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# Investigation of the Residual Stress State in an Epoxy Based Specimen

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**Abstract.** Process induced residual stresses may play an important role under service loading conditions for fiber reinforced composite. They may initiate premature cracks and alter the internal stress level. Therefore, the developed numerical models have to be validated with the experimental observations. In the present work, the formation of the residual stresses/strains are captured from experimental measurements and numerical models. An epoxy/steel based sample configuration is considered which creates an in-plane biaxial stress state during curing of the resin. A hole drilling process with a diameter of 5 mm is subsequently applied to the specimen and the released strains after drilling are measured using the Digital Image Correlation (DIC) technique. The material characterization of the utilized epoxy material is obtained from the experimental tests such as differential scanning calorimetry (DSC) for the curing behavior, dynamic mechanical analysis (DMA) for the elastic modulus evolution during the process and a thermo-mechanical analysis (TMA) for the coefficient of thermal expansion (CTE) and curing shrinkage. A numerical process model is also developed by taking the constitutive material models, i.e. cure kinetics, elastic modulus, CTE, chemical shrinkage, etc. together with the drilling process using the finite element method. The measured and predicted in-plane residual strain states are compared for the epoxy/metal biaxial stress specimen.

## Introduction

The usage of fiber reinforced polymer (FRP) composites has been increasing extensively in several industries such as aerospace, construction, wind energy, automotive, etc owing to their desirable properties such as high strength/weight ratio and corrosion resistance. Structural load carrying parts are also being produced using the FRP composites such as bridge components, wind turbine blade reinforcements and reinforcing rebar for concrete. There are several techniques to manufacture the FRP composites, here in particular for thermosetting composites, such as resin transfer molding, vacuum infusion, pultrusion, filament winding, braiding, hand lay-up, etc. In order to have a reliable product after the manufacturing process in terms of mechanical strength and failure, the process induced residual stresses have to be well characterized since they may alter the internal stress level during the service loading or introduce premature cracks before the service loading [1-3].

The main mechanisms generating the process induced stresses and shape distortions in composite manufacturing are [3] i) Different coefficient of thermal expansion (CTE) for the thermosetting resin and fiber reinforcements, ii) Mismatch in the ply-level CTEs, iii) Chemical shrinkage of the resin system during curing, iv) The through-thickness temperature and cure degree gradients inside the composite and v) The interaction between the tool and the manufactured part. In order to address these challenges in composite manufacturing processes, a numerical process simulation tool is necessary since a satisfactory process experiments take considerable time which is not a cost efficient method. On the other hand, the validation of the results coming from the

simulation tool is essential to gain trust, enhance the understanding and further the usability of the predicted results. There have been several studies presented in literature for the numerical modelling of residual stresses and shape distortions during composite manufacturing process [4-8].

In addition to the computational analyses, there are various experimental techniques available to quantify the residual stresses which can be categorized as: i) non-destructive testing including X-ray diffraction [9], and photoelastic stress analysis or measurement of shape distortions, ii) semi-destructive testing including the hole drilling techniques [10] and iii) destructive testing such as the contour method [11].

In the present work, an epoxy/steel specimen is considered to investigate the residual stress state in the epoxy both experimentally and numerically. The aim is to validate the numerical process model in terms of residual strains and subsequently use the model in the future for more complex simulations on industrial applications.

## Experiments

The residual stress specimens consist of a neat epoxy (Bisphenol-A and an Amine curing agent) and a steel reinforcing plate. A schematic view of the samples is depicted in Fig. 1(*left*). The residual stresses and strains start taking place when a perfect bonding prevails at the epoxy-steel interface during curing. Here, the gauge area indicates the region in which the residual stresses are built up during the process. The steel plate was submerged at the middle of the specimen using custom made spacers to obtain an equal thickness of the resin on each side of the 1mm thick steel plate. The total thickness of the epoxy/steel sample was approximately 3 mm. The specimens were cured in an oven using an elevated temperature profile.

A hole with 5 mm diameter was drilled at the center of the specimen after the curing process to measure the residual strains release associated with the induced stresses [12]. A low rotational speed of the drilling tool was chosen (300 rev/min) in order to avoid the introduction of additional stresses during the drilling process. The DIC technique was utilized to measure the released residual strain at the gauge area due to the hole drilling. For this purpose, the commercial DIC system Aramis from Gom was employed. Firstly, a reference image was captured of the sample before hole drilling. Then a second image was captured after hole drilling (denoted the deformed image) and the two images were correlated. The deformation of the pixels were tracked and the 2D strain field was generated. The strain release associated with the hole drilling is shown in Fig. 1(*right*) in a Cartesian coordinate system. The released radial strain (correspond to $\varepsilon_{xx}$ in the x-direction) is negative in magnitude and decay with the distance from the hole and approach a zero strain release. The corresponding tangential strains are positive and the magnitude decays toward zero with increasing distance from the hole.

