ROUTES TO MESOSTRUCTURE CHARACTERIZATION OF COMPOSITES USING DISCONTINUOUS PREPREG AND MECHANICAL PERFORMANCE

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Abstract

Chopped carbon fiber platelet-based epoxy infused pre-preg material as Sheet Molding Compound is used in this work for rapid compression molding of high-volume and complex automotive parts. These composites demonstrate impressive mechanical performance of near 40 GPa tensile modulus and 300 MPa strength based on coupon samples extracted from compression molded flat plaques. The work described herein is targeted at evaluating the microstructure of this new material system both qualitatively and quantitatively in three dimensions. Physically important quantities for fiber reinforced composites corresponding to spatially varying platelet (chopped fiber bundles) orientation and fiber volume fraction are evaluated using advanced characterization methods including non-invasive X-ray micro-computed tomography, optical microscopy, and a new procedure developed by the authors for this material system, Thermal Digital Image correlation (TDIC). Understanding the microstructure at multiple length scales and the process to property conditions through which certain performance criteria are met is the objective for this research. Such detailed material science will lead to tailored processing conditions for a targeted complex automotive component without the need for multiple characterization studies for molded parts of varying size and complexity. To probe the microstructure and performance of this epoxy and carbon fiber-based platelet material system, 100 mm x 12 mm coupons were extracted from 300 mm x 300 mm flat plaques and from multiple flat locations of a molded double dome geometry (a component with very complex shape). Due to the flow of reinforced epoxy platelet-based charge material during compression molding, significant microstructural changes occur spatially for the double dome part that are not present in the flat plaque geometries. Regions of high platelet orientation normal to loading direction and regions with low fiber volume fraction resulting from material flow during compression molding provide lower bound properties in terms of tensile modulus and strength. The morphology of intact platelet structure that was observed for simply geometrical shape corresponding to a flat plaque (resulting in very strong tensile properties) did not translate for complex shaped compression molded parts such as a double dome. This important insight will help provide the research team with a path to optimize mechanical properties of complex shaped components from chopped carbon fiber based platelet charge and its optimization.

Background/Introduction

Carbon fiber composites offer tremendous application potential for transportation materials due to their excellent specific strength and modulus, improving energy efficiency. The most common limitation regarding the implementation of composite materials is cost, which includes both the acquisition of raw materials and the manufacturing these into usable parts. Recently, developments in manufacturing techniques have demonstrated significant improvements in the processability of carbon/epoxy based composite systems and increased the application space by developing a rapid manufacturing process for complex part geometries without the need for substantial retrofitting of current infrastructure. The novelty of this manufacturing approach relies on the use of platelet-based carbon SMCs (sheet molding compounds) processed using compression molding techniques to near net shape with minimal post processing rapidly. Substantial effort has been put forth by our collaborative team from Dow Chemical company to demonstrate that sufficient material volumes can be produced to meet the anticipated demand of an OEM. The current paper seeks to demonstrate the validation and performance of parts with complex geometries and the methods of characterization.

The chopped fiber platelet system in development here has been geometrically and chemically optimized for performance and rapid production. The goal of this material system is to compete with other energy efficient and light-weight materials currently being utilized in automotive space, such as aluminum alloys. Target mechanical performance has been selected at 300 MPa failure strength and 40 GPa Young's Modulus in tension. Initial testing using materials extracted from 300 x 300mm flat composite plaques have achieved these targets consistently. Currently, work is being done to determine the primary material parameters necessary for those benchmarks to be achieved in a molded complex part at sufficiently large scale and expected geometric complexities. This paper is focusing on defining spatially the fiber orientation state and developed microstructure from one such complex shaped part that mimics a production part at large length scales and desired manufacturing rapid cycle time.

It is well-established that carbon fibers are mechanically anisotropic with significantly higher modulus along the fiber direction compared to transverse or radial direction. It is also well recognized that failure initiation sites for fiber reinforced composites occur along the fiber matrix interfaces, making the failure strength significantly lower transverse to the fiber direction [1]. Hence, the first area of interrogation for this platelet based composite system would be to determine if the part has preferred orientation or if the platelets are distributed randomly after manufacturing for a given geometry of the part and molding conditions associated charge placement, processing variables, and the amount of flow necessary for successful part filling. It is thought that the initial charge pattern is close to random, but due to pressure generated flow, it is probable that fibers will reorient along the flow direction. When the final material deviates from the orientation present originally with the initial charge, material performance is significantly altered. Three techniques are currently being used to understand the material orientation state: X-Ray Computed Tomography (XCT) with image processing for non-invasively obtained three-dimensional orientation, traditional optical microscopy approach on extracted and polished samples with information limited to small regions of interest and in two-dimensions, and surface based Thermal Digital Image Correlation (TDIC), a new technique developed for this material system by the authors. XCT creates a 3D density of map of the sample to distinguish between matrix and resin phases. However, the density between the two phases present in the composite, carbon fiber-based platelets and cured epoxy resin, is small, which limits the achievable contrast for direct segmentation-based measurements. After the data was acquired, the composite processing toolbox in commercially available VGstudio© software was used to map out orientations spatially over a predefined mesh. TDIC is a technique where a sample is thermally loaded below its glass transition temperature, while the surface deformations are monitored. A custom prost-processing of these deformations can then be used to interpret local platelet dominant orientations if present. Lastly, optical high magnification digital microscopy is a destructive technique that uses polished specimens on certain planes under a microscope. Typically, orientations can then be determined by observing the elliptical geometry of round fibers. However, the low-cost carbon fibers used in this work are kidney bean shaped, as shown in Figure 1, and cannot be analyzed with existing published techniques that rely on relating orientation to the elliptical nature of the fiber [2]. Thus, a new technique has been developed by the authors for this study that depends on grey-scale intensity changes driven by the reflective nature of light from rotated planes in carbon fiber using a laser microscope for this study. This technique assumes the fibers orientations are primarily in the plane of the thin part being observed. However, since an accurate cross-section of the fiber is no longer necessary, low mag images can be used increasing the field of view. This technique is readily automated to analyze dense fiber volume fraction with touching fibers and a suitable algorithm was developed for removing matrix/fiber boundaries.

