



# In-Situ Monitoring of the Manufacturing Process and Residual Stress Evolution in Thin-Ply Composites

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**Thin-ply composite laminates are of interest for several applications in aerospace and other high-performance industries due to their ability to delay transverse microcracking and delamination in static, fatigue, and impact loadings. It is essential to understand the evolution of thermal residual stresses during cure to optimize the manufacturing process of thin-ply composites for deep-space applications. In this research, processing induced residual stresses in thin-ply laminates are evaluated by devising a novel in-situ experimental approach. Thin-ply prepreg laminates are cured in a specially designed autoclave with viewports with plies laid upon a flat tool and a curved tool. The curved tool configuration used in this research is designed to simulate cryogenic fuel tank surfaces. The evolution of residual stresses in terms of out-of-plane displacement is characterized using Digital Image Correlation (DIC) during the autoclave cure cycle.**

## I. Introduction

Carbon fiber prepreg composites have been widely used for structural applications. Lightweight, high specific strength, resistance to corrosion and flexibility in design, etc. are some of the properties displayed by these materials that have benefited many industries such as aerospace, automotive, and marine for several decades. More recently, these composites have become a mainstay of the aerospace industry. For example, commercial aircraft such as Boeing 787 and Airbus 380 feature composites in the fuselage and other primary structures [1]. Despite these benefits, the susceptibility of composite materials to failure modes such as delamination and micro-cracking creates a major concern related to structural integrity [2]. Delaying or even suppressing these failures will incentivize the replacement of metallic structures with carbon fiber.

In recent years, thin-ply composites, i.e. composites made of thinner plies are commercially available down to about 20 $\mu$ m per ply depending on the type of fiber. Several experimental studies have shown that thin-ply composites can delay, and in some cases even suppress transverse microcracking and delamination in static, fatigue, and impact loadings [3–5]. Another benefit of using thinner plies in a given structure, and of constant laminate thickness, is the design freedom to use a larger number of ply orientations to achieve an optimal solution in the same thickness [6]. Reducing ply thickness has been shown to improve mechanical properties due to low void volume fractions because of high fiber dispersion with homogeneous microstructures in thinner plies. The higher onset of damage and strength in plain tension [7], higher fatigue life [6], and higher bearing strength in bolted assemblies [8] are achieved by using thin-ply composites. The impact [9], tension after the impact [10], and compression after impact [11] performance of thin-ply laminates have been examined in the literature. Thin-ply laminates typically exhibit increases of 10-20% in compression and tension after impact strengths, compared with standard thickness laminates [12]. Researchers have studied damage mechanisms resulting from impact and other out-of-plane loads in thin-ply composites [13]. Yokozeki et al. investigated the damage characteristics of carbon fiber/toughened epoxy thin-ply laminates subjected to transverse loadings and observed that the accumulated delamination position has a significant effect on the fiber fractures [5]. Häsä and Pinho investigated a metal hybrid/crossed lamellar block with aluminum and titanium for the metal. These structures were able to withstand very large applied curvatures upon bending compared to other hybrid

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CFRPs and quasi-isotropic CFRPs [14]. These advantages of thin-ply composites over standard composites make them attractive for several applications in both aeronautics and space.

One of the interesting applications of thin-ply composites is for the habitation systems or cryogenic tanks aboard space vehicles. Resistance to microcracking and high structural efficiency makes thin-ply composites an ideal material for designing pressurized structures like cryogenic tanks. Cryogenic tanks are extensively used on spacecraft, rockets, and space vehicles. Those tanks experience boil-off as the stored propellant in liquid state evaporates. Such a phenomenon is undesirable as it causes propellant loss through venting to the surrounding environment to prevent tank burst at high storage pressure. Cryogenic tank design relies on two main factors; those are its structural integrity to withstand internal and external loads as well as its isolative and cooling ability to keep the propellant in the liquid state. Laminated composite materials have been recently used in aerospace propellant tanks for their attractive strength and stiffness to weight ratio and their low thermal expansion coefficient in the development of cryo tank technologies compared to metallic structural materials [15]. Thick ply stitched, textile and laminated configurations encounter delamination and micro-cracking failure at the boundaries of their differently oriented layers leading to leakage in pressurized tanks [16]. As a conventional solution, inner metal liners are inserted at the interface with the cryogenic propellant. Such liners pose fabrication and maintenance hitches. Also, the difference in material constitution between the metallic liner and the wrapping composite material emphasizes debonding. Thin-ply laminates emerged to address these issues.

