.g. 1-3]. This paper presents results from a study where the interrupted cure technique has been applied to measuring the development of spring-in of curved composites [4]. Results are compared with curvatures from interrupted cures of unsymmetric cross-ply laminates [5], and show quite different behaviour. The results are explained in terms of the relative importance of differential thermal contraction and cure shrinkage, and the differences in properties when the material is above and below the glass transition temperature.

2. DEVELOPMENT OF CURVATURE IN UNSYMMETRIC LAMINATES

Strips of AS4/8552 carbon-epoxy with dimensions of 300 x 30 x 1 mm were cured up to pre-determined points of the manufacturer's recommended cure cycle and then cooled very quickly to room temperature.

Specimens were placed on a 4 mm thick Aluminium plate, with release film, and cured in an oven under vacuum conditions only. For these flat laminates of relatively small in-plane dimensions the oven curing technique gives products of good quality, not dissimilar to those cured in an autoclave, with only a slight decrease in the fibre volume fraction. The manufacturer's recommended cure cycle consists of a first ramp of 2°C/min up to 120°C and a first hold at 120°C for 60 min, a second ramp of 2°C/min up to 180°C and a second hold at 180°C for 120 min. All laminates were cured with the 90° ply against the tool. Initial tests showed no significant difference in curvature between specimens with the 0° or 90° ply against the tool for vacuum only curing.

The specimens were quenched to prevent any additional cure during cooling. This was achieved by removing the Aluminium plate from the oven and putting it suddenly on a 19 mm thick Aluminium block at room temperature covered by a thin layer of water. In the first minute, the temperature dropped from 180°C to about 80°C. After another minute, an additional 20°C drop was registered. After five minutes the temperature was about 50°C. After about 30 minutes specimens were removed from the vacuum bag and measured.

The cured laminates showed a principal bow in the longitudinal direction and a lateral bow due to the anticlastic effect. About 5 mm was trimmed from each edge to remove the neat

resin areas and thin composite edges resulting from resin flow. The curvature in the longitudinal direction was then determined by setting the samples on a flat cast iron gauging block and using high precision height and length gauges to measure the dimensions. The measurements confirmed that the deformed shape was very close to a circular arc, and this assumption was used in calculating the curvatures.

The measurement of the specimens was carried out immediately after the cure, in order to avoid moisture infiltration into the laminate. This is important, as moisture absorption reduces the curvature.

Fig. 1 shows the curvatures for 8 specimens quenched at different points in the cure cycle [5].

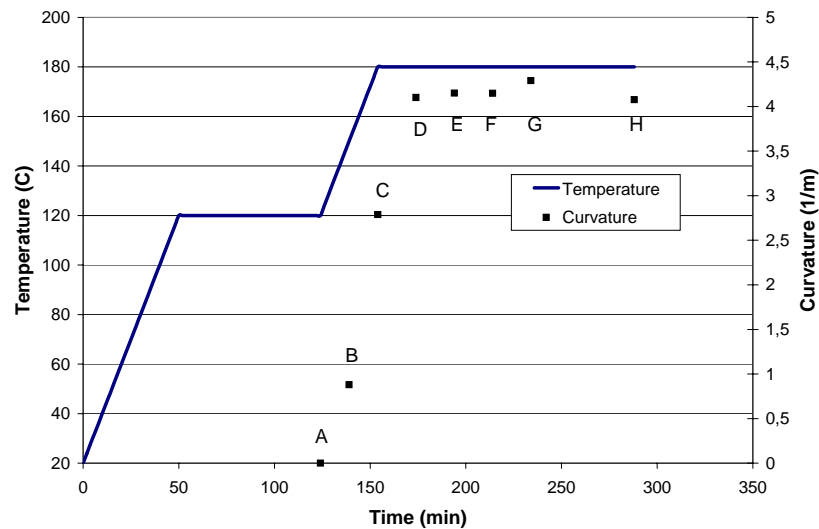


Fig. 1: Curvature of 0/90 laminates from interrupted cures

In order to understand these results it is important to know how the glass transition temperature, T_g , develops during the cure. Results are presented in Fig. 2 for T_g for specimens quenched at different stages in the cure cycle. These measurements were made using DSC at QinetiQ [6]. The fit to a cure kinetics model developed at QinetiQ is also shown.