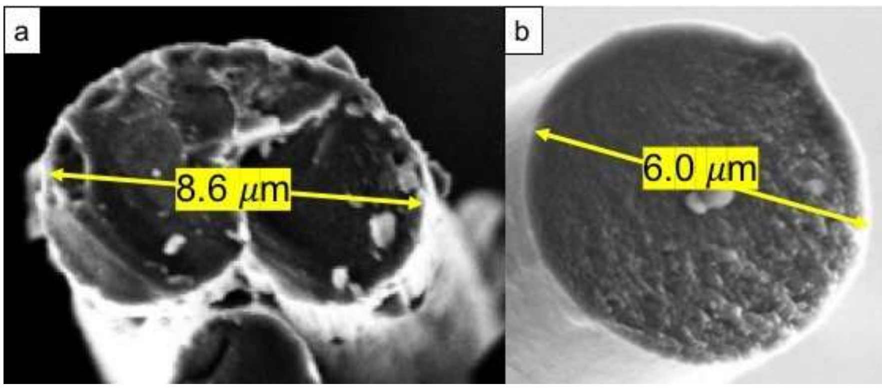

Figure 1 - Comparison of typical (a) Low-cost kidney bean shaped carbon fiber and (b) a reference Toray T700 round carbon fiber.

Presented in this work is the mechanical performance of tensile coupons extracted from molded flat plaques (Figure 2a) and double domes (Figure 2b). These two parts offers a stark difference in the internal development of microstructure as function of molding conditions utilized. The goal is to relate the spatial variation of mechanical properties to the changing internal microstructure induced from the material flow using the tools described herein. A relationship between the evolution of the microstructure under induced flow and the resulting mechanical properties will allow engineers to tailor the charge and molding process to meet performance criteria in critical zones in a predictive fashion.

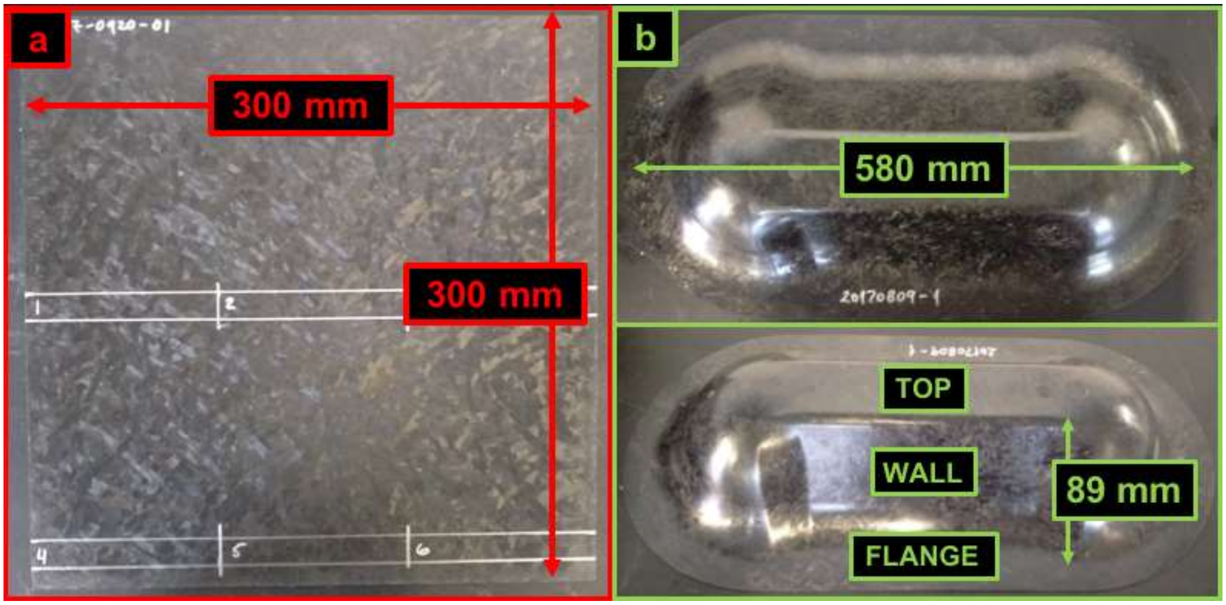

Figure 2 – Example of carbon fiber SMC flat plaque (a) and double dome (b) parts studied in this work. Tensile coupon extraction occurred along the long axis of the dome in three locations: top, wall, and flange.

Results/Discussion

Mechanical Performance

Tensile coupons (100x12mm) were extracted from the flat plaques at three locations from the double dome which allowed for 50.8 mm gauge region and 25.4 mm grip region. The double dome regions included in this paper correspond to locations identified as top, wall, and flange, as shown in Figure 2b. The 100x12mm coupon is smaller than the suggested in ASTM D3039 standard, but in order to study the internal structure at 15-micron voxel resolution for XCT studies prior to mechanical loading non-invasively, a smaller sample was necessary [3]. Two tomography scans were utilized to cover the complete 50mm gauge length at the target resolution which was chosen for several important reasons. The flat plaque tensile tests demonstrated properties on par with the target mechanical performance. The average failure stress and modulus were found to be 300 MPa and 39.0 GPa. However, the mechanical performance for the same material molded in the double dome part with severe geometric complexities demonstrated a large variation in measured modulus and failure strength in tension. A graphical presentation of the general stress/strain behavior is given in Figure 3. This shows that the highest modulus behavior was obtained in the flange and lowest modulus in the wall for those samples extracted from the complex part. All double dome samples, regardless of extraction location, were found to have lower failure stress than the flat plaque.