Using thin-ply composites that suppress the residual stresses and thereby eliminate the need for metal liners to minimize diffusion through the walls is an ideal solution for cryogenic fuel tanks. To do that, it is necessary to understand the evolution of thermal residual stresses in thin-ply composites during processing as they can have deleterious effects on the structural integrity and dimensional stability. Residual stresses are typically measured through the deformations raised in a cured pre-stressed laminate after the removal of the stress [17]. Commonly used methods include layer removal [18], hole-drilling [19], and incremental slitting [20]. Most of these studies are limited to post-manufacturing examinations. In-autoclave and in-situ studies from literature are limited due to the experimental requirements of incorporating a monitoring setup inside an autoclave. To address these constraints, a novel in-situ experimental approach is developed in this research. To identify the conditions that cause the thermal residual stresses during manufacturing, a custom designed autoclave with borosilicate glass viewports is incorporated with a 3D Digital Image Correlation (DIC) camera setup to capture pictures of the composite prepreg laminate while curing. The out of plane ply-movement is quantified through the cure cycle. The objective of this study is to utilize the in-situ experimental approach to investigate how strains and residual stresses evolve during the manufacturing of thin-ply composites.

## II. Experimentation

### A. Materials

Unidirectional thin-ply carbon fiber prepreg was produced at CA Composites using TORAY T700-12K fibers with manufacturer specified tensile modulus of 120 GPa (17.4 Msi) and strength of 1.85 GPa (268 ksi). This prepreg is pre-impregnated with an epoxy resin system of density 1.2 g/cc and has a fabric areal weight of 30 g/m<sup>2</sup>. The manufacturer recommended curing temperature is 120°C (248°F) with a one hour hold time. Stretchlon 800 transparent bagging film from Fibre Glast Corporation is used for vacuum bagging [21].

### B. Experimental Setup

A high temperature (400°C/752°F) and high pressure (1.38 MPa/200 psi) autoclave from ASC Systems is instrumented with 3D digital image correlation (DIC) as shown in Fig. 1(a) is used in this study. This autoclave is equipped with a borosilicate glass viewport as seen in Fig. 1(b). This glass is made of silica and boron trioxide and has very low coefficients of thermal expansion (CTE) which makes the viewports more resistant to thermal shock compared to soda-lime glass [22]. The autoclave is also equipped with an interior light which enables the DIC measurement while the autoclave is operating on a cure cycle (typically 120°C (248°F) and 0.69 MPa (100 psi) pressure) for the composite fabrication. The cure cycle from the manufacturer is converted into a step by step recipe, which is then programmed into composite processing control (CPC) of the autoclave computer. CPC constantly monitors the cure parameters of the cycle throughout the cure and makes changes dynamically if needed.

Digital image correlation is a non-contact optical technique to measure deformations based on image processing and numerical computation. In this research, DIC is used to measure the surface ply deformations and strains during the composite processing. The VIC-3D Real-Time DIC System from Correlated Solutions consists of two 6-megapixel high-resolution monochromatic cameras capturing images of the sample throughout the cure. DIC technique works by tracking the changes in the gray value pattern in small neighborhoods called subsets in the images [23]. A reference

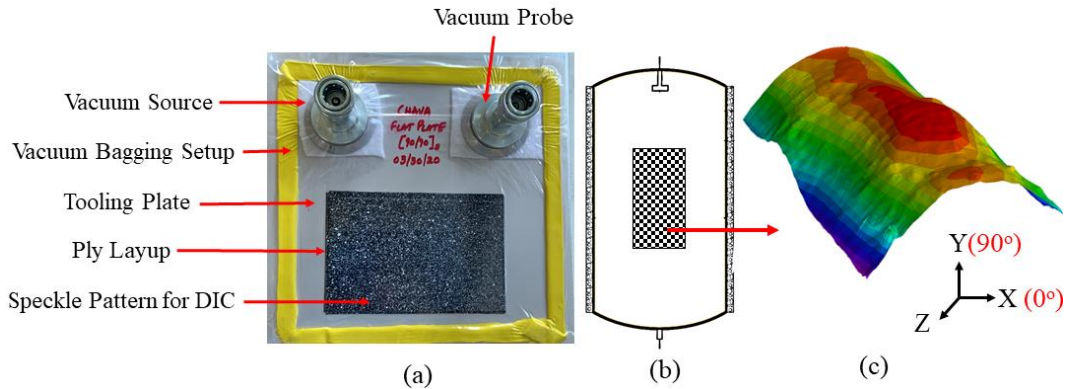
square subset with sufficient intensity variations is selected from the reference image and compared with a deformed subset from the target image. The differences between the reference subset and the target subset yield the subset center's displacement vector. DIC extracts full-field information by either coherent light illumination or through the application of a high contrast random speckle pattern.