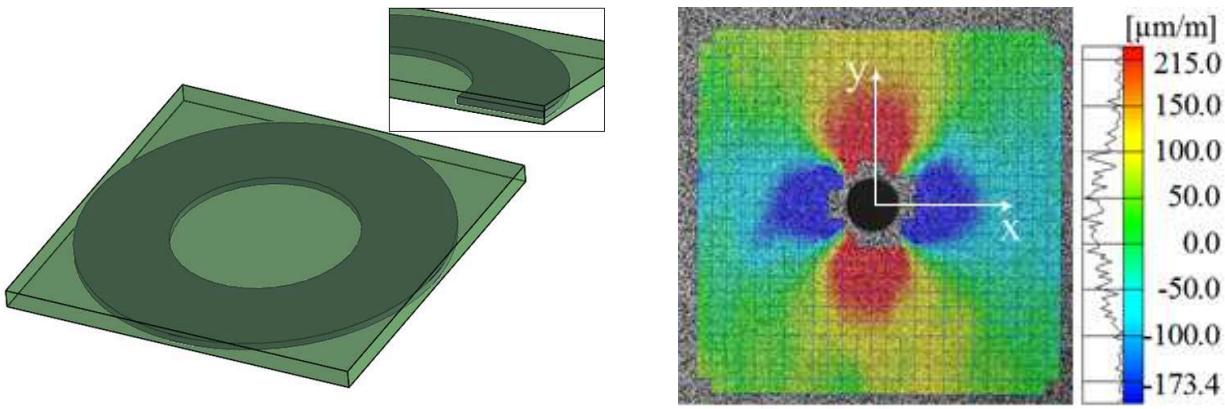


Figure 1. Picture of the epoxy/steel specimens (*left*) and the released residual strains ($\varepsilon_{xx}$) after hole drilling (*right*) [12].

## Numerical Implementation

**Thermokinetics and curing.** The temperature and the cure degree fields were calculated in a transient heat transfer analysis using the general purpose finite element package ABAQUS. The 3D transient energy equations for the epoxy resin and steel plate were solved using the finite element method. The nonlinear internal heat generation together with the resin kinetics of the epoxy were coupled with the heat transfer in an explicit manner in order to obtain a straightforward and fast numerical procedure in ABAQUS. The details of the thermo-chemical analysis can be found in [4-6]. The cure kinetics model specifically developed for the utilized epoxy resin in [13] was employed in the present work.

**Elastic modulus.** A temperature- and cure-dependent elastic modulus was defined from a series of DMA measurements of neat epoxy samples [14]. One measurement of a fully cured epoxy sample heated from room temperature to 120C. An additional sample where tested at isothermal conditions but for the entire conversion range (liquid-solid). This was done by encapsulating the sample in a flexible silicone tube and subjects it to a 3 point bending loading condition. The test results from the later test were only the qualitatively measurements but scaled to meet the properties of the first test to obtain quantitative measures for the Young's modulus. A modified cure hardening instantaneous linear elastic (CHILE) model was developed in the present work. Similar models have been utilized in the literature [4-6, 8].

The evolution of glass transition temperature ($T_g$) with the degree of cure was modelled considering the experimental data from thermo-mechanical analysis (TMA) [14]. Partially cured samples of neat epoxy with a known degree of cure were analyzed in a TMA using several temperature scans through the current $T_g$ of the sample. The cure kinetic model presented in [13] was integrated to convert the TMA data from time domain to degree of cure domain such a $T_g$ vs. degree of cure relation can be established. The corresponding relation was expressed in [14]. A fitting analysis was performed to obtain the constants in $T_g$ eqution in [14] which gave the best agreement with the measured modulus data.

**Thermal expansion and chemical shrinkage.** The coefficient of thermal expansion (CTE) of the epoxy resin was measured using a modulated TMA as reported in [13]. The CTE at material temperatures above the current material glass transition (rubbery state) was measured to be approximately 175 μm/(mºC) which was approximately 2.5 times of the measured CTE in glass state 76 μm/(mºC). The total volumetric shrinkage of the resin sample was measured to be 1.5%. It should be noted that there is almost no dimensional change up to gelation point at which the degree of cure is approximately 0.61 as reported in [13].

