Full Field Nondestructive Techniques for Imaging Composite Fiber Volume Fraction

Joseph Nomasa Zalameda

College of William & Mary - Arts & Sciences

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FULL FIELD NONDESTRUCTIVE TECHNIQUES FOR IMAGING
COMPOSITE FIBER VOLUME FRACTION

A Thesis
Presented to
The Faculty of the Applied Science Department
The College of William and Mary in Virginia

In Partial Fulfillment
Of the requirements for the Degree of
Master of Arts.

by
Joseph N. Zalameda
1996
APPROVAL SHEET

This thesis is submitted in partial fulfillment of the requirements for the degree of Master of Arts

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ABSTRACT

In a composite the matrix distributes the load onto and between the fibers. It is therefore important to know the volume fraction of fibers to ensure proper load distribution and predicted structural performance. The most common method of determining composite fiber volume fraction (FVF) involves removal of the matrix by burn off or acid digestion. While estimates of the fiber, matrix and void volume fractions are obtained using this destructive method, it is time consuming and requires the disposal of toxic waste. This technique also requires the removal of a small composite section to be destructively tested. Structural parts in production are sometimes made with an excess area designated for removal for destructive testing. This type of testing determines the FVF only within that area thus making it a localized measurement. This causes uncertainty when the FVF varies within the manufactured part.

In this study the application of several nondestructive evaluation (NDE) imaging techniques to map out FVF variations were performed. The technologies investigated were thermography, ultrasound, and radiography. Theoretical models of each measurement technology was used to relate the physical property measurement (e.g. ultrasonic velocity, thermal diffusivity) to the FVF. For practical applications to varied ply orientations, measurements were made where no knowledge of the ply lay-up was required. Images were generated of the measured FVF and these results were compared to the destructive testing FVF images using a mean square difference metric.

On the basis of this metric it was found that the thermal technique provided the best agreement to the destructive results by a factor of 10 as compared to the ultrasonic velocity measurement for T-300 fibers in a 934 resin. The difference in transverse modulus between 934 resin and T-300 fiber was not significant enough to measure FVF ultrasonically, however it was very sensitive to porosity. The X-ray technology did not provide quantitative results. Mapping thickness variations of less than 5 percent did not significantly prove worthwhile in reducing the mean square difference. Finally the combined technique of using ultrasonic velocity to measure porosity and incorporating that information in the thermal model proved best overall in measuring FVF for porosity levels less than 5 percent.
FULL FIELD NONDESTRUCTIVE TECHNIQUES FOR IMAGING
COMPOSITE FIBER VOLUME FRACTION
CHAPTER 1

INTRODUCTION

Next generation structures are making increased use of composite materials. A specific example of a composite is graphite or fiberglass fibers combined with an epoxy resin to form a structural element. The fibers provide the desired stiffness and the resin acts as the glue to hold the fibers together thus providing the compression strength. Examples of composites with fibers embedded in a matrix are shown schematically in figure 1.1.

![Stacked Configuration](image1)

![Woven Configuration](image2)

Figure 1.1: Examples of stacked and woven composite configurations.

These fibers can be embedded in different orientations (by stacking or weaving) within the matrix to achieve desired structural characteristics.
Some examples of everyday structures using composites are fiberglass boats and graphite tennis rackets. Also newer vehicle structures such as fixed and rotary winged military/commercial aircraft, tanks, and cars are starting to make increased use of composite materials.

The US Army has recognized the importance of composites and is actively incorporating these structures into various areas. As the percentage of composite structure uses within the Army increases it is imperative to have reliable nondestructive inspection techniques to ensure expected structural performance. For example, the Composite Armored Vehicle (CAV) represents the potential of composites being used in warfare vehicles. The advantages of composites are lightweight and low signature while maintaining ballistic tolerance. To achieve these goals the CAV represents a combination of unique materials and structures. The CAV armor has a ceramic core sandwiched with inner and outer layers of fiberglass composites which makes up a significant portion of the armor. A CAV vehicle prototype is currently being developed for testing and new state of the art Nondestructive Evaluation (NDE) technologies are being assessed [1]. Next generation man portable weapon systems will have to meet more demanding weight specifications, and composites materials such as graphite/epoxy and Kevlar are being studied to meet missile launch tube requirements. Fielding of these systems has caused some concerns
regarding their susceptibility to damage and subsequent reduction in ultimate burst strength. Studies have been performed to evaluate defects within composite missile tube cylinders [2]. The focus of future work is to eventually relate defects to burst strength. Also all new fixed and rotary winged military aircraft will make extensive use of composite materials to reduce weight and potentially reduce fabrication cost [3]. These materials have been extensively used on blade and secondary structure for more than a decade and are now beginning to be routinely used on primary structure.