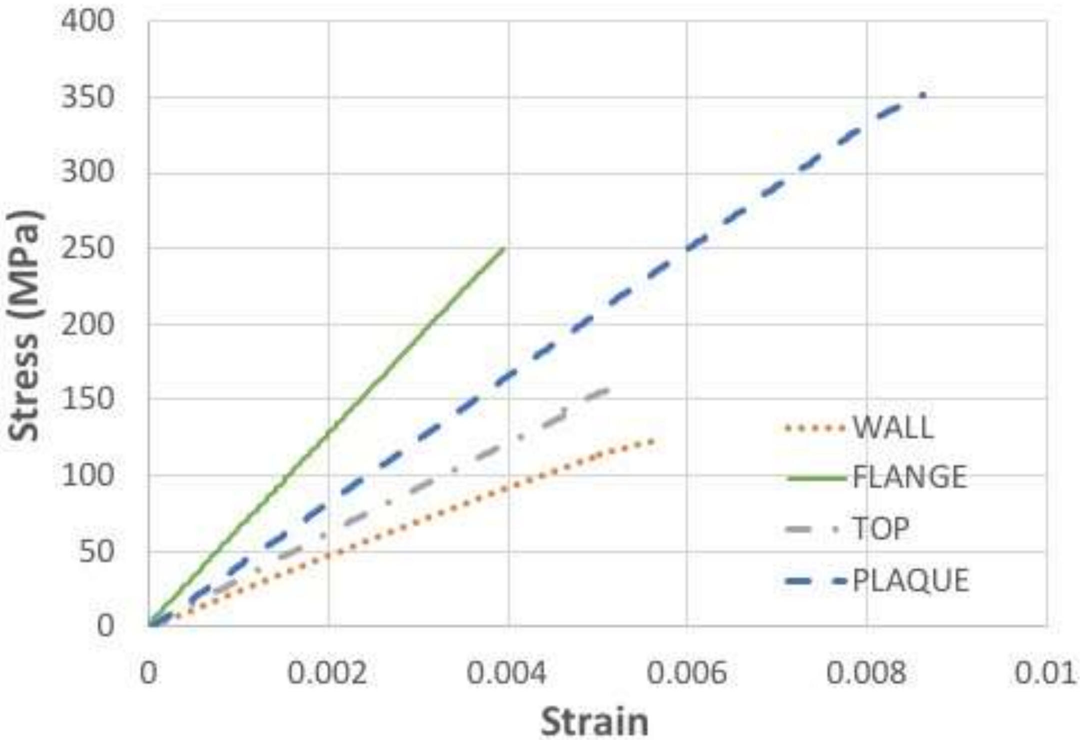

Figure 3 - Stress/strain response of tensile coupons extracted from molded flat plaques and the locations the double dome, demonstrating typical mechanical performance observed for these regions. The greater modulus for the flange and lesser modulus in the wall indicates a reduction in fibers orientation along the tensile axis of the coupon.

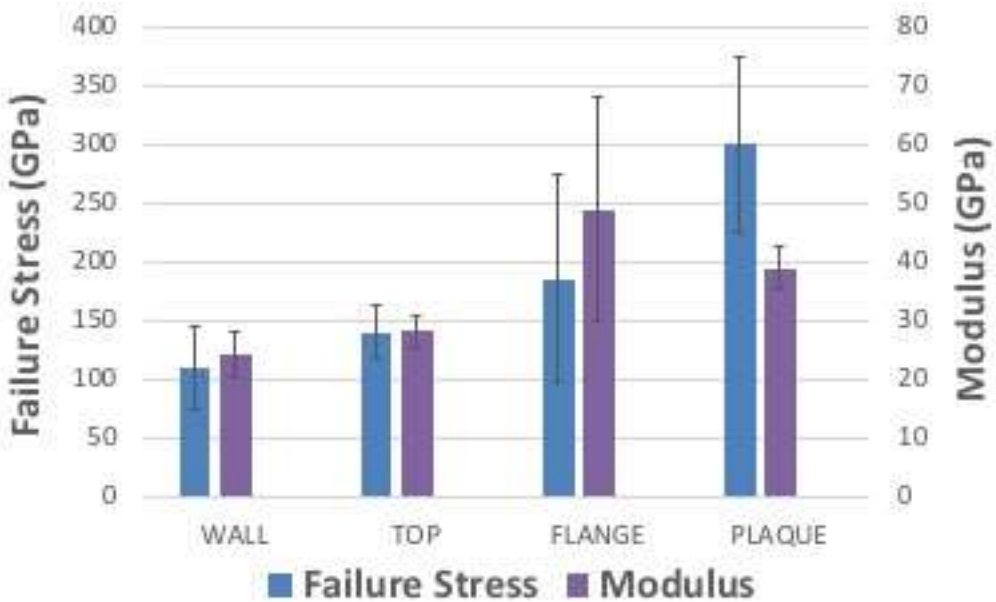

Figure 4 – Average behavior of tensile coupons tested. The highest failure stresses come from the coupons extracted from the plaque parts, which require less material flow to complete the part.