## Process Model

The curing process of the residual stress specimen and the hole drilling process were modelled using general purpose finite element package ABAQUS. 1/8 model was considered due to the symmetric thermal and mechanical boundary conditions. A schematic view of the model is depicted in Fig. 2. A fully coupled three dimensional (3D) thermo-chemical-mechanical model was developed to predict the evolution of residual stresses and strains together with the temperature and degree of cure history of the epoxy resin. After the thermo-chemical-mechanical analysis, a hole drilling simulation was sequentially coupled with the curing model. The hole drilling process was simulated by simply removing the elements at the hole drilling region (see Fig. 2(b)) after the curing simulation ended. This provides an initial stress condition and hence a quasi-static analysis was performed without using the elements defined at hole drilling region where any mechanical boundary condition was applied.

A perfect bonding was considered at the epoxy-steel interface. However, the initiation of the bonding was defined according to the vitrification (glass transition at cured state) point of the epoxy resin such that when $T_g > T + T_{comp}$ there is a perfect bond defined between epoxy and steel. Here, $T_{comp}$ was defined as the critical temperature difference at the completion of the vitrification for

perfect bonding and $T$ is the instantaneous temperature of the epoxy. On the other hand, when $T_g < T + T_{comp}$, the resin is in the rubbery or viscous state and hence it is assumed that there is hardly a bonding at the interface which yields a stress free situation. Therefore, it is assumed that the thermal and chemical shrinkage strains were set to 0 for both resin and steel plate when $T_g < T + T_{comp}$ in order to satisfy the stress free condition since there is no bonding at the interface. A parameter study was carried out based on $T_{comp}$ since it has been not known exactly when a perfect bonding is obtained at the interface.

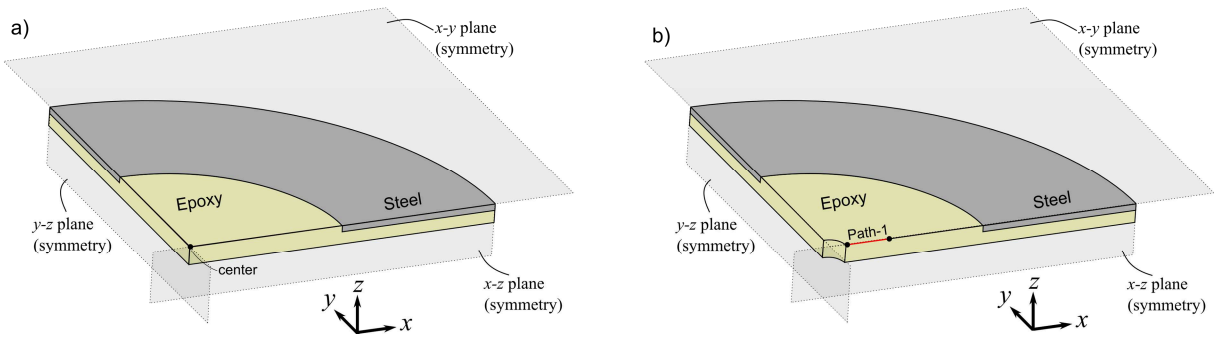


Figure 2. Schematic view of the epoxy/steel specimen used in the thermo-chemical-mechanical model (a) and hole drilling model (b). Note that the geometry is not scaled.

## Results and Discussions

The temperature and degree of cure profiles were calculated using the proposed thermo-chemical-mechanical model in ABAQUS. The results are depicted in Fig. 3 at the center of the specimen. The evolution of the glass transition temperature is also presented in Fig. 3(a). Here, the completion of the glass transition is represented by $T_{comp}$, as aforementioned. It is seen that $T_g$ crosses the resin temperature (66ºC) at approximately 530 min. The degree of cure was found to be approximately 0.94 at 530 min. and it is seen that the curing reaction is almost ended before $T_g$ crosses the epoxy temperature.