A composite’s strength is determined by the interaction between the fiber and matrix. Since the matrix distributes the load onto and between the fibers it is important to know the respective volume amounts to ensure proper load distribution. For example, high tensile strength is a property that makes composites widely used. A simple model [4] relating the axial tensile strength (critical buckling load stress) for a unidirectional composite with the respective volume fractions of the matrix \( (1 - V_f) \) and fiber volume fraction (FVF) \( V_f \) is given as:

\[
\sigma_{\text{tensile}} = \sigma_{\text{fiber}} V_f + \sigma_{\text{matrix}} (1 - V_f).
\]
The $\sigma_{\text{fiber}}$ is the fiber tensile strength proportional to the fiber failure strain. The $\sigma_{\text{matrix}}$ is the matrix stress when the fibers are strained to their ultimate tensile strength. This simple model is based on the assumptions that the fibers are unidirectional, there is no variability of individual fiber strength, composite fracture depends mostly on the fibers, and the loading is in the fiber direction. This model clearly shows that the tensile strength is dependent on fiber volume fraction (FVF). In a similar way the axial compressive strength is dependent on fiber volume fraction. In this case the composite is subject to compression loading in the direction of the fibers and failure can result in two modes. These failure modes are extension and shear. The extension failure mode results when some of the fibers buckle out of phase with each other resulting in a perpendicular extension of the matrix in between fibers. The shear mode failure results when some of the fibers buckle in phase and at the same frequency with each other resulting in shear deformation of the matrix. Examples of the extension and shear mode failures are shown in figure 1.2. The equations for axial compressive strength in the extension and inelastic shear failure modes are obtained from [5] and given respectively as:
\[ \sigma_{\text{comp. extension}} = 2 V_f \left( \frac{V_f E_f E_m}{3 (1 - V_f)} \right)^{\frac{1}{2}} \]  

(2)

and

\[ \sigma_{\text{comp. shear}} = \frac{G_m}{(1 - V_f)}. \]  

(3)

For the extension mode compressive strength \( E_f \) is the fiber Young’s modulus and \( E_{\text{matrix}} \) is the matrix Young’s modulus. For the shear mode compressive strength equation \( G_m \) is the matrix shear modulus. These equations predict that FVF can also affect the type of failure modes. For example, the extension mode dominates for lower FVF and shear mode failure dominates in higher FVF. An example is shown in figure 1.3 for a fiberglass composite. These equations show simplistic examples of how FVF can effect the strength of a structure and also type of failure due to the interaction between the fiber and matrix.

It is important to know the respective volume amounts of fiber and matrix because the strength and type of failure modes within a composite is
dependent on FVF. If the FVF varied within the manufactured part this
could cause some concerns. Improper tooling of process controls can
result in variations in FVF [6] causing difficulty in producing uniform
parts. A study by Cilley [7] was performed in 1974 where a variety of
methods for measuring FVF was performed. These methods included acid
digestion, water take up, and quantitative microscopy. Their findings
revealed the acid digestion technique to be the most practical.
Figure 1.3: Compression strength for extension and shear mode failure.

Source: ref [5].

This conclusion verifies what is found in industry today; the most commonly used method for determining composite FVF is by the removal of the matrix. It is a localized measurement performed by cutting a test section typically .635 cm square. These sections are then tested by removal of the matrix using heat or chemical digestion. This process is time consuming (approximately 1 hour per test) and the chemicals used produce toxic waste. In addition, variations of the FVF spatially would not be known unless the entire structure is sectioned and tested. The research reported in this thesis furthers the work of Cilley and considers state of the
art nondestructive techniques as a means to map FVF over a large area of a structure by using scanned or full field imaging techniques.

