LONG-FIBER THERMOPLASTIC INJECTION MOLDED COMPOSITES: FROM PROCESS MODELING TO PROPERTY PREDICTION[1]

Ba Nghiep Nguyen, James D Holbery, Kenneth I Johnson, and Mark T Smith

Pacific Northwest National Laboratory – P.O. Box 999, Richland, WA 99352, USA

Abstract

Recently, long-fiber filled thermoplastics have attracted great interest within the automotive industry since these materials offer much better structural performance (e.g. higher elastic moduli, strength, and durability) than their short-fiber analogues, and they can be processed through injection molding with some specific tool design. However, in order that long-fiber thermoplastic injection molded composites can be used efficiently for automotive applications, there is a tremendous need to develop process and constitutive models as well as computational tools to predict the microstructure of the as-formed composite, and its resulting properties and macroscopic responses from processing to the final product. The microstructure and properties of such a composite are governed by *i) flow-induced fiber orientation, ii) fiber breakage during injection molding,* and *iii) processing conditions* (e,g. *pressure, mold and melt temperatures, mold geometries, injection speed, etc.*). This paper highlights our efforts to address these challenging issues. The work is an integrated part of a research program supported by the US Department of Energy, which includes

- The development of process models for long-fiber filled thermoplastics,
- The integrating process modeling and property prediction models as well as developing new constitutive models to perform linear and nonlinear structural analyses,
- Experimental characterization of model parameters and verification of the model predictions with forming experiments.

Background

Injection molding of fiber filled thermoplastics is a multi-step process which consists of i) pre-mixing and pelletizing the fibers and thermoplastic resin, ii) putting the pellets in an injection molding machine that melts them and injects them under high pressure into a mold (*mold filling*) iii) solidifying the as-formed composite in the mold (*packing, holding and cooling*), and iv) ejecting the part. Mold filling is of greater importance in injection molding of fiber-reinforced thermoplastics since the fiber orientations in the part are almost entirely determined by the flow patterns during filling. This phase is characterized by a *time-dependent non-isothermal and non-Newtonian flow* with free surface [1]. As a result, it is very challenging to accurately predict the flow-induced fiber orientation. The difficulty is greater when the fibers are long (> 1 mm) and/or concentrated fiber volume fractions are considered, and the part to be molded presents three-dimensional features. Basically, the mold filling problem is governed by three general equations from continuum mechanics which express the balances of mass, momentum and energy of the

[1] This manuscript has been authored by Battelle Memorial Institute, Pacific Northwest Division, under Contract No. DE-AC06-76RL0 1830 with the U.S. Department of Energy. The United States Government retains and the publisher, by accepting the article for publication, acknowledges that the United States Government retains a non-exclusive, paid-up, irrevocable, world-wide license to publish or reproduce the published form of this manuscript, or allow others to do so, for United States Government purposes.

fiber suspension in addition to the constitutive relation and fiber orientation equation. To complete the problem formulation, a constitutive relation is needed to describe the suspension behavior, and an equation to describe how the fibers can orient in response to the flow and fiber-fiber interaction must be added ([2], [3]). These additional equations are essential but are the main sources of difficulty in process modeling. Therefore, assumptions have often been made to simplify the suspension rheology, flow behavior, coupled phenomena, fiber-fiber interactions, etc.