Strain on the tensile coupons was measured using Digital Image Correlation (DIC) through a commercially available software, Vic3D by Correlated Solutions. The surface strain maps obtained demonstrated significant spatial variability as a result of high orientation regions and the density of platelets. Hence, the gage region over which the modulus is measured can have a significant impact on the reported result. For this reason, two points located on both ends of the gage region were selected to be reference points for the optical extensometer, which measures the relative displacement of these points and calculates the engineering strain encompassing the entire sample. A demonstration of high strain region and low strain region and the resulting modulus is given in Figure 5 and Figure 6. These figures show spatial variation of tensile strain in the axial direction along the length of the sample using lower resolution 3D-DIC acquisition conditions. As will be seen later, one needs to acquire DIC images at high resolution for obtaining spatially resolved transverse strains.

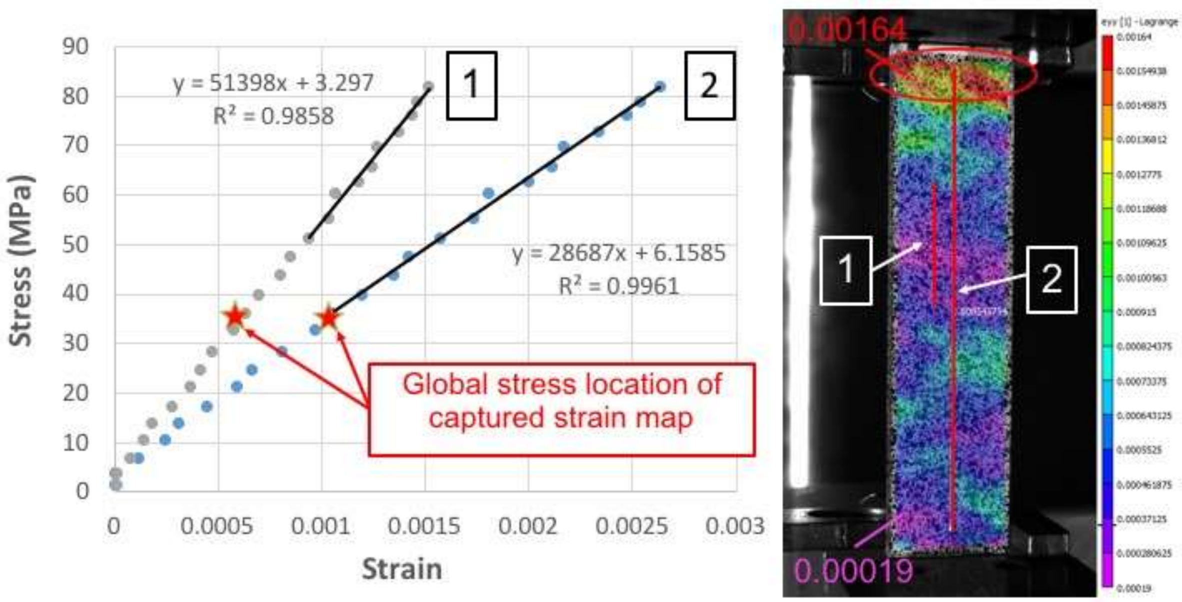

Figure 5 - Stress/strain behavoir of a tensile coupon from the flange section (sample FBD1) of the double dome with the strain map at the starred stress position. A signficant strain concentration where the sample ultimately failed is observed at the top.

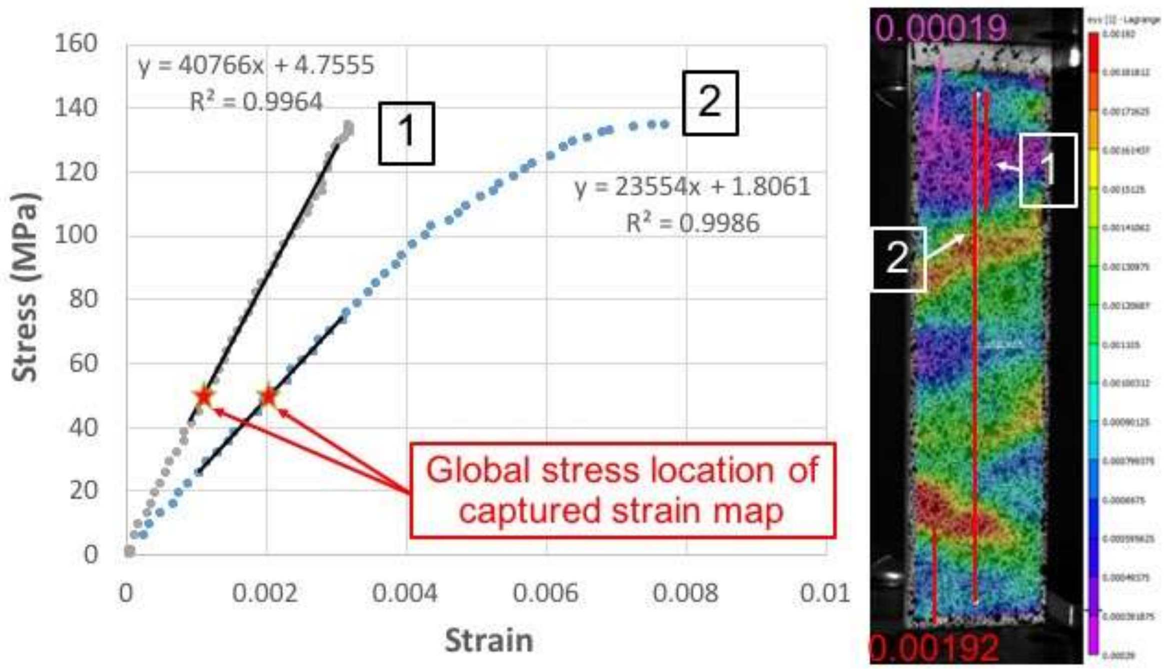

Figure 6 – Stress/strain behavoir of a tensile coupon from the wall section (sample WBD2) of double dome with the strain map at the starred stress position. This sample broke in the mid point where a congregated region of transversely oriented fibers was located.

