



**HAL**  
open science

## Characterization and modelling of spring-in effect on Z-shape composite part

C. Sonnenfeld, R. Agogu , P. Beauch ne, Y. Nawab, A. Saouab, E. Anfray, B.  
Desjoyaux

► **To cite this version:**

C. Sonnenfeld, R. Agogu , P. Beauch ne, Y. Nawab, A. Saouab, et al.. Characterization and modelling of spring-in effect on Z-shape composite part. ECCM17, Jun 2016, MUNICH, Germany. hal-01354668

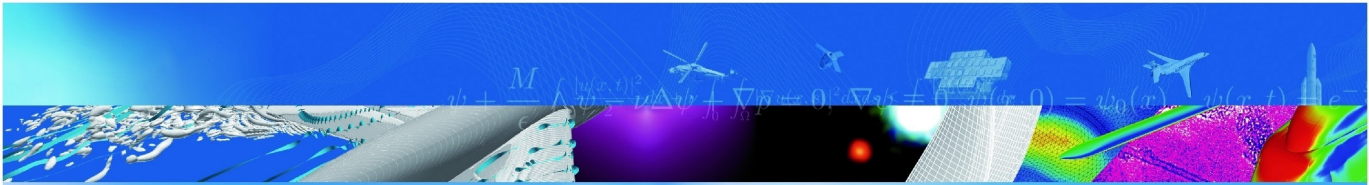
**HAL Id: hal-01354668**

**<https://hal.science/hal-01354668>**

Submitted on 19 Aug 2016

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destin e au d p t et   la diffusion de documents scientifiques de niveau recherche, publi s ou non,  manant des  tablissements d'enseignement et de recherche fran ais ou  trangers, des laboratoires publics ou priv s.



COMMUNICATION A CONGRES

**Characterization and modelling of  
spring-in effect on Z-shape  
composite part**

C. Sonnenfeld (ONERA), R. Agogu  (ONERA),  
P. Beauch ne (ONERA), A. Saouab, E. Anfray,  
B. Desjoyeaux

ECCM17  
MUNICH, ALLEMAGNE  
26-30 juin 2016

TP 2016-443

**70** 2016  
ans

**ONERA**

THE FRENCH AEROSPACE LAB

## CHARACTERIZATION AND MODELLING OF SPRING-IN EFFECT ON Z-SHAPE COMPOSITE PART

C. Sonnenfeld<sup>1</sup>, R. Agogué<sup>1</sup>, P. Beauchêne<sup>1</sup>, Y. Nawab<sup>2</sup>, A. Saouab<sup>2</sup>, E. Anfray<sup>3</sup>, B. Desjoyeaux<sup>3</sup>

<sup>1</sup>ONERA, the French Aerospace Lab, 29, avenue de la division Leclerc, Châtillon, France

Email: camille.sonnenfeld@onera.fr, Web Page: <http://www.onera.fr>

<sup>2</sup>Laboratoire d'Ondes et Milieux Complexes, UMR 6294 CNRS, Université du Havre, 53, rue Prony,  
Le Havre, France

Email: abdelghani.saouab@univ-lehavre.fr, Web Page: <http://www.lomc.fr/>

<sup>3</sup>Aircelle SAFRAN, Route du Pont VIII, Gonfreville-l'Orcher, France

Email: emmanuel.anfray@aircelle.com, Web Page: <http://www.aircelle.com/>

**Keywords:** Spring-in, residual stress, cure modeling

### Abstract

In this study, a simple finite-element model is developed to predict the spring-in of Z-section composite parts made of carbon fiber/ epoxy resin manufactured with resin transfer molding process. The different mechanisms occurring during manufacturing and effective in the formation of residual stresses and shape distortions are implemented. They are mainly the thermal effect and the chemical shrinkage of the resin during the cross-linking polymerization reaction. A dedicated experimental procedure allowing separation of strains to cure shrinkage and thermal expansion is presented to characterize the resin cure shrinkage. Cure kinetic properties of the resin are determined with differential scanning calorimetry measurements. The spring-in angles predicted by the finite element analysis are compared to the angles measured on the Z-section specimens of various thicknesses.