Generally, it has been verified that the use of orientation tensors provides an efficient and accurate way to quantify the orientation state ([3]). Although injection molding of fiber polymers in general involves flows of non-Newtonian suspending liquids, the assumption of a generalized Newtonian fluid is useful and allows qualitative predictions of fiber orientations in injection molded parts. In addition, many of these parts possess thicknesses that are much smaller than other relevant dimensions, and consequently, the generalized Hele-Shaw model [1] which reduces the fully three-dimensional flow problem to a two-dimensional (2-D) formulation for the pressure is valid to model flows in thin cavities. However, the Hele-Shaw approximation has severe limitations to model flows in three-dimensional (3-D) geometries since it does not provide accurate velocity fields in 3-D features. Another important assumption consists of solving the flow equations as if the fibers were absent, then using the obtained velocity results to solve the fiber orientation problem. This uncoupled approach is often acceptable especially in the major (lubrication) part of the flow regions. Fiber-fiber interactions which are important in semi-concentrated and concentrated regimes have been modeled by adding a rotary diffusion term in the Jeffery equation [4]. This term is the key element of the Folgar-Tucker [2] model that is implemented in current commercial process modeling codes. It is noted that capabilities of 3-D solutions for the governing equations and/or coupled flow-orientation also exist (see [5], [6]). 3-D analyses using coupled or uncoupled approaches have improved the results for 3-D feature problems.

Current process models for injection molding have been applied to short-fiber filled polymers. The fact that these models are able to provide good qualitative fiber orientations is thanks to the acceptable validity of the simplifying assumptions for short-fiber suspensions. However, to date, no models exist to describe the flow of a long-fiber suspension. Here, we arrive at a principal difficulty which is linked to the rheology of the suspension and its microstructure. Long fibers have limited mobility due to their length and multiple fiber-fiber contacts [7]. Hence, they do not freely rotate and convect with the flow. Their behavior needs to be understood and accounted for in the establishment of the fiber orientation equation. This equation should be able to describe the fiber-fiber interaction when the fibers are long and it should include the effect of fiber breakage during injection molding. The first objective of our work is therefore to establish a fiber orientation model for long-fiber injection molded thermoplastics. Next, the goal of predictive engineering is also to develop capabilities to predict the macro (effective) properties and responses of the composite based on the information about its microstructure predicted by process modeling. This stage includes two important phases. The first phase is to interface process modeling to structural modeling so that the analysis of an as-formed composite can be achieved based on the composite microstructure predicted through process modeling. The second phase is related to the formulation and implementation of constitutive models for different types of structural analysis (e.g. thermo-elasto-plastic, damage, fatigue, creep, and impact analyses) that are needed for the design of composite structures.

Due to processing, a discontinuous fiber injection-molded composite possesses a microstructure that is strongly governed by the fiber orientation and aspect ratio, fiber volume fraction, fiber-matrix interfaces, and presence of defects plus the fiber and matrix properties. This complexity makes the prediction of the composite properties and responses to different loading conditions another highly challenging task in addition to the process modeling work. The difficulty is still greater since the composite microstructure often varies from one region to another in the part. Therefore, it is important to tract the microstructure as a function of the space coordinates. To date, most of the work in property prediction is limited to the prediction of the composite thermo-elastic properties using micromechanical models (e.g. the Mori-Tanaka method [8],[9]). There exists little work on the nonlinear behaviors. Some micromechanical modeling of damage is given in Refs [10] and [11]. The material nonlinearities are numerous and have different origins. For instance, under monotonic or cyclic loads, the composite can experience damage due to matrix cracking, fiber-matrix debonding, fiber pull-out and breakage. Also, the thermoplastic matrix undergoes plastic strains, and in general, plasticity is coupled with damage. Under constant applied stress, the composite also suffers from creep deformations. In order that long fiber thermoplastic injection molded composites can be used for semi-structural or structural automotive applications, it is essential to be able to predict the material durability in terms of strength, damage tolerance, fatigue and creep lifetimes. It is noted that the environmental effects are also important but are not discussed in this paper.

Recently, Nguyen et al. [12], [13] have carried out some structural modeling work, which uses constitutive material properties (fiber and resin) along with fiber orientation distribution functions and a multiscale mechanistic approach to predict damage and fatigue damage in discontinuous fiber polymer composites. In this approach, the stiffness reduction law is obtained by means of micromechanical modeling while the macroscopic response is determined using a continuum damage mechanics description based on thermodynamics of continuous media. This work constitutes a preliminary effort to link the overall response of the composite to its microstructure. An important industry need is to extend these predictive capabilities to include plasticity, damage, fatigue, creep and impact as the functions of the *local composite microstructure* provided by *process modeling*. Such an *integrated tool* will allow the user to predict the properties and durability of long fiber injection molded parts under specific loading conditions.