**Fig. 1 (a) Autoclave with 3d DIC. (b) Autoclave viewports and DIC cameras**

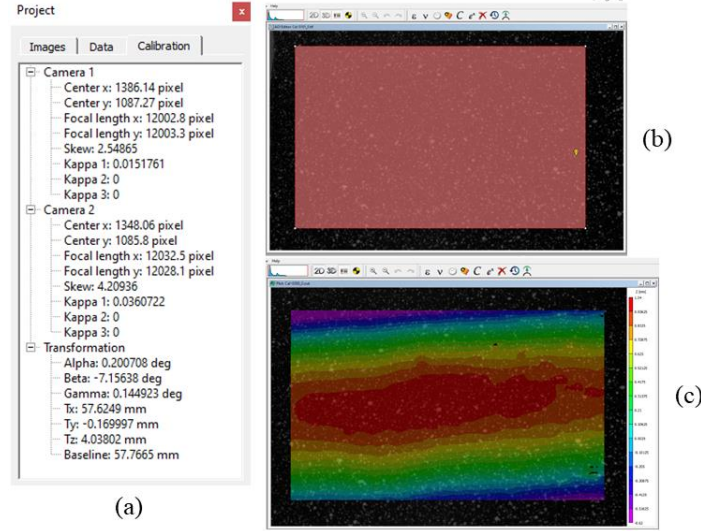
### C. Experimental Procedure

Composite specimens of dimensions 203.2 mm x 152.4 mm (8 inch x 6 inch) are prepared for fabrication using four plies of unidirectional thin-ply carbon fiber prepreg with ply orientation of  $[90/90]_s$  laid upon a flat mold and a curved mold. A white speckle pattern is sprayed on the top layer of the layup as shown in Fig. 2(a). This creates a random and high contrast pattern for digital image correlation analysis. The curved mold configuration used in this research is designed to simulate the surface of a cryogenic fuel tank. Figure 2 (b) and (c) show the schematic of the curved mold location on the tank and DIC analysis of the specimen respectively. The plies are laid upon the mold and prepared for cure using the vacuum bagging technique as seen in Fig. 2(a). The manufacturer provided cure cycle is programmed into the autoclave. It consists of a ramp-up to 120° C (248° F) at 5° C/min and a pressure of 0.69 MPa (100 psi) for a hold time of 1hr followed by a ramp down.



**Fig. 2 (a) Vacuum bagging of the flat sample. (b) Cryogenic tank schematic. (c) Analysis of curved sample in 3D**

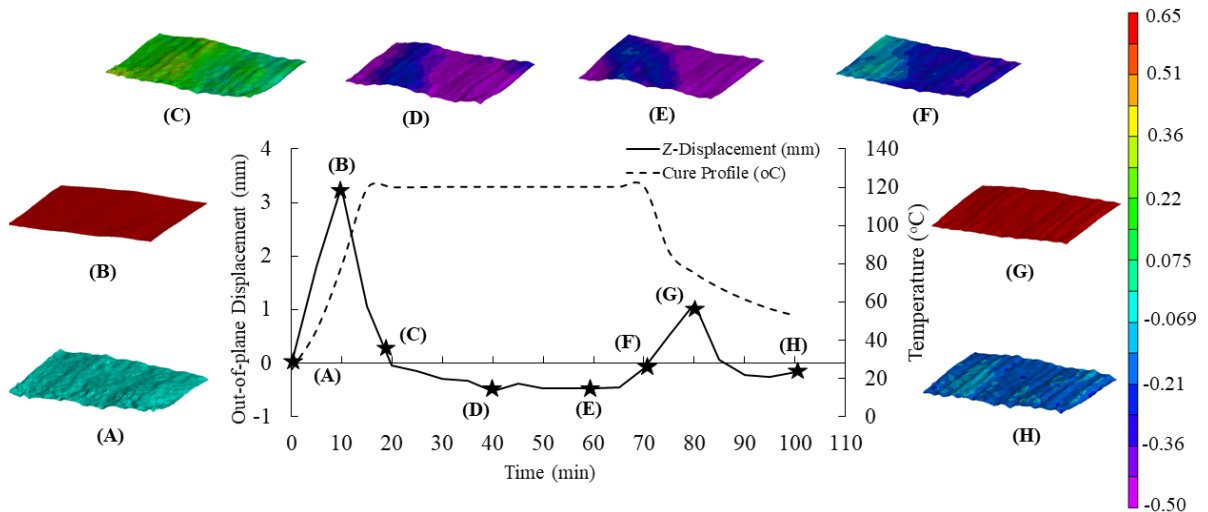
The cameras of the DIC system are set up pointing at the composite layup through the viewport of the autoclave. Calibrating the DIC system helps the cameras identify their location in space and the angles between them (see Fig. 3(a)). After transferring the layup to the autoclave both the autoclave and the DIC are started at the same time capturing pictures throughout the cure cycle. Due to the formation of thermal residual stresses inside the specimen, there will be a relative movement in the speckles on the top layer of the layup. This relative movement of the speckle pattern over the region of interest (ROI) is analyzed in VIC-3D software to determine the surface displacements as seen in Fig. 3(b) & 3(c). The results of this experimental approach provide an insight into the development of residual stresses during the processing. Further, it can be used to quantify the process conditions (temperature, pressure, cure cycle) and the tool geometry to decrease the effect of residual stresses.