### 1. Introduction

Composite parts with curved geometry present shape distortion when they come out of the mold after heating. Shape distortion such as spring-in effect induces difficulties in assembling composite structures leading to increased costs and reduced properties. Because of the lack of established methods for prediction of shape distortions, mold development and manufacturing are mainly based on extensive trial and error strategies which are costly and time consuming. This is especially true for components with large and complex geometries. Therefore, there is a great interest in improving the understanding and the prediction of shape distortions and residual stresses [1]. In the context of understanding the damage behavior of woven composites, being able to quantify the induced residual stresses is of major importance since it will allow predicting the part durability.

Several phenomena occurring during the cure of the resin are responsible for the composite shape distortions: mainly the thermal effect and the chemical shrinkage of the resin during the cross-linking polymerization reaction. Therefore a good knowledge of these parameters is required for an improved prediction of the part geometry after manufacturing. The present paper focuses on cure induced mechanisms responsible for shape distortions in Z-shape thermosetting composite parts with a special attention to be paid to the characterization of thermal and cure shrinkage strain.

To do so, Z-shape composite parts were produced by RTM (resin transfer molding) process with a mold designed to create composite part with two different radii of curvature and of different

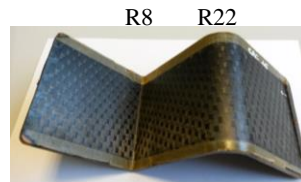
thicknesses. This specific RTM mold allows a parametric study of the spring-in angle using the same manufacturing tool which reduces experimental dispersion. The results of the spring-in angle values measured on parts of different thicknesses were compared to finite element simulations of the part curing process. Characterization of thermal and cure shrinkage of the resin are performed. The results of the resin characterizations were implemented within the finite element model. Finally, we conclude on the accuracy of our model to simulate the induced shape distortions of Z-shape composite parts.

## 2. Material properties

The resin has been characterized to measure its cure kinetic, its thermal dilatation and its cure shrinkage. We present an experimental set-up specifically developed to separated thermal and cure shrinkage in a single test.

### 2.1. Fabrication cycle and spring-in angles

The material used for all parts in this study is carbon fiber /epoxy RTM6 system. The parts are balanced and symmetric. The composite parts are manufactured with RTM process in a mold designed to create Z-sections with two different radii of curvature and different thicknesses. A classical cure cycle for RTM6 resin is selected: heating from room temperature to 180°C maintained 2 hours and cooling to room temperature.



**Figure 1.** Example of Z-section manufactured in this study. The R8 and R22 label refer to the radii of curvature of 8 and 22 mm. The arm length is approximately 80 mm and the width of the part is 90 mm.

After demolding, approximately 10 mm was trimmed off the width of each side to eliminate the effect of edge thinning on spring-in measurements (corresponding to the brown part in Figure 1). The ends were not trimmed but measurements were not taken close from the ends to avoid edge effects. The list of samples manufactured samples is given in Table 1 associated with the spring-in angles measured for the different radii of curvature.

**Table 1.** Manufactured specimens and spring-in angles for the different radii of curvature.

| Specimen label | Thickness (plies) | Spring-in of R8 (°) | Spring-in of R22 (°) |
|----------------|-------------------|---------------------|----------------------|
| S1             | 9                 | 0.9                 | 0.8                  |
| S2             | 11                | 0.6                 | 0.5                  |
| S3             | 4                 | 1.0                 | 1.0                  |
| S4             | 5                 | 1.2                 | 1.0                  |

### 2.2. Cure kinetic of the resin

The reaction kinetic of the resin is determined with a 2920 DSC from Mettler-Toledo by measuring the heat of reaction of samples of few milligrams at different heating rates. In this study, the resin cure is model with a phenomenological autocatalytic kinetic equation (Eq. 1) proposed by Kamal-Sourour [2] which has been widely used in literature for modelling epoxy cure kinetics.

$$\frac{\partial \alpha}{dt} = (K_1 + K_2 \alpha^m)(1 - \alpha)^n \quad \text{with} \quad K_i = A_i \exp\left(-\frac{E_i}{RT}\right) \quad (1)$$

In which  $\alpha$  is the fractional conversion or degree of cure,  $d\alpha/dt$  is the reaction rate,  $m$ ,  $n$  are the reaction orders,  $A_i$  is the pre-exponential factor of the  $i^{\text{th}}$  reaction,  $E_i$  is the activation energy of the  $i^{\text{th}}$  reaction,  $T$  is the absolute temperature and  $R$  is the universal gas constant. The parameters of the model are given in Table 2 and are in close agreement with data given in previous studies [3].