Fiber Orientation by Tomography

To determine the internal orientation of these platelet based SCM carbon composites, two XCT scans were captured over ~25mm sample windows at 15-micron voxel resolution and stitched together. The typical fiber size is on the order of ~8 microns and thus, a single voxel is slightly larger than 'average' diameter of carbon fiber. Hence, the tomographic reconstructions are dependent on depicting the spatial arrangements of the macroscopic platelet structure. Thus, the intent is not to map single fiber orientations [4]. This approach provides desired information of microstructure at suitable length scales and represents statically significant region of volume for the related analysis. The acquired tomographic data is then processed in the Fiber Composite Analysis Module in VGStudio©. An average orientation tensor is calculated for each mesh element in a 3D mesh overlay on the specified sample region. Mesh elements in this study were 0.7x0.7x0.1 mm, which produced approximately 20 spatial orientation maps through the ~2.0mm thickness. The fiber direction corresponds to the Eigen vector associated with the maximum Eigen value of the orientation tensor. Extracting the coupon specimens defined in Figure 7 and calculating the orientations as described, the global histogram of all mesh element fiber orientations is plotted. The shape of these histograms gives critical insight into the flow behavior of the carbon fiber SMC during compression molding. As the charge pattern was centrally weighted in the mold, platelets flowed from the center of the double dome to the ends, which is captured in the histograms. Additionally, due to the boundary condition along the mold edge, the flange coupon revealed greater orientation along its axis, as material flowed parallel to edge of the mold. Figure 8 demonstrates a tomography slice with the corresponding orientation map for the two regions on double dome shown in Figure 7. Observing the high strain and failure locations on the coupons from DIC strain maps during tensile testing in Figure 5 and Figure 6. it is clear these regions correlate well with the locations of orientation transverse to the tensile axis. Thus, transverse loading to regions of high orientation produce high strains and failure locations.

These regions develop when the material flows inside the mold during compression molding and indicate the likely cause for the decreased failure stress in the double dome compared to the flat plaque parts for the considered loading direction or strain path.

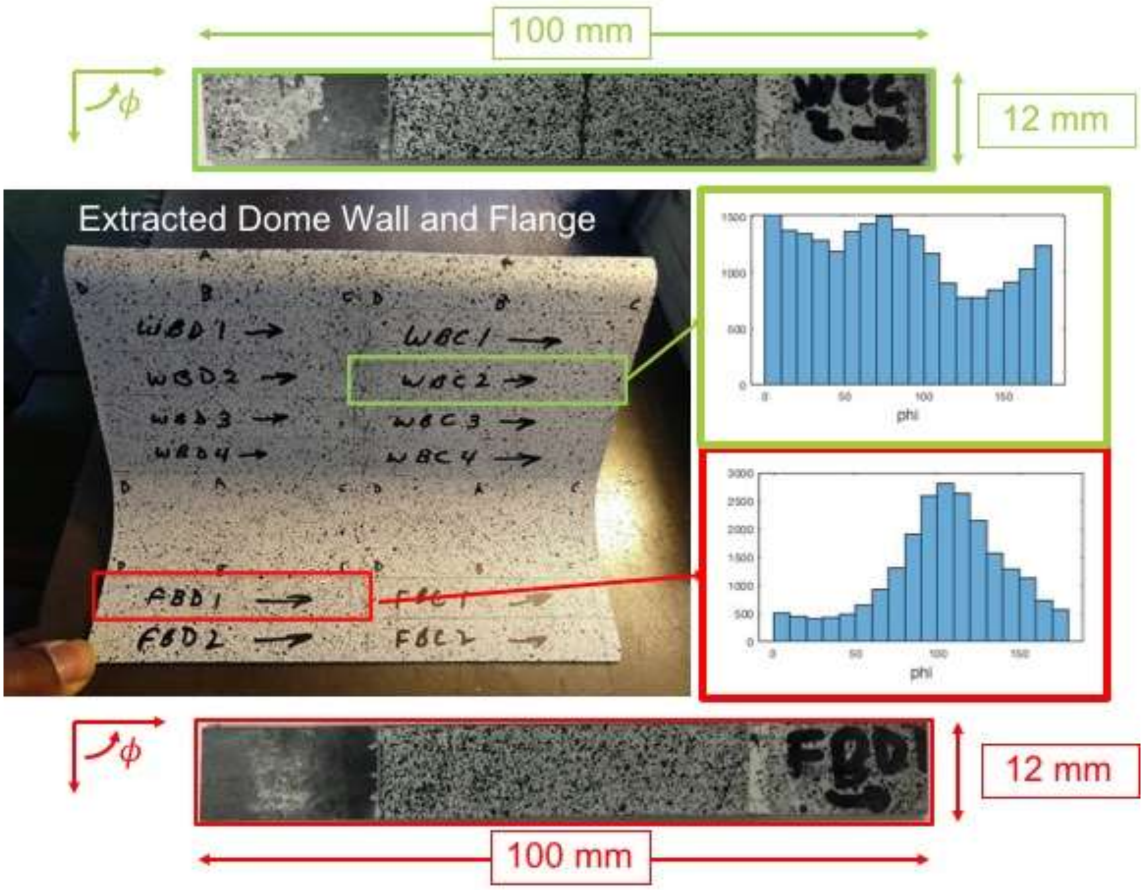

Figure 7 – Histograms of all mesh elements from the analyzed tomography data for two locations on the double dome, demonstrating the direction of material flow during compression molding.