**Fig. 3 (a) Snapshot of DIC calibration. (b) Specimen before analysis. (c) Specimen after analysis in 2D**

### III. Results & Discussion

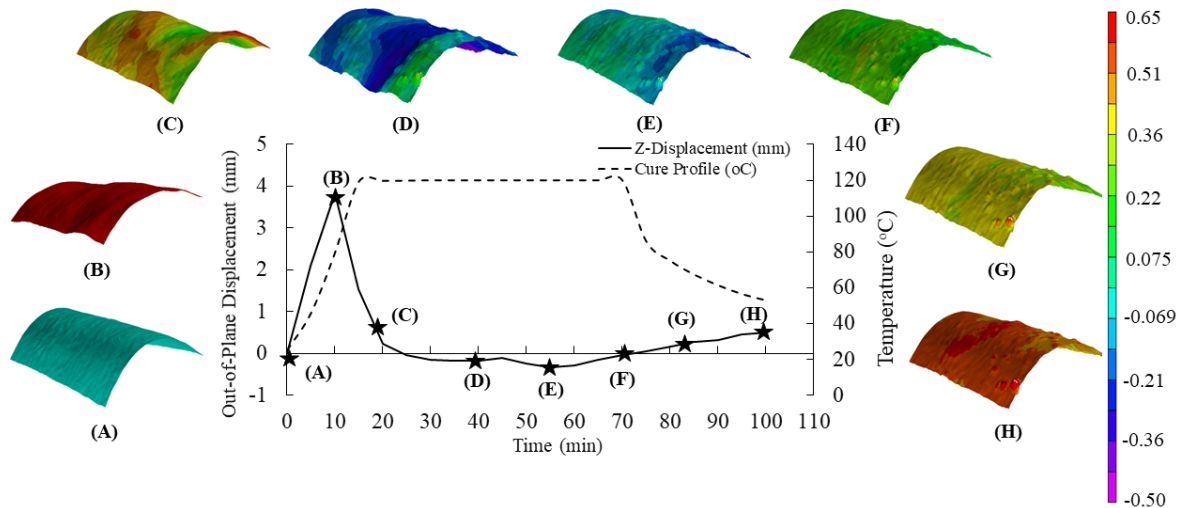
The pictures obtained from the DIC cameras for two different molds (flat and curved) are analyzed in VIC-3D software for change in displacements in the out-of-plane Z direction. These displacements are the result of the interfacial ply movement happening due to the combination of the thermal loads during the cure as well as residual stresses generated due to the crosslinking of the polymer bonds [24]. These results from the DIC are correlated to the cure cycle temperature profile of the thin-ply carbon fiber composite plotted against time. The cure profile, average out-of-plane (Z) displacement, and respective displacement contours for a four-ply laminate with ply orientation of  $[90/90]_s$  over a flat mold and a cylindrical mold can be seen in Fig. 4 and Fig. 5 respectively. The sample in Fig. 5 is laid upon a curved mold to simulate the curvature of a cryogenic fuel tank. The dotted line in Fig. 4 and 5 represents the cure profile of the thin-ply composite material and the solid line represents the average displacement in the Z (out-of-plane) direction. The corresponding contour plots at multiple points throughout the cure show the distribution of the average displacement in the region of interest.