Nondestructive evaluation (NDE) is a measurement science relating measurable physical phenomena to structural characterization. Since these technologies are nondestructive, removal of a section or polishing of an edge is not required. Performing an NDE inspection can be thought of as a “health examination” for a material or structure either during the manufacturing process or during use. The safe, reliable, and efficient use of a composite can be greatly enhanced by our ability to measure its “health”. Growth in the use of composites is generating new material evaluation concerns such as structural design integrity, manufacturing quality assurance, damage detection and repair validation. The importance of NDE solutions arises because failure mechanisms of these composites are significantly different from metallic structures and the materials are often more complex internally presenting new and different risks to the users. Inspection technologies must be developed that meet these new requirements.

Technologies of interest investigated were thermography, ultrasonics and X-ray radiography. These technologies were chosen based on potential to image large areas and the ability of the energy to penetrate through the composite. The theory of each measurement technology is discussed along
with the quantitative measurement results and an assessment of the technology’s potential. For practical applications to varied ply orientations, measurements were made such that knowledge of the ply lay-up was not required. These results were compared using the destructive testing as a baseline. Finally what is presented in this study is a technique to map FVF nondestructively. The benefit of such a technology includes relatively rapid determination of FVF for on-line manufacturing inspections. In addition another benefit of measuring the FVF over an area is that mapped values can be used as input into a finite element model to accurately simulate the composite’s performance. This information could be used for design strength studies.
CHAPTER II

TECHNOLOGIES FOR MEASURING FIBER VOLUME FRACTION

Many techniques are currently being used to determine fiber volume fraction (FVF). Currently the most widely used technique to determine FVF is by destructive matrix digestion. The procedure for destructive matrix digestion FVF testing, standard D-3171-76, is described in the American Society for Testing and Materials (ASTM) Handbook [8]. The description of this test standard includes cautions pertaining to the use of hazardous chemicals and procedures. This test involves cutting out a small test coupon approximately .635 x .635 to 1.27 x 1.27 cm. The test coupon is weighed and then the volume is measured using a water displacement technique. The resin is then removed either by acid digestion or oven burn off. This removal procedure can take up to 5 hours to insure that the resin is totally removed. Some recent work has been done to decrease this time using microwave energy in addition to acid attack [9]. The next step is to wash the fibers in acetone and water and then heat at 100 degrees Celsius to remove any residuals. The fibers are then weighed, and the resin and FVF are calculated from the following equations respectively.
Resin Volume % = 100 \left( 1 - \frac{\text{Fiber Weight}}{\text{Coupon Weight}} \right) \left( \frac{\text{Coupon Density}}{\text{Resin Density}} \right) \quad (1)

Fiber Volume % = 100 \left( \frac{\text{Fiber Weight}}{\text{Coupon Weight}} \right) \left( \frac{\text{Coupon Density}}{\text{Fiber Density}} \right) \quad (2)

Also the void volume fraction can be calculated if the resin and fiber volumes do not add up to 100%. The equation is given below.

\text{Void Volume %} = 100 - \left( \text{Fiber Volume %} + \text{Resin Volume %} \right) \quad (3)

In addition to being destructive and operator dependent, this technique is time consuming and requires the disposal of toxic waste. This technique also requires the removal of a small composite section to be destructively tested. Structural parts in production are sometimes made with an excess area specifically for removal for destructive testing. This type of testing determines the FVF only within the area that was removed thus making it a localized measurement. Nevertheless, this technique is the industry standard and widely accepted.

Another method used to determine FVF is by quantitative microscopy. This optical method again provides a local measurement of FVF. Here an edge region on a composite, either a removed coupon or
local section, is polished and then viewed through a microscopic image capture system to obtain a digital photomicrograph. The photomicrographs are then thresholded to identify the different regions corresponding to fiber and matrix. A statistical analysis is then performed to determine the respective volume amounts. An example of a photomicrograph obtained from [10] is shown in figure 2.1. The darkest areas show porosity, the semi-dark areas are the fibers oriented in stacked layers zero degrees (along the page) and ninety degrees (coming out of the page), and the lightest area is the matrix. The advantages of this technique are that no harmful waste by products are produced and even after accounting for the specimen surface preparation time this technique is much faster than matrix removal methods. This technology

![Figure 2.1: Photomicrograph of polished composite edge. Source: ref [10].]
has been shown to produce results that correlate well with destructive tests [11]. The disadvantages of this technique are first, the identification of fiber matrix boundaries by thresholding can be subjective. Consistent specimen polishing can also effect image thresholding levels. Secondly, since an edge is measured this technique is highly localized.