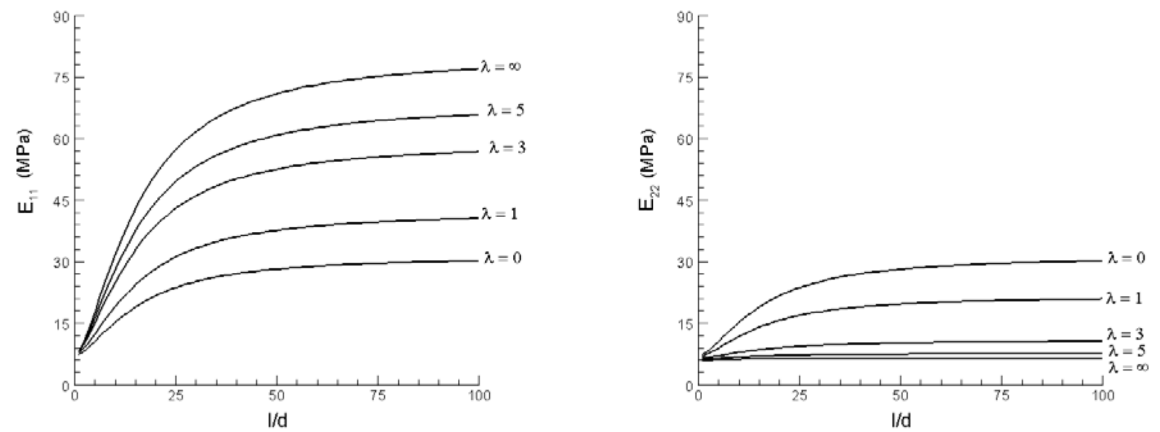
Research to Develop Long Fiber Orientation Models

Current fiber orientation models can be applied to short-fiber injection molded materials. However, no models exist for long-fiber systems. By examining the terms of the Folgar-Tucker equation [2], the limitation of this model can be understood. Advani and Tucker [3] have expressed the Folgar-Tucker model in terms of fiber orientation tensor components. According to this model, the fibers are convected with the fluid, and they re-orient in response to the deformation and rotation of the fluid according to:

$$\frac{Da_{ij}}{Dt} + \frac{1}{2}(\omega_{ik}a_{kj} - a_{ik}\omega_{kj}) = \frac{1}{2}\kappa(\dot{\gamma}_{ik}a_{kj} + a_{ik}\dot{\gamma}_{kj} - 2\dot{\gamma}_{kl}a_{ijkl}) + 2C_{\mathrm{I}}\dot{\gamma}(\delta_{ij} - 3a_{ij}) \quad (1)$$

where a_{ij} and a_{ijkl} are respectively the second and fourth-order orientation tensors, ω_{ij} is the vorticity tensor, $\dot{\gamma}_{ij}$ is the rate of the deformation tensor whose scalar magnitude is $\dot{\gamma}$. κ and C_{I} are material constants; κ depends on fiber aspect ratio r_{e}, and C_{I} is called the interaction coefficient. If $C_{\mathrm{I}} = 0$ and $\kappa = (r_{\mathrm{e}}^2 - 1)/(r_{\mathrm{e}}^2 + 1)$, Equation (1) is then Jeffrey's equation for the motion of a rigid ellipsoidal shape fiber in a Newtonian solvent. This is strictly valid for dilute suspensions in which the fiber-fiber interaction is absent or negligible. Jeffrey's equation imposes no restriction on the fiber aspect ratio. The last term of Equation (1) has been added to model the randomizing effect of fiber-fiber interaction in semi-concentrated suspensions.