**Table 2.** Parameters of the Kama-Sourour for modeling the RTM6 cure kinetic.

| $E_1$<br>(J/mol) | $E_2$<br>(J/mol) | $A_1$<br>(1/s) | $A_2$<br>(1/s) | $m$<br>(ua) | $n$<br>(ua) |
|------------------|------------------|----------------|----------------|-------------|-------------|
| 73610            | 27612            | 54490          | 7050           | 1.14        | 1.38        |

### 2.3. Thermal deformation

The linear coefficient of thermal expansion ( $CTE_{lin}$ ) of the resin is necessary to calculate by homogenization the  $CTE_{lin}$  of the composite. Measurements have been realized with a thermal mechanical analyzer (TMA) from SETSYS Evolution 16/18. A thermal gradient is applied on pre-dried samples under inert atmosphere while monitoring its dilatation. The  $CTE_{lin}$  variation is assumed to be linear with temperature, the  $CTE_{lin}$  values are determined by a simple linear regression of the thermal dilatation over a certain range of temperature. Values of  $CTE_{lin}$  in the range 40°C to 160°C have been measured around  $68.6 \times 10^{-6} \pm 1.5 \times 10^{-6}/^\circ\text{C}$  for RTM6 resin.

### 2.4. Chemical shrinkage

Several methods exist to determine the matrix volumetric shrinkage due to curing and thermal effects as explained in [4]. In our study, we used a gravimetric method in which the volumetric changes of a sample of resin can be monitored during the entire cure cycle. It is based on the buoyancy principle: the buoyancy force of a sample partially or fully immersed in a fluid is equal to the weight of the fluid displaced by the specimen body. This method has been already used in literature in the case of isothermal cure [5,6]. In the case of our study, a cure cycle is applied to the immersed sample of resin which allows measuring the resin volume variation ( $\Delta V/V_0$ ) due to both thermal expansion and cure shrinkage during the full cure cycle (Eq. 2).

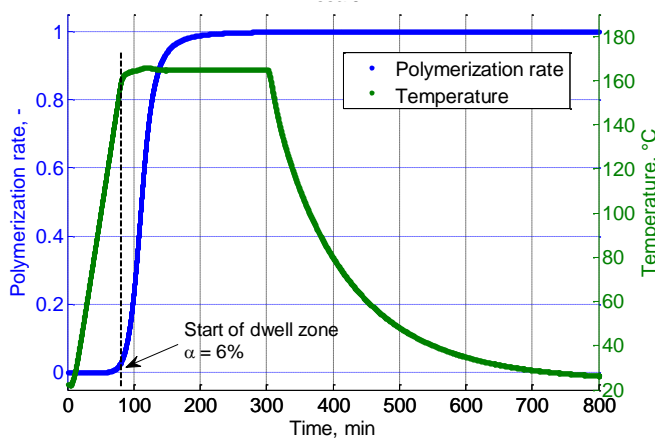
$$\frac{\Delta V}{V_0} = CTE_{vol} \cdot \Delta T - CS \cdot \Delta \alpha \quad (2)$$

In which  $CTE_{vol}$  is the coefficient of volumetric thermal expansion,  $CS$  is the cure shrinkage coefficient,  $\Delta T$  is the change of temperature of the resin, and  $\Delta \alpha$  is the degree of cure. From experimental data, one can deduce the resin volume variation which is linked to the variation of the immersed sample mass  $m^{im}(T)$ , the initial sample density  $\rho_{sample}(T_0)$ , the initial mass of the sample in air  $m_0^{air}$  and the variation of the fluid density with temperature  $\rho_{fl}(T)$  as defined by Eq. 3.