The advantages of a thermal inspection system are many. Since an infrared imaging system is used, large areas can be inspected at once which makes the measurement efficient and fast. Also the measurement is noncontact and therefore couplants or dyes are not necessary. This advantage also allows the inspection of more complex geometries (ie curved surfaces). No harmful radiation is used so the measurement is safe. In addition, the results are archivable in image form for later reference.

The disadvantages of this inspection technology are as follows. First if the material has a low thermal emissivity (reflective) then it is difficult to induce heat into the material and also to measure its temperature. Sometimes a water based emissivity coating is applied. Also this technique has limited depth sensitivity due to the minimum temperature resolution of the camera and the diffuse nature of thermal energy. Nevertheless thermal inspection techniques have shown much promise in detecting composite defects such as impact damage [12], foreign object damage (inclusions) [13], cavities in foam core structures [14], disbonds in tubular
structures [15], and FVF variations [16]. It is one purpose of this research to extend the FVF work from a single point measurement to the use of an infrared camera for full field mapping. Real time digital image processors are used to digitize and store images for processing. This data is then compared to an analytical model to obtain a quantitative measurement of thermal diffusivity. Thermal diffusivity is a material property governing the rate in which heat flows within a material and is defined as:

\[
\alpha = \frac{k}{\rho c}
\]  

(4)

where \( k \) is the thermal conductivity, \( \rho \) is the density and \( c \) is the specific heat. Thermal diffusivity can be defined as the ratio of heat conduction to the heat storage. Much work has been done developing techniques to measure this material property using a periodic phase lag method [17], high intensity arc method [18], and flash method [19]. The flash method is used often because the instantaneous source can be modeled by an impulse function and subsequent solutions can be written down directly from the Green's function [20]. The derivation and discussion of the temperature response of a composite relating thermal diffusivity with FVF is given in Appendix A.
The transmission of high frequency sound energy into a material is known as ultrasound. This inspection technique can determine various defects within a material by measurement of propagation velocity, energy attenuation, energy reflection through interfaces and energy mode conversions. The primary advantage of ultrasound is its high sensitivity to a number of varied composite defects such as: impact damage [21], porosity [22], FVF variations [23], thickness variations [24], and foreign objects [25]. These defects are detected using ultrasound because the wavelength is comparable to the defect size. Ultrasound is a mature technology, it is safe and it has single sided measurement capability. Although ultrasonics is quite useful the disadvantages of this technique are that couplants such as gel or water are required and also that the measurement requires physical contact with the structure.

The basic equations that describe ultrasonic wave propagation can be derived from equation of motion for a vibrating medium and Hooke’s Law which can be stated from [26] respectively as:

\[
\frac{\partial}{\partial r_j} T_{ij} = \rho \frac{\partial^2 u_i}{\partial t^2}
\]

and
\[ T_{ij} = c_{ijkl} S_{kl} \] where \( i, j, k, l = x, y, z \). \hspace{1cm} (6)

\( T_{ij} \) is the stress field, \( \rho \) is the density, \( u_i \) is the displacement, \( c_{ijkl} \) are the elastic stiffness constants and \( S_{kl} \) is the strain tensor. Hooke’s Law can be stated in abbreviated notation by using symmetry arguments and the following subscript notation from [26].

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Using the abbreviated notation, Hooke’s Law may now be stated as:

\[ T_1 = c_{1j} S_j. \] \hspace{1cm} (7)

The elastic constant tensor \( c_{ij} \) can be stated in matrix form [23] for a unidirectional composite (transverse isotropy) with the orientation shown in figure 2.2.
A y-propagating y-polarized displacement wave can be represented by:

\[ u_y = \cos(\omega t - ky) \]  \hspace{1cm} (9)

with a corresponding strain field:

\[ S_{yy} = k \sin(\omega t - ky). \]  \hspace{1cm} (10)
The stress field can then be calculated from the strain field using Hooke’s Law and since the stress field varies only in the y direction the longitudinal velocity can then be solved.

\[ V_1 = \sqrt{\frac{c_{11}}{\rho}} \]  