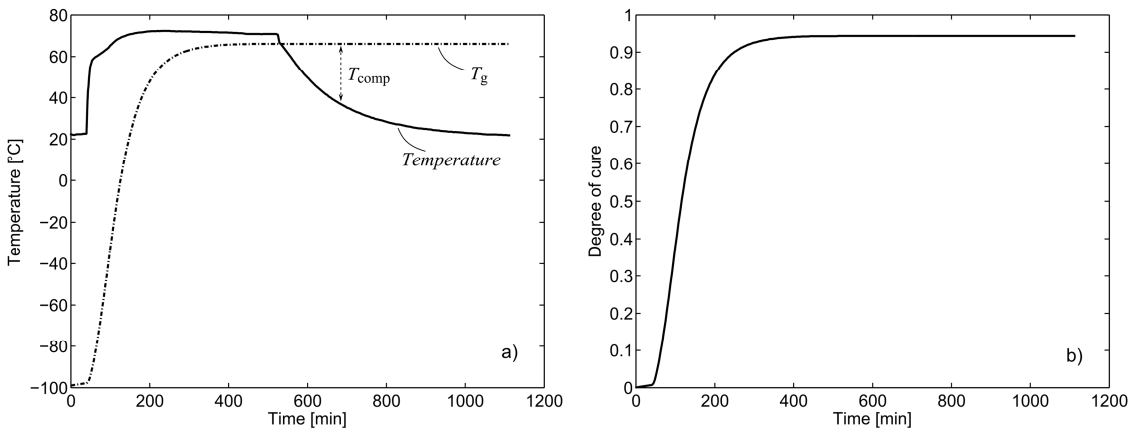


Figure 3. The predicted temperature (a) and degree of cure (b) profiles of the epoxy resin at the center of the specimen.

The calculated strain evolutions on 'Path-1' (see Fig. 2(b)) are shown in Fig. 4 (*left*) for different $T_{comp}$ values together with the measured strain using the DIC. It is seen that the strain level is getting smaller when a higher $T_{comp}$ value is utilized. This is an expected result since the bonding at epoxy-steel interface depends on $T_{comp}$ as aforementioned such that the bonding shiftsn time when $T_{comp}$ increases. Therefore, there is less time for strain built up when $T_{comp}$ increases yielding a lower strain level. It is found that the predicted strain evolution on 'Path-1' (see Fig. 2(b)) agrees

quite well with the measured strain using $T_{comp}$ = 30ºC. It should be noted that the residual strain level obtained from the curing process was subtracted from the strain level after the drilling in order to be in the same line with the DIC measurements since the relative deformation was captured in DIC.

The in-plane residual stress level is found to be approximately 11 MPa after curing process for $T_{comp}$ = 0ºC. The stress at r = 2.5 mm (the drilling surface) become relatively smaller after the hole drilling since there is no mechanical boundary condition defined at the drilling surface. The evolution of the stress at center of the specimen (see Fig. 2(a)) is depicted in Fig. 4 (*right*) for various $T_{comp}$ values. The stress starts increasing when the bonding at the epoxy-steel interface is activated based on the criteria $T_g > T + T_{comp}$. It is seen that the initiation of the stress shifts in time and the stress level decreases when a higher $T_{comp}$ value is used. The residual stress levels were found to be approximately 9 MPa, 7 MPa and 4 MPa for $T_{comp}$ = 10ºC, 20ºC and 30ºC, respectively, after the curing process.

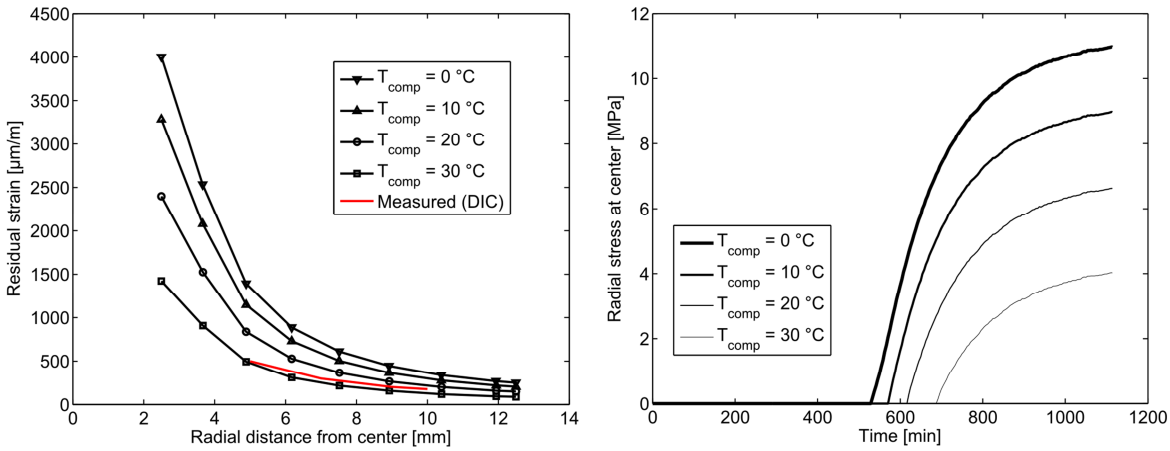


Figure 4. The radial strain evolution in magnitude on 'Path-1' for different $T_{comp}$ values (*left*). The evolution of the in-plane stresses at the center of the specimen during curing (*right*).