No curvature arises early in the cure (A) until T_g rises above room temperature (B). The distortion is primarily due to thermal stresses on the cooldown. When specimen B was reheated, it became flat again as T_g was reached. It was then heated further and cooled back to room temperature. It remained flat until T_g was passed on the cooldown, after which curvature developed linearly with decreasing temperature [5].

The final curvature increases steadily with further cure (C, D) as T_g develops. It then reaches a plateau after vitrification (D-H). During this phase there is further curing, and the T_g is rising. This does not cause further distortion because curvature is still determined by the thermal stresses on cooldown from 180°C to 20°C. The further curing from D to H is not accompanied by significant chemical shrinkage. On reheating fully cured specimens (H) they become flat at 186°C. This shows that only about 5% of the curvature is due to non-thermoelastic effects, mainly cure shrinkage.

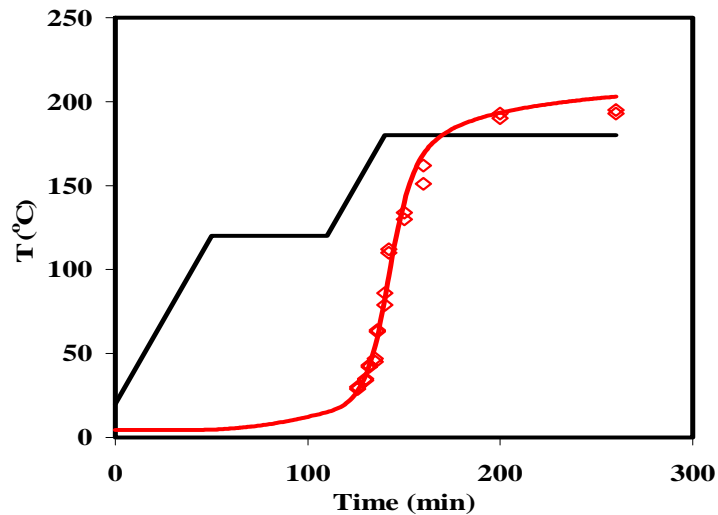


Fig. 2: Glass transition temperatures from interrupted cures

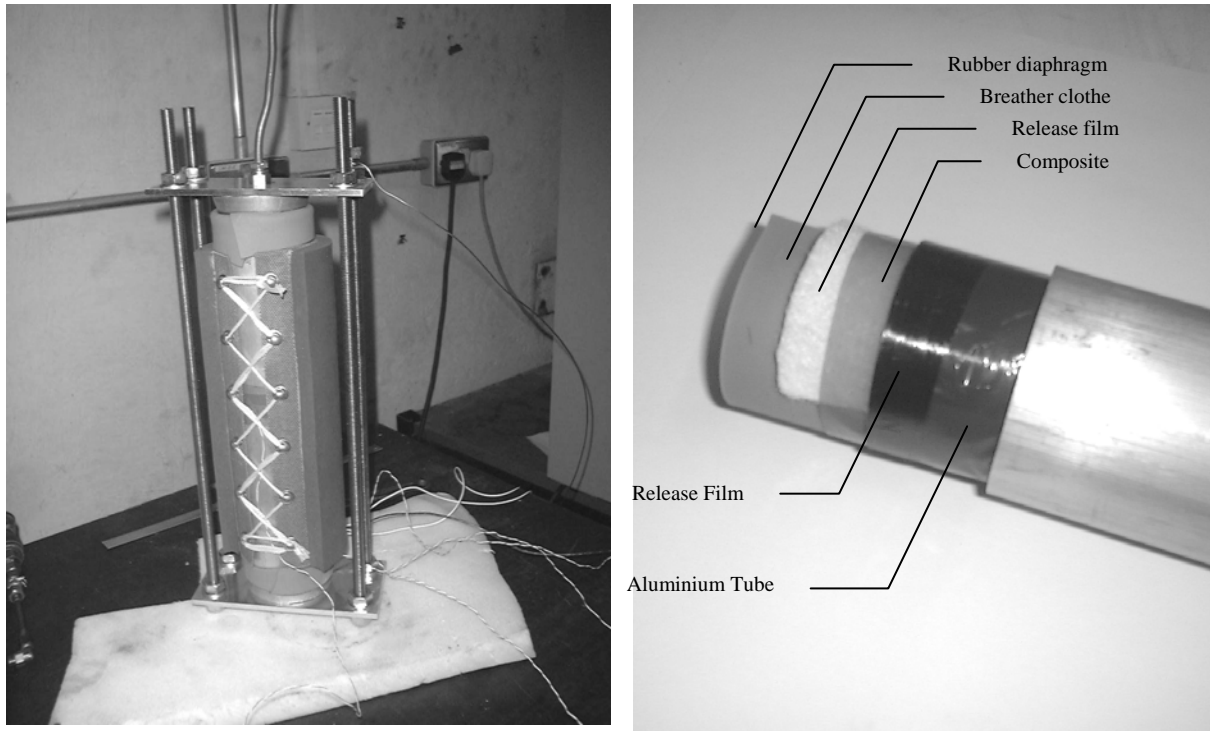
3. DEVELOPMENT OF SPRING-IN

Segments of 270° of tubes of AS4/8552 with the fibres in the hoop direction were cured on the inside of an aluminium tube. Specimens were quenched from various points during the cure, and the spring-in measured. The cure quench equipment is shown in Fig. 3 and consists of an aluminium tube with 63 mm inner diameter, 70 mm outer diameter and 300 mm length. A hole was drilled in the tube to allow application of vacuum and venting. Heat is applied with the help of a flexible silicone heating element wrapped around the tube (Fig. 3a). The heating element is fitted with boot hooks enabling quick release. After removing the heating element, the tube can be quenched in water. Fig. 3b shows the lay-up for curing the samples. The prepreg is wrapped around $\frac{3}{4}$ of the perimeter of a tube of comparable diameter to make a 8-ply C-shaped preform of 270 degrees, 2 mm thick. Layers of PTFE coated glass fabric release film were used between the aluminium tube and composite as well as between the composite and breather cloth. A silicone rubber diaphragm was used to transfer the pressure applied in the tube to the composite.