(11)

The longitudinal velocity is calculated for a wave propagating in the direction perpendicular to the fibers. By measuring only this component of the velocity, ply orientations have minimal effect on the propagation because the wave is always polarized perpendicular to the fibers. This has been confirmed by experimental measurements performed by Chang [27]. Because knowledge of the ply orientations are not required to be known this measurement has the best potential for practical application to various lay ups. Solving for the velocity using the other elastic constants such as \( c_{12} \) or \( c_{44} \) gives shear wave velocities with differing polarization with respect to the fiber. These velocities are more sensitive to the FVF but also very sensitive to the ply orientations [28]. Attenuation measurements are also sensitive to FVF but are more sensitive for porosity measurements [22,29]. Because of these factors the use of longitudinal wave velocity was chosen to image FVF and porosity. This technique is reported in the
literature by Martin [30] to have good sensitivity to FVF variations for 
elastic isotropic fibers such as glass and for elastic anisotropic fibers, such 
as graphite, there is some velocity variation. The theory is highly 
dependent on the difference between the transverse elastic modulus of the 
matrix and fiber. If there is a large difference in the elastic modulus then 
the measurement of FVF is more promising. If, however, the elastic 
modulus of the fiber and matrix are similar then the velocity measurement 
is more sensitive to porosity effects.

This work will focus on longitudinal measurements on T300 fiber 
and 934 resin. The theory used to relate the $c_{11}$ elastic constant to FVF is 
found by combining the work of Hashin [31] who takes into account the 
effect of voids in the matrix with Martin [30] and Smith [32]. The 
equations relating the $c_{11}$ elastic constant to the FVF and porosity shown in 
Appendix B are primarily taken from Smith. The longitudinal velocity and 
therefore the $c_{11}$ elastic constant is measured using a technique described 
by Sachse and Pao [33].

X-rays are commonly used for nondestructive evaluation of 
structures because of the ability of the X-rays to penetrate a material. A 
high voltage is used to accelerate electrons onto an anode. The electrons 
are usually focused to hit a target such as tungsten and as a result X-ray 
emissions are produced. The energy spectrum of the X-ray is determined
by the tube voltage. The X-rays are then directed toward a photographic film with the item tested in between. The governing equation for X-rays traveling through a structure is given by Lambert’s Law:

\[ I = I_0 \exp \left( -\mu \rho x \right). \]  

(12)

The initial intensity is \( I_0 \), the attenuated radiation intensity \( I \) is reduced exponentially as function of \( \mu \) the mass absorption coefficient at a given energy, \( \rho \) the material density, and \( x \) the material thickness. The total mass absorption coefficient is obtained from [19] for a composite as:

\[ \mu = \sum_i f_i \mu_i \]  

(13)

where \( f_i \) is the elemental weight fractions and \( \mu_i \) is the constituent mass absorption coefficients for a given energy. By converting the weight fractions to volume fractions the intensity equation (12) is given as:

\[ I = I_0 \exp \left[ - \left( V_f \mu_{\text{fiber}} \rho_{\text{fiber}} + (1 - V_f) \mu_{\text{matrix}} \rho_{\text{matrix}} \right) x \right]. \]  

(14)
Where $V_f$ is the FVF, $\mu_{\text{fiber}}$ is the fiber absorption coefficient, $\mu_{\text{matrix}}$ is the matrix absorption coefficient, and $\rho_{\text{fiber}}$ and $\rho_{\text{matrix}}$ are the fiber and matrix densities respectively. X-ray absorption measurements for the determination of FVF has been discussed by Martin [34]. His findings show that X-ray film technology is not feasible for small 1 percent variations in FVF based on insufficient film sensitivity. His work was based both on theory and experimental measurements performed on FVF sample variations around 61 - 66 percent. This work will use X-ray film technology to measure greater variations in FVF for a comparison to the other imaging techniques. More recent work in the early 1990's using sensitive scintillation detectors has shown promise for composites over film technology [35, 36]. A Reverse Geometry X-ray™ technique [37] using a scintillation detector was used in addition to conventional film technology. The unique capability of the Reverse Geometry X-ray™ system is that it uses a scanned X-ray source for radiographic imaging of materials and structures.
CHAPTER III