Here, an important question is raised when applying Equation (1) to long fiber suspensions. That is, long fibers do not freely rotate and convect with the suspending liquid. They remain organized in domains and have limited mobility. As a consequence, representing the fiber-fiber interaction through a rotary diffusion term dependent on a scalar coefficient (C_I) would be only a rough approximation. Furthermore, the assumption that the fibers are rigid cylinders of equal length and diameter is not valid for injection molding of long fiber filled polymers since fibers undergo bending leading to rupture which reduces the fiber length considerably. This is an important issue that has not been addressed until now.



(a) (b)

Figure 1: Variations of the elastic moduli, E_{11} (a) and E_{22} (b) with the fiber aspect ratio and orientation for a carbon-PPS system. Planar orientation is assumed with the distribution density $\rho(\theta) = 1 - e^{-\lambda\theta}$, where θ is the orientation angle measured with respect to the 1-axis.

The effects of fiber orientation and aspect ratio on the elastic properties are illustrated in Figures 1a and 1b which respectively present the variations of the longitudinal (E_{11}) and transverse (E_{22}) moduli as a function of these parameters for a carbon-PPS (polypropylene sulfite) system. The calculations were carried out using the Mori-Tanaka method with the Karcir et al [14] fiber orientation distribution function dependent on parameter λ assuming planar orientation. These figures show significant variations of the moduli with the fiber aspect ratio, *l/d* in the range [0, 100]. This is the range of practical engineering interest which includes short and long fibers. Particularly, in the [0, 40] range, a small variation of *l/d* results in a much larger variation of the properties. Breakage of long fibers during injection molding reduces the fiber aspect ratio, which greatly affects the composite properties. It is therefore important to model this phenomenon to improve the property prediction of long-fiber composite systems. These figures also show the important effect of fiber orientation through the parameter λ. Between two limits which correspond to the case of aligned fibers ($\lambda = \infty$) and that of completely random fibers ($\lambda = 0$), the elastic moduli strongly vary with the fiber orientation. As a consequence, imprecision in fiber orientation prediction can result in important errors in property prediction.

Interface between Process Modeling and Property Prediction for Structural

Analyses

The ultimate goal of process modeling is to provide a realistic picture of the composite microstructure (i.e. fiber orientation, volume fraction, and aspect ratio, etc.) so that its effective thermoelastic properties and its nonlinear responses to various types of loading can be predicted. Also, if it is necessary, the composite properties can be optimized through returning to the process modeling step to adjust the process parameters to achieve the desired composite microstructure. This optimization through the process-linked structural analyses can be carried out only if an interface between these two distinct computation components exists. Hence, the next objective of our work is to establish this interface. Figure 2 shows a schematic picture of the optimization process. The analysis starts with an injection molding simulation to obtain a predicted composite part whose microstructure is determined at every point of the part. In finite element simulations, the final mesh obtained in process modeling will be imported to be used in structural modeling. Next, homogenization is carried out to compute the effective properties of the composite and its response to the prescribed loading.