The temperature was controlled with a programmable PID process controller within a $\pm 3^\circ\text{C}$ band around the process temperature. The uniformity of the temperature was checked with eight thermocouples around the outer surface of the tube. A pressure of 0.7 MPa was applied throughout the manufacturer's recommended cure cycle. The quench rate attained during the experiments was $600^\circ\text{C}/\text{min}$.

The outer diameters of the composite parts were measured at room temperature and spring-in angles were calculated for a 90° angle with respect to the tube inner diameter measured at 180°C using a ball nosed gauge.

Fig. 4 shows the results [4]. The amount of spring-in increases, peaks just after the start of the 180°C hold, and then decreases, again reaching a plateau after vitrification. This reducing spring-in with increasing cure is due to the effect of volume changes while the material is above T_g , and is illustrated in Fig. 5. On cooldown from point A the partially cured material is initially above T_g where it has a high through-thickness thermal expansion coefficient, α . When it reaches T_g at point E, α reduces, and the rate of spring-in decreases as the material cools to room temperature (point F). When further curing has occurred, the material reaches T_g on the cooldown earlier, at point C, and the final spring-in at point D is reduced. Finally, when the material vitrifies at the cure temperature, it cools from point A immediately with the



(a) Quenchable tube with flexible silicone heater

(b) The lay-up of the samples

Fig. 3: Cure quench experiment setup

lower value of α , attaining an even smaller final spring-in angle at point B. The development of T_g during the cure was shown in Fig. 2.