EXPERIMENTAL MEASUREMENTS PERFORMED

The samples measured were T300 fiber 934 resin 16 and 32 ply graphite epoxy composite plates with lay ups of [0/90] 4s and [0/90] 8s. The target FVF’s were 60, 65, and 70 percent. The 65 and 70 percent FVF were fabricated by prebleeding the plies before curing. Unfortunately significant levels of porosity was introduced into the structure by using this technique. A press curing system was utilized for consistent thickness. The plates were 30.48 x 30.48 cm in size and were sectioned into three sub plates two 15.24 x 15.24 and one 30.48 x 15.24 cm. Destructive test coupons were obtained at the middle and opposite corners of each plate to determine the FVF. Using this manufacturing technique it was hoped that the plate FVF would be consistent. This was not the case, however, as the FVF varied throughout the plates, especially for the higher target FVF’s. In addition, for the higher FVF plates significant porosity levels were unfortunately introduced.

Thickness measurements were performed to compensate for apparent measured changes in physical properties (i.e. diffusivity, velocity, etc.).
By removing the thickness contributions a more accurate determination of the internal structure’s FVF was desired. Thickness variations within these test samples were measured to vary by about on average +/- 2 percent for each plate. The thickness measurement setup used a dual contact robotic scanning arm. The spatial resolution was 0.13 cm and the thickness was computed from the difference in the arm deflections. The sample was mounted in a firm bracket holder to prevent bending. The time required to scan one 15.24 x 15.24 cm panel was approximately 4 hours for 12,995 points.

Representative thickness imaging results are shown in figures 3.1 and 3.2. These images are 115 x 113 in pixel size. Each pixel represents a 0.13 cm increment. Areas of interest, 5.08 x 5.08 cm and 5.08 x 10.16 cm, were marked within each plate sample using a grid with a spatial sample size of 1.27 x 1.27 cm. Since the destructive testing sample size is 1.27 x 1.27 cm, the thickness image areas chosen for destructive testing was averaged spatially down to the same sample size. These results are shown in figure 3.3.
Figure 3.1: Thickness image of 60 and 65 percent composite test samples.
Figure 3.2: Thickness images for 70 percent 16 and 32 ply composite test samples.
Figure 3.3: Thickness images spatially averaged for 16 and 32 ply composite test samples.
Thermal techniques show promise for measuring FVF because of the fiber's thermal conductivity is significantly greater than the matrix thermal conductivity. The implementation of a thermal diffusivity measurement technique is described. The setup used is shown in figure 3.4. The thermal inspection system consists of four main components: the heat source, infrared camera, image processor, and computer. The camera and heat source are on opposite sides. The heat source is a high power commercially available photographic flash lamp. The infrared camera is a scanned HgCdTe detector with a minimum temperature resolution of .1 degrees Celsius and a 2 milliradians instantaneous field of view. The camera output, 30 frames/sec., is connected to a real time image processor for digitizing, averaging, and storage. The thermal images were 256 x 256 in pixel size. The computer controlled experimental parameters such as lamp turn on, digitizing delay times and number of frames averaged. The computer was also used for data reduction and storage. No emissivity coating was applied to conduct the thermography measurements.

The through-transmission heat up response of the plate front surface was measured by averaging the digitized temperature images and sequentially storing these images for analysis. A cyclic averaging scheme was used where the flash lamps were turned on 16 times. The flash
duration was on the order of 5 - 10 milliseconds and is considered to be instantaneous compared to the thermal response of the samples and sampling rate. There were a total of 32 storage frame locations used and each location had 1, 2, or 4 video frames averaged within for each cycle depending on the thickness. In addition there was a delay between each cycle of approximately 1 - 2 minutes. The total measurement time was around 20 - 25 minutes depending on number of frames averaged. As the heat diffuses through to the other side it will rise to a steady state value. A representative data curve, along with a fitted equation from Appendix A, for a 1.27 x 1.27 cm area is shown in figure 3.5 and represents 100 averaged data points. The data shows a temperature rise of approximately 8 degrees above ambient.
Figure 3.5: Typical normalized thermal data curve with fitted result.