## Conclusions

An epoxy/steel specimen was utilized to investigate the residual stresses and strains experimentally and numerically. A detailed material characterization analysis was conducted for the epoxy resin. The curing kinetics model was obtained using the DSC. The temperature- and cure-dependent resin elastic modulus model was developed according to the DMA measurements. The glass transition temperature, CTEs and chemical shrinkage characteristics were obtained using the TMA. After the samples were cured, a 5mm hole was drilled at the center of the specimen. The DIC technique was employed to measure the released strain after the drilling. A strain decay was found to prevail in the gauge region starting from the drilling surface. A parameter analysis was carried using the proposed thermo-chemical-mechanical process model based on various $T_{comp}$ values since the time for having a proper bonding between the epoxy and steel has not been known exactly. After the process model, the elements at the drilling region were removed and subsequently a quasi-static mechanical analysis was conducted in which the predicted residual stress state was utilized as an initial stress state. The released strain as well as the strain decay observed via DIC were predicted. The strain level was found to match well with the measured one for $T_{comp}$ = 30ºC.

The proposed epoxy/steel specimen as well as the measurement techniques was found to be relatively simple and useful for validating the numerical process models in terms of residual stresses and strains. However, a more advanced experimental set-up would be need to specify when the bonding occurs at the epoxy/steel interface.

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**References**

[1] I. Baran, C.C. Tutum, J.H. Hattel, R. Akkerman, Pultrusion of a vertical axis wind turbine blade part-I: 3D thermo-chemical process simulation, Int J Mat Form (2014). DOI: 10.1007/s12289-014-1179-6.

[2] I. Baran, J.H. Hattel, C.C. Tutum, R. Akkerman, Pultrusion of a vertical axis wind turbine blade part-ii: combining the manufacturing process simulation with a subsequent loading scenario, Int J Mat Form (2014). DOI: 10.1007/s12289-014-1178-7.

[3] M.R. Wisnom, M. Gigliotti, N. Ersoy, M. Campbell, K.D. Potter, Mechanisms generating residual stresses and distortion during manufacture of polymer-matrix composite structures, Compos Part A-Appl S 37 (2006) 522-529.

[4] I. Baran, C.C. Tutum, M.W. Nielsen, J.H. Hattel, Process induced residual stresses and distortions in pultrusion, Compos Part B: Eng 51 (2013), 148-161.

[5] I. Baran, R. Akkerman, J.H. Hattel, Modelling the pultrusion process of an industrial L-shaped composite profile, Compos. Struct 118 (2014), 37-48.

[6] I. Baran, J.H. Hattel, R. Akkerman, Investigation of process induced warpage for pultrusion of a rectangular hollow profile, Compos Part B: Eng 68 (2015) 365-374.

[7] T.A. Bogetti, Jr.J.W. Gillespie, Process-induced stress and deformation in thick section thermoset composite laminates, J Compos Mater 26(5) (1992) 626-660.

[8] A. Johnston, R. Vaziri, A. Poursartip, A plane strain model for process-induced deformation of laminated composite structures, J Compos Mater, 35(16) (2001) 1435-1469.

[9] B. Benedikt, M. Kumosa, P. Predecki, et al., An analysis of residual thermal stresses in a unidirectional graphite/PMR-15 composite based on X-ray diffraction measurements. Compos Sci Technol 61(14) (2001) 1977-1994.

[10] C. Prasad, R. Prabhakaran, S. Tompkins, Determination of calibration constants for the hole drilling residual stress measurement technique applied to orthotropic composites - part II: experimental evaluations. Compos Struct 8(3) (1987) 165-172.

[11] M.B. Prime, Cross-sectional mapping of residual stresses by measuring the surface contour after a cut, J Eng Mater Technol 123(2) (2001) 162-168.

[12]J. Jakobsen, J. H. Andreasen, M. Jensen, A novel biaxial specimen for inducing residual stresses in thermoset polymers and fibre composite material, J Compos Mat (2014). DOI: 10.1177/0021998314554120

[13] J. Jakobsen, M. Jensen, J.H. Andreasen, Thermo-mechanical characterisation of in-plane properties for CSM E-glass epoxy polymer composite materials – Part 1: Thermal and chemical strain, Polymer Testing 32 (2013) 1350-1357.

[14] J. Jakobsen, M. Jensen, J.H. Andreasen, Thermo-mechanical characterisation of in-plane properties for CSM E-glass epoxy polymer composite materials – Part 2: Young's modulus, Polymer Testing 32 (2013) 1417-1422.