Initially chemical shrinkage increases, adding to the effect of thermal contraction and leading to increasing spring-in with cure. As T_g increases, more and more of the contraction on cooldown takes place after vitrification, leading to reduced spring-in. Once T_g reaches the cure temperature, all the cooldown is in the vitrified state, and since there is little further chemical shrinkage, spring-in remains constant.

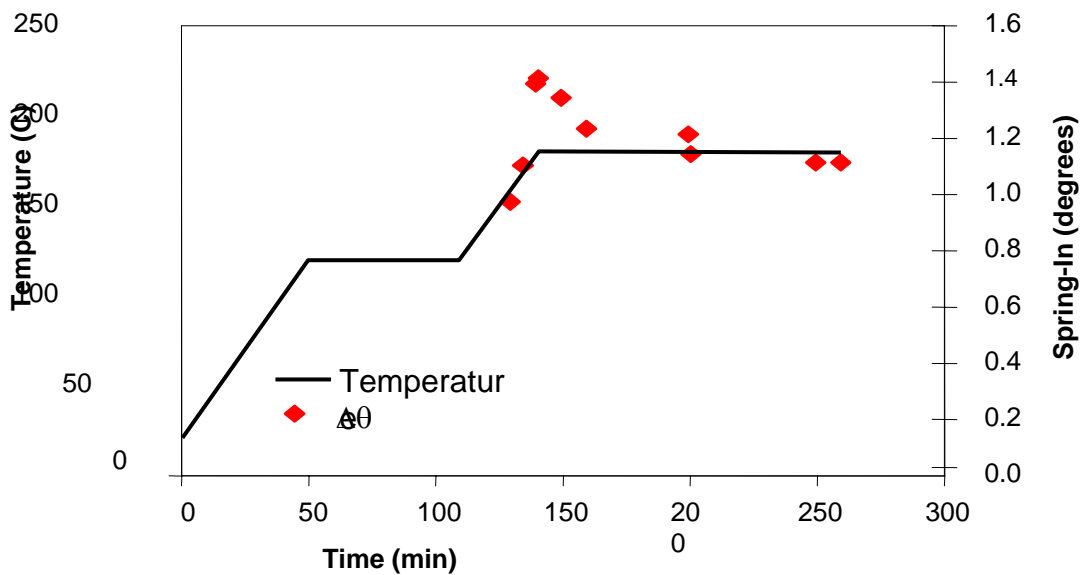


Fig. 4: Spring-in of curved specimens from interrupted cures

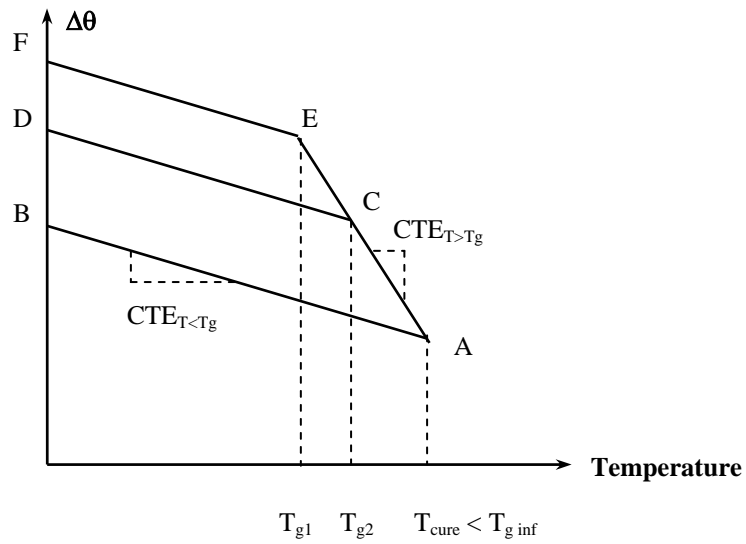


Fig. 5: Spring-in above and below the glass transition temperature

The three components of spring-in due to chemical shrinkage, thermal contraction above T_g and thermal contraction below T_g can be quantified based on the thermal and shrinkage coefficients, allowing the total spring-in throughout the cure to be determined [4]. This is shown in Fig. 6, and the result matches the experimental measurements very well. Chemical shrinkage is responsible for around 50% of the spring-in.