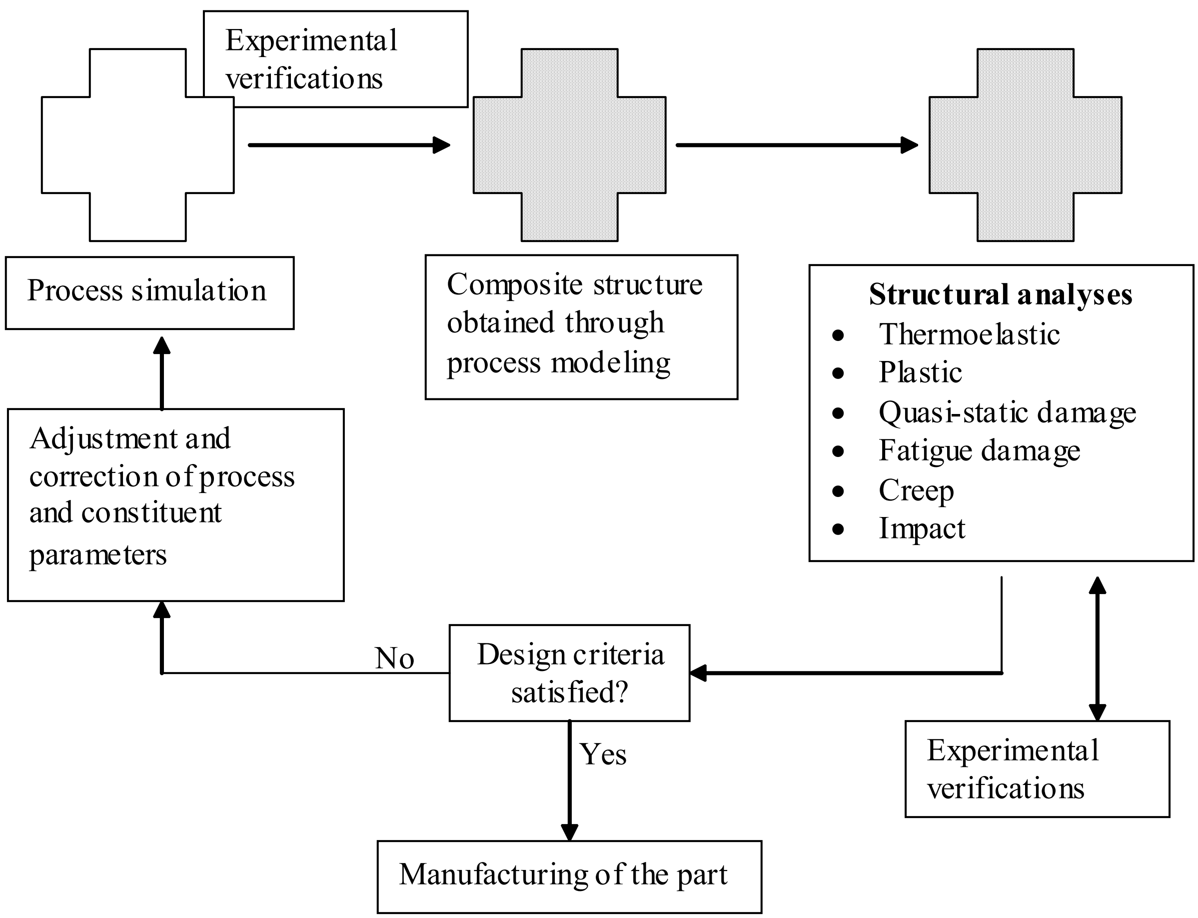


Figure 2: Schematic of the linkage between process and structural analyses that is needed for the design of an injection-molded composite structure through numerical simulations.

This phase will be implemented using structural finite element analysis, with design evaluation criteria that are specific to the type of loading (i.e. quasi-static, cyclic, creep, and impact), For instance, if the objective is to obtain a fatigue lifetime of 200,000 cycles, the fatigue damage analysis must be achieved to determine damage accumulation as a function of the number of cycles. The analysis would predict that the part could be manufactured if the prescribed fatigue damage tolerance is satisfied at 200,000 cycles. If the structure is predicted to fail before the desired maximum number of cycles, it would be necessary to improve the composite fatigue strength by optimizing its microstructure based on adjustment and correction of the process and/or constituent parameters. Process and structural re-analyses are then necessary until an optimized composite structure is obtained that satisfies the design criterion. It is noted that experiments to characterize the composite microstructure (e.g. fiber morphology, orientation, etc.) are needed to verify process modeling predictions. Experiments are also needed to obtain the model parameters and to verify the constitutive modeling. Experimental aspects will briefly be discussed in the next section.