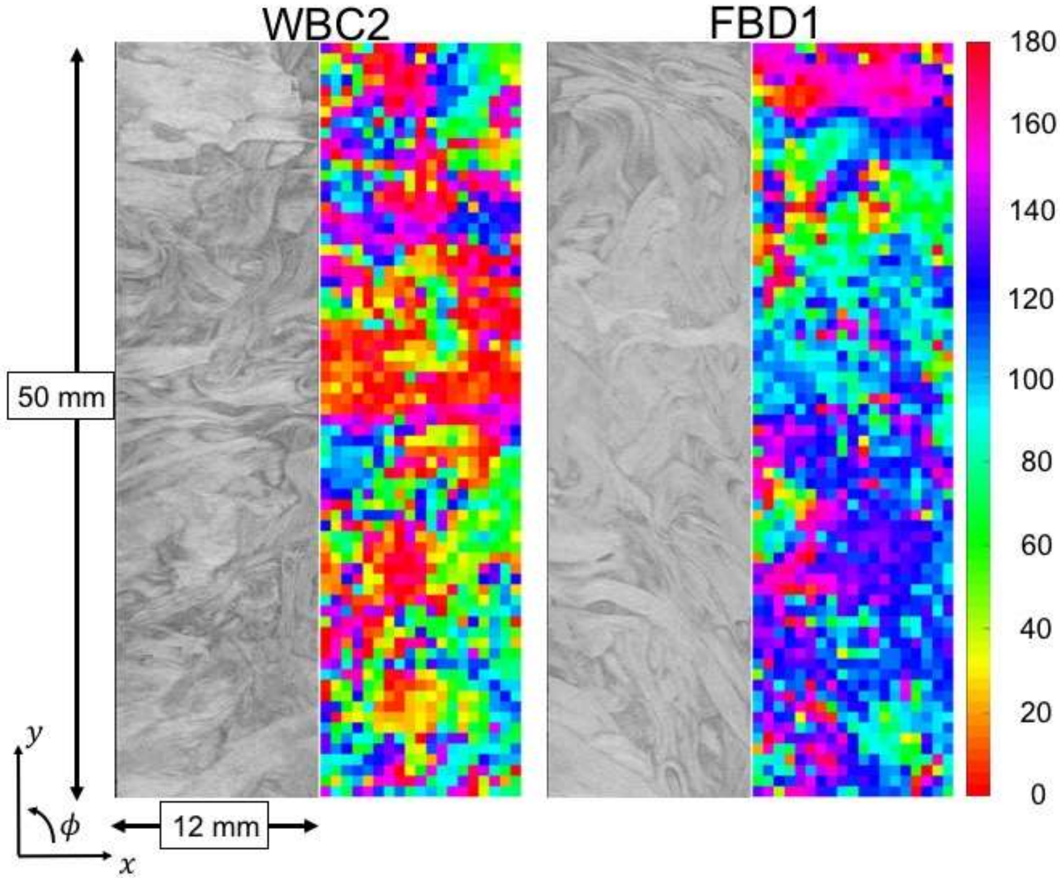

Figure 8 – Example of tomography and corresponding orientation map obtained after analysis for two parts on the double dome. The large areas of mis-orientation (i.e. warm colors) with the tensile axis of the coupon correlate well with high stain and failure zones.

Thermal Digital Image Correlation

Thermal Digital Image Correlation is a novel technique developed specifically for this material system in order to rapidly evaluate the preferred orientations spatially and validate large parts non-destructively. Essentially, the approach seeks to detect anisotropic mechanical thermal expansions, which arise from highly orientated fiber regions. Carbon fibers, which have a negative thermal expansion along their axis and have highly anisotropic response to thermal loading, and epoxy, which has a relatively large isotropic thermal expansion, are ideal candidates for sensitivity to this testing technique due to the contrast in their Coefficient of Thermal Expansion (CTE). For the tests performed here, parts were speckled with spray paint to obtain a trackable surface pattern, heated to in an oven for 30 min at 90C (or until they were isothermally loaded), and then imaged for DIC. An important note is that for industrial practice, the technique could easily be integrated into production by monitoring the cooling rather than subsequent thermal loading. Initial results suggest that excellent orientation mapping can be achieved with this surface only, approximate method, and in the least, regions of strong orientation can be identified over large areas. The fiber orientation vectors obtained from our TDIC technique are shown in Figure 9 for both the front and back surfaces of the same parts for which tomography data was presented in Figure 8 for select regions based on extracted samples from those locations using high resolution

XCT. A histogram of all the surface orientations obtained across the front and back of the coupons by TDIC is shown in Figure 10. Notably, the distributions from font and back match well and appear to correspond well with platelet orientation histograms obtained from a very precise X-ray tomography technique identified in Figure 7. These orientation vectors and histograms replicate the known manufacturing conditions, specifically that the charge material was densely placed in the center of the double dome part and flowed off the top of the double dome toward center flange and the end flanges.