**Fig. 4 Average displacement (Z) of flat sample and respective contours in the region of interest**

A maximum displacement of 3.2 mm is observed for the sample over flat mold during the ramp-up phase of the cure. This displacement is due to the thermal expansion of the matrix and fibers as well as the readjustment of plies as temperature increases rapidly to cure temperature. The initial displacement gradually decreases during the hold-phase of the cure and the laminate observes negative z-displacement in this phase due to cure shrinkage. This

phenomenon is happening for samples over both the molds as seen in Fig. 4 and 5. The resin reaches the vitrification point, the point where the resin changes from a rubbery state to a glassy state, at the end of the hold-phase. After vitrification, the displacement for the flat mold increases to 1.11 mm during the cool-down phase as the motion of plies is restricted post vitrification towards the end of the cure. This results in the formation of thermal residual stresses during the cool-down phase. Finally, at the end of the cure, it can be observed from Fig. 4 (H) that the average movement is -0.16 mm. The negative sign implies the shrinkage of thickness in the Z-direction.



**Fig. 5 Average displacement (Z) of curved sample and respective contours in the region of interest**

On the other hand, a similar trend is observed for the sample over curved mold as seen in Fig. 5 during the ramp-up and hold phase of the cure. The displacement is high during the ramp-up temperatures reaching an average of 3.8 mm and decreases during the hold-phase of the cure. A negative displacement can be seen in this phase due to cure shrinkage of the prepreg. The combination of the glassy matrix after vitrification and the thermal relaxation of the matrix and fibers during the cool-down phase creates residual stresses when the laminate is cooled to ambient temperatures. This can be observed in the sample as the displacement at the end of the cure due to the development of residual stresses increases to 0.49 mm as seen in Fig. 5 (H). The contours respective to the points A to H in Fig. 5 show the average displacement of the sample over the region of interest. The difference in displacement at the end of the cure for the flat thin-ply sample and the curved thin-ply sample is due to the difference in residual stresses developed in these samples during the cure. Due to the pre-stressed nature of the curved thin-ply sample, the average displacement and the residual stresses in the thin-ply laminate at the end of the cure are higher compared to the flat thin-ply sample.

Understanding the development of residual stresses in curved composite parts is especially important as more and more parts made of composite materials are used in the next generation of rockets and spacecraft such as a pressurized, cryogenic propellant tank for space exploration [25]. Residual stresses in composite materials can be measured by destructive, semi-destructive, and non-destructive techniques. In general, these methods can be categorized as mechanical, optical, diffraction, and stress-relevant properties methods. Among different measurement techniques for measuring the residual stresses, mechanical techniques are most often used in literature as they are easier compared to other techniques. Most of the mechanical techniques are destructive such as the layer removal method [26], milling outer plies [27], etc. Applying these techniques to thin-ply composite laminates is challenging. In addition to the destructive techniques, non-destructive methods like x-ray diffraction [28], neutron diffraction [29], have also been used for measuring the residual stresses. However, requirements like crystallinity limit the application of x-ray diffraction and neutron diffraction methods for carbon fiber composites [30]. The novel non-destructive in-situ method for monitoring processing induced displacements and related residual stresses developed in this research overcomes the existing challenges and can also be applied to thin-ply composites as shown in Fig. 4 and 5. This experimental approach can be utilized to understand how various processing parameters affect the manufacturing of thin-ply composite parts and approaches can be devised to adjust the cure parameters such as cure temperature and pressure to minimize the processing induced residual stresses.



#### IV. Summary

In summary, two mold configurations, a flat and a curved mold, were developed to prepare thin-ply composite laminates. A novel in-situ experimental approach is developed to identify the conditions that cause thermal residual stresses during the manufacturing of these thin-ply composite laminates. A specially designed autoclave with borosilicate glass viewports is incorporated with a 3D Digital Image Correlation (DIC) camera setup to capture pictures of the composite prepreg laminate while curing. The out of plane ply-movement is quantified through the cure cycle. Residual stresses developed during the cool-down phase of the cure are correlated to the out-of-plane ply displacement. The research approach developed in this paper can be used to improve the manufacturing process of thin-ply composite parts by developing approaches that minimize processing induced residual stresses.

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