Experimental Characterization and Verification

Short-fiber reinforced thermoplastics are commonly used in automotive parts because of commonalities in process equipment with neat resin products, homogeneities within the fiber filler, consistent fiber lengths that are generally 1 mm or less, and a generally well-defined procedure for predicting fiber orientation within the molded part. Long-fiber filled thermoplastic composites, on the other hand, pose significant challenges in both processing and fiber orientation prediction. Specifically, factors that are inherent in long-fiber injection molded parts include fiber breakage, large variations in viscosity due to decreased fiber mobility – especially at the mold front, non-homogeneous yield stresses, increased fiber-wall interactions, and poor fiber dispersion that can be manifested in the presence of fiber bundles in the molding [15]. The experiments aim at correlating the mechanical properties of the selected injection-molded composite materials with the predictive methods developed in the modeling activities (see the previous sections). Experimental characterization of the composite microstructure (i.e. fiber orientation and aspect ratio distributions, fiber organization structure) is essential since all the modeling work relies on it. This section lays out the potential techniques that can be used to determine a realistic "picture" of the composite microstructure. Finally, for the validation of the predicted composite properties and responses, established static, quasi-static and long-term durability test methods that have been developed for standard molded composite specimens will be used and later extended to testing of complex component geometries.

Injection molding of simple geometries will be performed using long glass or carbon fibers. The injection mold screw must be modified to provide the correct fiber shear to minimize fiber breakage and provide uniform distribution. However, the fiber aspect ratio of long-fibers results in an increased core thickness possibly reflecting the segregated state at which long fibers enter the mold cavity [16]. Steric interactions that inhibit the motion and reorientation of longer fibers can result in the fiber orientation of both the core and shell layers being different from that in short-fiber composites [17]. All of these factors require that careful experimental characterization and fiber orientation characterization be pursued in conjunction with any fiber orientation prediction modeling.

Nearly any measurement technique could be used provided that it effectively generates images of fibers on 2D sections through the material. Techniques could range from contact X-ray microradiography, scanning acoustic microscopy, to nuclear magnetic resonance [18]. For studies of mesostructural artifacts, optical techniques are preferred despite requiring a polished section of the sample. A review by Guild and Summerscales addressing use of image analysis for the characterization of composites summarizes the techniques [19]. McGrath and Willie reported an optical technique for determining the 3D fiber orientation distribution in injection molded thermoplastics that is highly dependent upon the refractive index of the matrix matching the reinforcing fiber within the presence of a small percentage of opaque tracer fibers during processing, usually 0.2% by weight within 30% by weight glass fibers [20].

If samples are to be prepared for 2D optical reflection microscopy and analysis by image processing techniques, great care must be taken with both sectioning and final polishing. Physical sectioning of long fiber reinforcements can easily cause fiber pullout and the sectioning of aligned fiber composites can leave chipped fibers and fragments at the surface. Clearly, fiber orientation characteristics that result from any 2D image analyzer depend upon the quality of the surface preparation.

Image reconstruction from projections using computed tomography (CT) produces a non-invasive measure of structure from external measurements. In X-ray CT, radiation is passed along straight lines through the object to a detector with the specimen located between the source and detector. With a stationary source and detector, the specimen is rotated and stopped at predetermined locations to acquire projections through the specimen. The projected signal is proportional to the amount of radiation reaching the detector. The logarithm of these measurements can be considered as line integrals of the X-ray attenuation coefficients of the specimen; knowledge of all line integrals allows the reconstruction of the interior structure [21]. X-ray tomographic reconstruction produces a two-dimensional map of X-ray attenuation coefficients of the irradiated cross section of the specimen [22].

Transmission tomography such as X-ray CT employs scanners capable of discerning between linear attenuation coefficients values by as little as 0.1%. In practice, density measurement from X-ray tomographic data can be performed either by calibrating the CT machines with objects of known density and obtaining a correlation equation that relates density with attenuation coefficients or by utilizing dual energy scanning to determine directly the density of the material [23]. Typically volumetric imaging of the sample is obtained by stacking a series of two-dimensional slices of CT image data. The quality and utility of the CT data ultimately depends on the machine resolution.