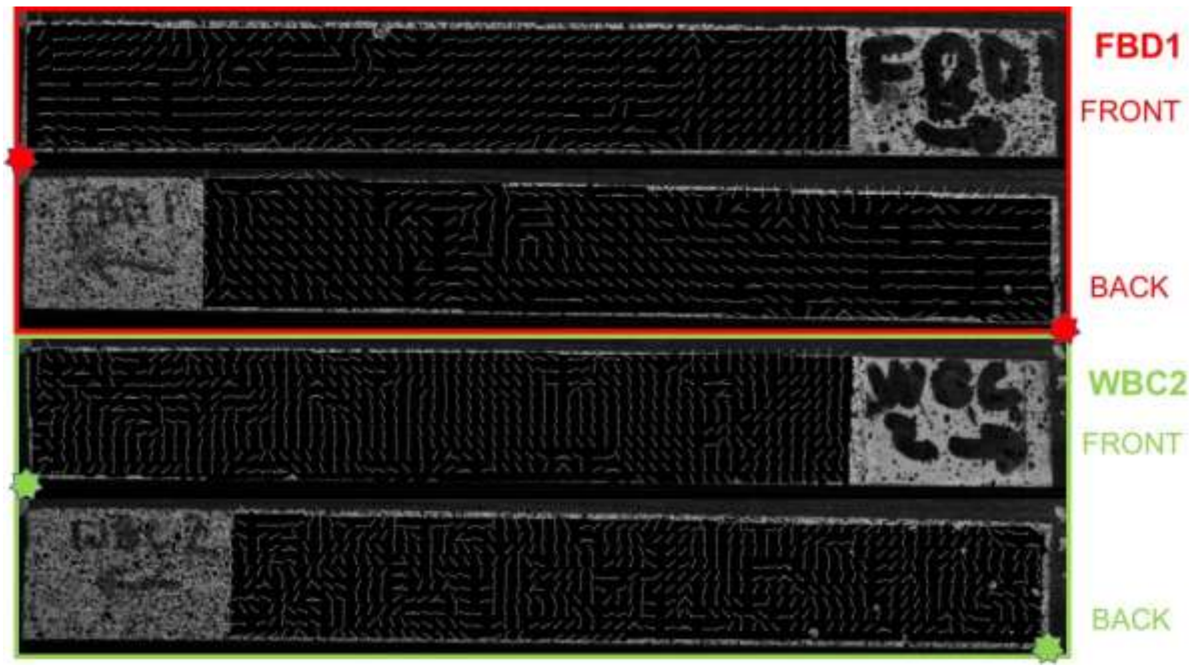

Figure 9 – Surface vector maps of fiber orientations obtained from the TDIC technique. The stars represent the same corners when the samples are imaged on the front and the back. This techniqe gives useful insight into the global orientation flow patterns in particular region of a large part.

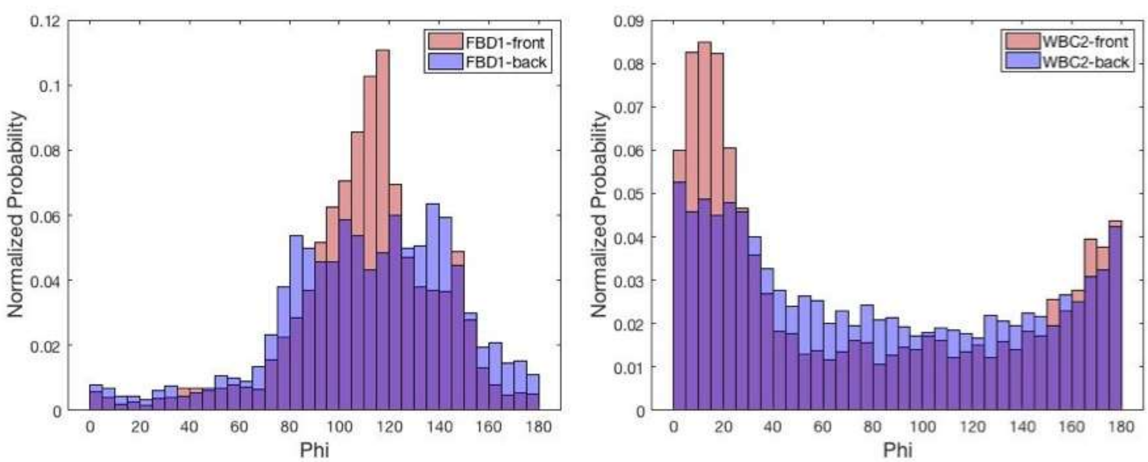

Figure 10 – The distribution of orientaion angles for Wall (WBC2) and Flange (FBD1) samples from TDIC for both front and back of the coupon. The corresponding green and red stars represent the same corners after the sample has been flipped over. These plots suggest the general orienations from TDIC match well from front to back and with

the histograms for these samples from tomography in Figure 7.

Optical Microscopy and Digital Image Analysis

Directly observing the surface by optical microscopy provides the most reliable method to date to confirm the orientation and internal microstructure, which includes the deformation associated with pre-molded platelets and any development of resin rich pockets which is undesirable. However, for orientation evaluation in three dimensions of non-circular fibers, like the low-cost carbon fibers used in making the platelets for this study, considering only the two-dimensional optical images, currently there is no established method. Thus, demonstrated here in this paper is a quick and simple approach to determine the fiber orientation for non-circular cross-sections using the variation of grey-scale intensity in relation to the orientation of considered plane of optical images. The goal is to capture the high intensity reflection using a monochromatic laser of the more orientated fibers against the low intensity reflection of fibers perpendicular to the polished surface and scale that intensity change as predominately associated with in-plane fiber orientation changes, the angle phi as described in Figure 8. Hence, out of plan orientation must be neglected, which is appropriate for composite parts with small thickness typically below 5 mm. A significant advantage is that stitched images using lower magnifications can be utilized (for example x20), increasing the field of view, since only a few pixels are necessary to obtain a cross-sectional intensity rather than many pixels necessary to accurately measure the cross-sectional area. Figure 11 documents example results from this approach. An automated method was quickly established for routine implementation of this approach that bins fiber intensities over a specified width, eliminating pixels with intensities that are below a threshold value which represents the matrix phase. When this method is applied to the fiber cross-section in Figure 11, excellent correlation is obtained locating fibers of high mis-alignment with the polished surface. This approach can be rapidly adapted to compare with orientation data obtained from large area with considered volume corresponding to tomography measurements, confirming and providing confidence in methods that are not directly based upon observing individual fibers.