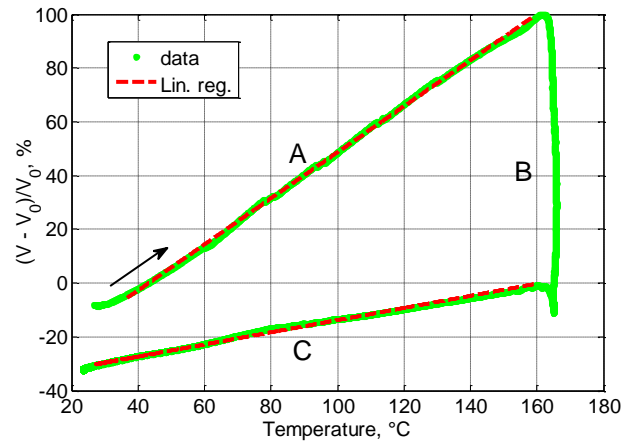
$$\frac{\Delta V}{V_0} = \frac{m_0^{air} - m^{im}(T)}{m_0^{air}} \cdot \frac{\rho_{sample}(T_0)}{\rho_{fl}(T)} - 1 \quad (3)$$

In order to dissociate the variation of volume due to thermal expansion and to cure shrinkage during the cure cycle, a specific cure cycle is applied on the resin sample: the resin is heated up at a rate of

2°C/min, a dwell temperature of 160°C is applied for 3 hours then the sample is cooled down. From the thermo-kinetic model proposed in Section 2.2, the degree of cure is calculated from the thermal profile. As displayed on Figure 2, at the beginning of the dwell temperature, the reaction rate has only reached 6%, which means that the main part of cure shrinkage occurs on the dwell zone. In Figure 3, in which the volumetric variation of the resin sample as a function of temperature cycle is presented. Part A is associated with the thermal dilatation of the liquid resin and part C represents the contraction of the solidified resin during cooling. The slope of this curve portion represents the  $CTE_{vol}$ , which reported to the  $CTE_{lin}$  presents a good agreement with the value measured with TMA (less than 10% of error). At constant temperature, part B is related to the cure shrinkage of the resin. Finally, the difference between the start and the end of the curve represents the apparent volumetric shrinkage. Using the thermo-kinetic model of the resin, one can determine the evolution of the resin volumetric cure shrinkage as a function of the degree of cure.



**Figure 2.** Thermal profile applied on the resin sample and advancement of polymerization rate.



**Figure 3.** Normalized variation of volume of the resin sample during the cure cycle calculated from Eq. 3.

### 3. Comparison between model and experimental results

#### 3.1. Material behavior

The material behavior of the composite is defined by a thermoelastic law as follows

$$\underline{\underline{\sigma}} = \underline{\underline{\tilde{C}}} : (\underline{\underline{\varepsilon}} - \underline{\underline{\varepsilon}}^{th} - \underline{\underline{\varepsilon}}^{cs}) \quad (4)$$

where  $\underline{\underline{\sigma}}$  and  $\underline{\underline{\varepsilon}}$  are respectively the stress and strain tensors.  $\underline{\underline{\tilde{C}}}$  is the effective elastic stiffness tensor.  $\underline{\underline{\varepsilon}}^{th}$  and  $\underline{\underline{\varepsilon}}^{cs}$  are respectively the thermal and chemical strains defined as

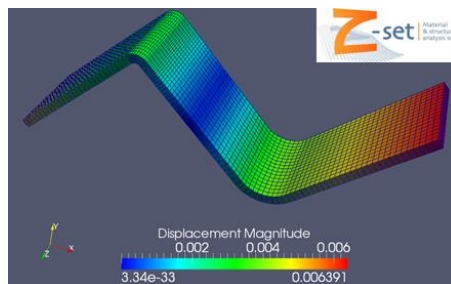
$$\underline{\underline{\varepsilon}}^{th} = \underline{\underline{CTE}} : \Delta(T - T_0) \quad \text{and} \quad \underline{\underline{\varepsilon}}^{cs} = \underline{\underline{CS}} : \Delta\alpha \quad (5)$$

where  $\underline{\underline{CTE}}$  is the thermal tensor,  $T$  is the temperature and  $T_0$  is the stress free temperature.  $\underline{\underline{CS}}$  is the cure shrinkage tensor and  $\Delta\alpha$  is the degree of cure. The thermal and cure shrinkage tensors have been calculated through a simple rule of mixture as explained in [7] using as input data thermo-elastic properties of the fiber and the resin and the fiber volume content. The stress free temperature is the temperature at which a component would reach a stress-free state upon re-heating at the end of the cure process. This parameter has been measured to 160°C by heating a cured Z-section part until it

turns back to a 90° angle (mold angle). This method is described in [8] for asymmetric panels in curved configurations.