Application of the principles of CT at the microscale level, or micro-tomography, allows quantitative investigation of objects in three dimensions. Only recently, practical micro-tomography systems have been developed; early micro-tomography systems produced 2D images while current systems produce true 3D image data. In addition to electron impact X-ray sources, X-rays from synchrotrons have also been used in microtomography. Spatial resolution of the order of 1 μm and 15 μm can be achieved with the use of synchrotron radiation [24] and conventional microfocus X-ray generators [25].

For many years, the standard method of acquiring a volumetric CT scan has been by scanning a sample one slice at a time. In this method, a linear detector array and an X-ray point source are mounted opposite each other. A fan-shaped X-ray beam traverses the sample and the attenuated fan is stored as a 1-D linear image. By rotating the source/detector pair around the sample, a series of linear images are obtained which are subsequently used for the 2-D slice reconstruction. A volumetric representation is obtained by advancing the table on which the sample rests after each rotation in order to acquire a stack of such slices. Recently, cone-beam CT has been developed to acquire a 3-D data set with only one rotation of the sample. This provides for fast data acquisition and better X-ray utilization. In a cone-beam design, each projection of the object is, in essence, a radiograph. Attenuation measurements are simultaneously made for the entire object rather than for a single slice. The reconstruction algorithm is a generalization in three dimensions of the widely used convolution-back projection method.

Summary

This paper highlights the scope of our research program focusing on long-fiber injection molded thermoplastics. The challenge resides in three important tasks: process modeling, property prediction, and experimental characterization and verification. First, process models predicting fiber orientation and length attrition must be developed that account for the suspension rheology and microstructure. Once process modeling has predicted the composite microstructure, it is necessary to develop constitutive models that incorporate the features of this microstructure. These models will be used in macro finite element analyses of the composite under prescribed loads. In parallel, experimental characterization and verification need to be performed at three levels which are the microstructure, standard specimen, and actual part levels. The as-formed composite structure under specific loading will be modeled and tested to validate the integrated analysis which links processing to structural modeling.

Acknowledgements

This work has been funded by the U.S. Department of Energy's Office of FreedomCAR and Vehicle Technologies. The support by Dr. Joseph Carpenter Jr., Technology Area Development Manager, is gratefully acknowledged.

References

1. Crochet, M.J., F. Dupret and V. Verleye. "Injection Molding." in: Flow and Rheology in Polymer Composites Manufacturing, S.G. Advani, Ed., Elsevier Science (1994), pp. 415-463.
2. Folgar, F. and C.L. Tucker III. "Orientation Behavior of Fibers in Concentrated Suspensions." Journal of Reinforced Plastic Composites, (1984) 3, pp. 98-119.
3. Advani, S.G. and C.L. Tucker III. "The Use of Tensors to Describe and Predict Fiber Orientation in Short-Fiber Composites." Journal of Rheology, (1987) 31 (8), pp. 751-784.
4. Jeffrey, G.B. "The Motion of Ellipsoidal Particles Immersed in A Viscous Fluid." Proceedings of The Royal Society (1922) A 102, pp. 161-179.
5. VerWeyst, B.E., C.L. Tucker III, P.H. Foss and J.F. O'Gara. "Fiber Orientation in 3-D Injection Molded Features: Prediction and Experiment." International Polymer Processing XIV (4), (1999) pp. 409-420.
6. VerWeyst, B.E. and C.L. Tucker III. "Fiber Suspensions in Complex Geometries: Flow/Orientation Coupling." Canadian Journal of Chemical Engineering, (2002) 80(6), pp. 1093-1106.
7. Servais, C., A. Luciani, and J.-A. E. Manson. "Squeeze Flow of Concentrated Long Fiber Suspensions: Experiments and Model." Journal of Non-Newtonian Fluid Mechanics, (2002) 104, pp. 165-184.
8. Mori, T. and K. Tanaka. "Average Stress in Matrix and Average Elastic Energy of Materials with Misfitting Inclusions." Acta Metallurgica, (1973) 21, pp. 571-574.
9. Benveniste, Y. "A New Approach to The Application of Mori-Tanaka's Theory in Composite Materials," Mechanics of Materials, (1987) 6, pp. 147-157.
10. Meraghni, F. and M.L. Benzeggagh. "Micromechanical Modeling of Matrix Degradation in Randomly Oriented Discontinuous-Fiber Composites." Composites Science and Technology, (1995) 55, pp. 171-186.
11. Meraghni, F., C.J. Blakeman, and Benzeggagh M.L. "Effect of Interfacial Decohesion on Stiffness Reduction in A Random Discontinuous-Fiber Composite Containing Matrix Microcracks." Composites Science and Technology, (1996) 56, pp. 541-555.
12. Nguyen, B.N., and M.A. Khaleel "A Mechanistic Approach to Damage in Short-Fiber Composites Based on Micromechanical and Continuum Damage Mechanics Descriptions." Composites Science and Technology, 2004 (64), pp. 607-617.