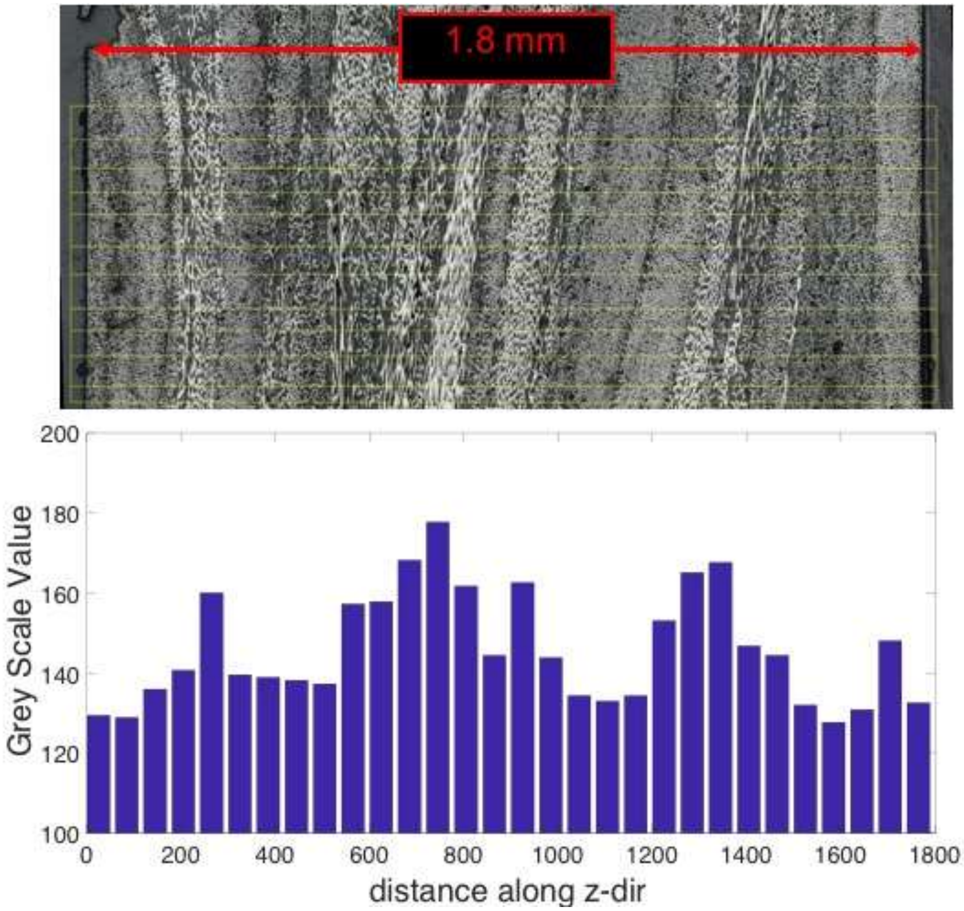

Figure 11

Conclusions

The considered novel carbon fiber platelet-based SMC system, which favorably competes with aluminum as an alternative light weight engineering material, demonstrates strong potential for compression molded based automotive part manufacturing and continued refinement is ongoing using results from this study. This composite material system is capable of achieving superb mechanical performance, 300 MPa failure stress and 40 GPa modulus for flat plaques. Furthermore, complex geometry parts can be fabricated rapidly, at very competitive cycle times, using largely existing infrastructure of automotive manufacturing employing compression molding approach. Presented in this paper were novel characterization methods used to understand the performance of these materials in order to harness its complete potential in a predictive fashion. Platelet orientation has been identified as a critical parameter in predicting the performance of these SMC material systems. Highly oriented regions of platelets develop where the material must flow to completely fill a mold without spreading. These can create weakness in mechanical properties transverse direction unless layers above and below this location can compensate similar to isotropic laminate design concept, and the detection of these locations often requires the destruction of the part using currently existing microscopy-based methods with very small field of view and extremely laborious effort. Thus, the mechanical characterization of large parts for quality control and performance benchmarks is challenging and essential for rapid commercialization of the considered material system. A new technique, Thermal Digital Image

Correlation (TDIC), demonstrated here, has been presented as viable solution to understand the orientation behavior of the carbon/epoxy-based platelets in a molded part. This technique is extremely fast, easily scalable for large part size, and can simply be implemented in a manufacture assembly line without the disruption of the manufacturing process. Further validation and development is currently being done to demonstrate the viability of this approach in a real-time manufacturing environment of fiber reinforced SMC type materials for structural applications in automotive space.

Acknowledgements

The authors would like to thank Dave Bank, Michael Lowe, Jason Reese from Dow Chemical Company, and Jeff Dahl and Patrick Blanchard from Ford Motor Company for related collaboration. Authors also gratefully acknowledge significant collaborative discussions and ideas with Purdue University collaborators (Professor Pipes, Drs. Goodsell & Denos, Mr. Kravchenko, and Mr. Favaloro), Michigan State University (Professor Drzal and Dr. Mike Rich), and Oak Ridge National Laboratory (Mr. Cliff Eberle and Mr. Robert Norris). This material is based upon work supported by the U.S. Department of Energy's Office of Energy Efficiency and Renewable Energy (EERE) under the support of the Task 3.2 of Institute for Advanced Composites Manufacturing and Innovation (IACMI), Award Number DE-EE006926 managed by John Winkel from DOE and John Unser from IACMI.

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