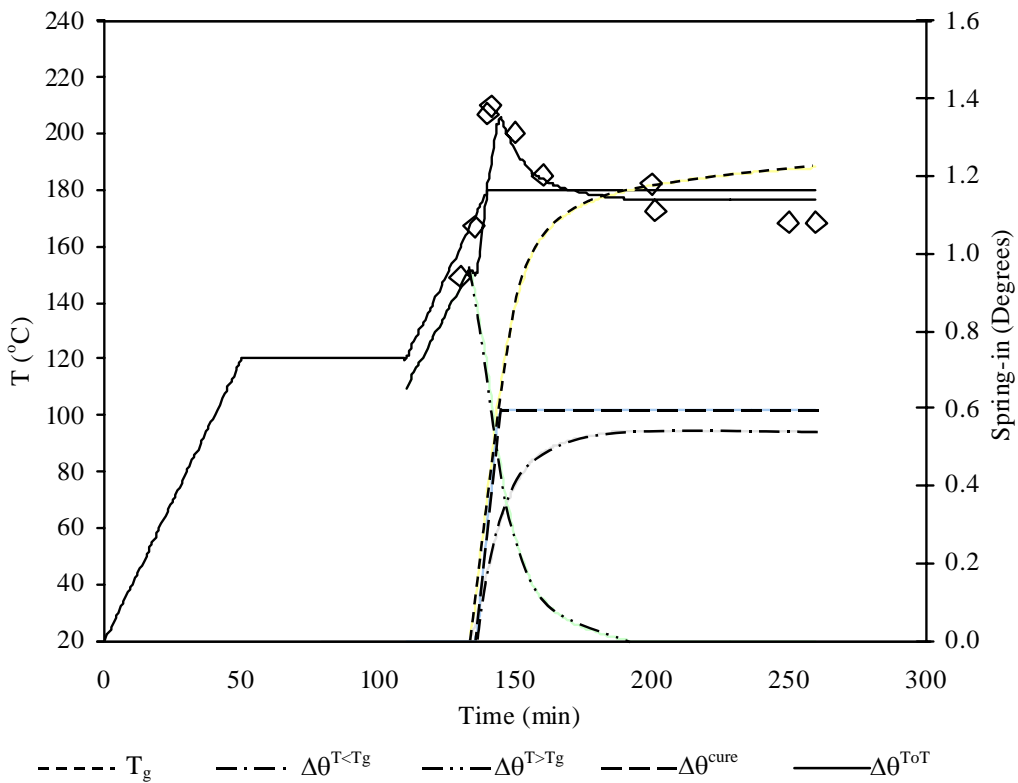


Fig. 6: Components contributing to the final spring-in

4. CONCLUDING DISCUSSION

Results for interrupted cures on non-symmetric laminates showed distortion starting when the T_g increases above room temperature. Curvature then increased steadily, reaching a plateau at the point where vitrification occurs at the cure temperature.

In contrast distortion due to spring-in reached a peak at the start of the final dwell, and then reduced, again reaching a plateau at vitrification. This difference in behaviour is because volume changes while the material is above T_g contribute to spring-in, but not to curvature of unsymmetric laminates. The reason chemical shrinkage and thermal effects before vitrification contribute to spring-in is because they cause a geometrical change in shape which does not require stresses to sustain it. In contrast unsymmetric laminates require a moment to bend, and the low stresses before vitrification are not sufficient to produce significant distortion.

For the unsymmetric laminates nearly all the curvature was due to differential thermal contraction. However for the spring-in, chemical shrinkage was responsible for only around 50% of the distortion. These results illustrate the relative importance of thermal and cure shrinkage effects, and show how these mechanisms contribute to development of stresses and distortion in components during the cure.

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