13. Nguyen, B.N., B.J. Tucker, and M.A. Khaleel. “A Mechanistic Approach to Matrix Cracking Coupled with Fiber-Matrix Debonding in Short-Fiber Composites.” Journal of Engineering Materials and Technology, (2005) 127, pp.337-350.
14. Karcir, L, M. Narkis M, and O. Ishai. “Oriented Short Glass-Fiber Composites: I Preparation and Statistical Analysis of Aligned Fiber Materials.” Polymer Engineering Sciences, (1975) 15, pp. 525-531.
15. Truckenmuller, F. and H.G. Fritz. “Injection Molding of Long-Fiber Reinforced Thermoplastics: A Comparison of Extruded and Pultruded Materials with Direct Addition of Roving Strands.” Polymer Engineering Science, (1991) 18, pp. 1316-1329.
16. Spahr, D.E., K. Friedrich, J.M. Schultz, and R.S. Baily. “Microstructure and Fracture Behavior of Short and Long Fiber-Reinforced Polypropylene Composites.” Journal of Materials Science, (1990) 25, 4427-4439.
17. Bailey, R. and B. Rzepka. “Fiber Orientation Mechanisms for Injection Molding of Long Fiber Composites.” International Polymer Processing, (1991) 1, pp. 35-41.
18. Clarke, A.R., G. Archenhold, and N.C. Davidson. “A Novel Technique for Determining the 3D Spatial Distribution of Glass Fibres in Polymer Composites.” Composite Science and Technology, (1995) 55, pp. 75-91.
19. Guild, F.J. and J. Summerscales. “Microstructural Image Analysis Applied to Fibre Composite Materials: A Review”, Composites, (1993) 24, pp. 383-393.
20. McGrath, J.J. and J.M. Willie. “Determination of 3D Fiber Orientation Distribution in Thermoplastic Injection Moulding.” Composites Science and Technology, (1995) 53, pp.133-143.
21. Kak, A.C. and M. Slaney. “Principles of Computerized Tomographic Imaging.” IEEE Press, New York, (1987).
22. Lin, C.L. and J.D. Miller. “A New Cone Beam X-Ray Microtomography Facility for 3D Analysis of Multiphase Materials.” Second World Congress on Industrial Process Tomography, Hannover, Germany, (2001) pp. 98-109.
23. Lin. C.L. and J.D. Miller. “Cone Beam X-ray Microtomography for three-dimensional Liberation Analysis in the 21st Century.” International Journal of Mineral Processing, (1996) 47, pp. 61-73.
24. Kinney, J.H., Q.C. Johnson, and R.A. Saroyan. “Energy-Modulated X-ray Microtomography.” Review Scientific Instruments, (1988) 59, pp. 196-197.
25. Dunsmuir, J.H., S.R. Ferguson, K.L. D’amico, B.P. Flannery, and H.W. Deckman. “X-ray Microtomography: Quantitative Three-dimensional X-ray Microscopy”, Review of Progress in Quantitative Nondestructive Evaluation, D.O Thompson and D.E. Chimenti, Eds. (1991), Plenum Press, New York, 10A, pp. 443-449.