### 3.2. Finite element model

A finite element (FE) analysis has been performed to check the thermoelastic model on the prediction of spring-in of Z-shape composite parts using the FE solver Zebulon/Zset [9]. Z-shaped parts have been modelled using 3D twenty-node brick elements. A homogeneous temperature is applied on the all part (no thermal gradient) and the polymerization is assumed homogeneous. The spring-in is calculated from the nodal displacements (see Figure 4). The results of the finite element calculations gives a spring-in angle of 1.0° which is very close to the results presented in Table 1 for a 4/5 ply thicknesses.



**Figure 4. Shape prediction of the Z-shape composite part calculated using FE model.**

A sensitivity analysis has been performed to determine the influence of changes in stress free temperature  $T_0$  and in chemical shrinkage  $\epsilon^{cs}$  on the spring-in. It shows that a variation of  $\Delta T_0 = 20^\circ\text{C}$  induces a change of  $0.15^\circ$  which corresponds to 20% on the spring-in angle. This sensitivity analysis also shows that a variation of 20% on the value of the cure shrinkage strains induces only a variation of 8% on the spring-in angle. Finally, it allows concluding on the weight of thermal and chemical strain contribution on spring-in values: chemical strain contributes to 40% to the total spring-in. This result confirms the importance of being able to characterize precisely the cure shrinkage strain to be able to predict the final shape of composite parts.

### 4. Conclusion

In this work, a rather simple model of material behavior has been developed for predicting the shape of Z-shaped composite parts. This model has been implemented with thermal and cure shrinkage material properties which have been measured using dedicated set-up. A particular attention has been paid to the experimental characterization of the resin cure shrinkage. The prediction of the model has been compared to spring-in angles measured on Z-shape carbon/epoxy composite parts manufactured with RTM process. A good agreement between the model and the experimental data has been found.

### References

- [1] Nawab Y, Jaquemin F, Casari P, Boyard N, Sobotka V. Evolution of chemical and thermal curvatures in thermoset-laminated composite plates during the fabrication process. *Journal of Composite Materials* 2013;47:327–39.
- [2] Kamal MR, Sourour S. Kinetics and thermal characterization of thermoset cure. *Polymer Engineering & Science* 1973;13:59–64.
- [3] Navabpour P, Nesbitt A, Degamber B, Fernando G, Mann T, Day R. Comparison of the curing kinetics of the RTM6 epoxy resin system using differential scanning calorimetry and a microwave-heated calorimeter. *J Appl Polym Sci* 2006;99:3658–68.

- [4] Nawab Y, Shahid S, Boyard N, Jacquemin F. Chemical shrinkage characterization techniques for thermoset resins and associated composites. *Journal of Materials Science* 2013;48:5387–409.
- [5] Li C, Potter K, Wisnom MR, Stringer G. In-situ measurement of chemical shrinkage of MY750 epoxy resin by a novel gravimetric method. *Composites Science and Technology* 2004;64:55–64.
- [6] Parlevliet PP, Bersee HEN, Beukers A. Shrinkage determination of a reactive polymer with volumetric dilatometry. *Polymer Testing* 2010;29:433–9.
- [7] Gay D. *Matériaux composites*. Lavoisier; 2015.
- [8] Djokic D, Johnston A, Rogers A, Lee-Sullivan P, Mrad N. Residual stress development during the composite patch bonding process: measurement and modeling. *Composites Part A: Applied Science and Manufacturing* 2002;33:277–88.
- [9] Z-set | Non-linear material & structure analysis suite 2016. <http://www.zset-software.com/> (accessed April 11, 2016).

### **Acknowledgments**

This work has been made in the framework of PRC Composite research project which has been funded by DGAC, Safran and ONERA.