The diffusivity can be obtained from the fit if the thickness is known. The analysis is sensitive to thickness changes as well as diffusivity changes. If the thickness is known then the thermal diffusivity can be computed. The imaged diffusivity results are shown in figures 3.6 and 3.7. Again the grid areas correspond to the destructively tested areas. Figure 3.8 shows the destructively tested diffusivity images with a spatial sample size of 1.27 x 1.27 cm. These images were calculated using a single averaged thickness value for a given test plate. The images in figure 3.9 shows the thermal diffusivity results of the destructively tested regions with the thickness correction using the corresponding thickness images.
Figure 3.6: Diffusivity images of 60 and 65 percent composite test samples.
Figure 3.7: Diffusivity images for 70 percent 16 and 32 ply composite test samples.
Figure 3.8: Thermal diffusivity images of areas destructively tested, spatial sample size is 1.27 x 1.27 cm.
Figure 3.9: Thermal diffusivity images of areas destructively tested with thickness variations corrected, spatial sample size is 1.27 x 1.27 cm.
The matrix thermal conductivity was not known so the destructive data was used in conjunction with measured diffusivity to obtain a value. The measured diffusivity and the destructively determined FVF was also used to determine which thermal conductivity model SPM (Nusslet), SPAM (Springer and Tsai), and CCA (Hashin and Rosen) is best. The model was chosen based on minimization of the Chi Squared error using the matrix thermal conductivity as the best fit parameter. A more detailed description of the models is shown in Appendix A. In the fit, the destructively determined porosity information is also used. The thermal conductivity of the matrix was chosen as the fit parameter. An example of the measured data fitted to the model is shown in figure 3.10. The diffusivity measurement uncertainty is approximately +/- 2.5 percent and the destructive test error is on the order of +/- 1 percent. The model shown in this figure is the CCA model with porosity taken into account. Table 1 below shows the Chi-Squared merit function values and the fitted matrix thermal conductivity values. From the Table the CCA and the SPM model gave the best fit to the measured data. The matrix thermal conductivity value determined by the CCA model is within the range given for unfilled cast epoxy resin systems in [38]. Because of this the CCA model was chosen along with the corresponding matrix thermal conductivity value.
This matrix thermal conductivity value is then used with the corresponding CCA model to calculate a measured FVF from the known

Figure 3.10: Destructively measured FVF with diffusivity compared to model where destructive porosity information is also used.

<table>
<thead>
<tr>
<th>Model Type</th>
<th>Chi-Squared Value</th>
<th>Matrix Thermal Conductivity W/cm-C</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPM</td>
<td>0.00001225</td>
<td>0.00297</td>
</tr>
<tr>
<td>SPAM</td>
<td>0.00001308</td>
<td>0.00195</td>
</tr>
<tr>
<td>CCA</td>
<td>0.00001265</td>
<td>0.00180</td>
</tr>
</tbody>
</table>

Table 1: Quantitative comparison of different thermal models.
<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>FIBER</th>
<th>MATRIX</th>
<th>AIR</th>
</tr>
</thead>
<tbody>
<tr>
<td>VOLUMETRIC HEAT CAPACITY J/cm³°C</td>
<td>1.3275</td>
<td>2.444</td>
<td>.0012</td>
</tr>
<tr>
<td>THERMAL CONDUCTIVITY W/cm°C</td>
<td>1.73</td>
<td>.00180</td>
<td>.00026</td>
</tr>
<tr>
<td>DIFFUSIVITY cm²/sec</td>
<td>1.24</td>
<td>.00127</td>
<td>.2216</td>
</tr>
</tbody>
</table>

Table 2: Material properties used for thermal calculations with matrix thermal conductivity found from fit to data other values from [39].

diffusivity. A discussion of these results on an image by image basis is included in Chapter III of this report. The material property values used to calculate a measured FVF are shown in Table 2.

Ultrasonic velocity measurements were performed on the test specimens and the technique to measure the velocity using a phase spectrum approach is described. Ultrasonic velocity can vary with FVF and porosity changes. The ultrasonic measurement setup used a pulse echo configuration is shown in figure 3.11. A 5-megahertz 5.08 - cm fixed-focus compressional wave transducer was used. The transducer was scanned in two dimensions robotically and the position information was
stored in the computer. A broadband pulse was generated by the pulser and the return signal was amplified by the receiver. The output of the receiver was connected to a 100 Mhz digitizing oscilloscope with 8 bit resolution. The data was then stored by the computer. The scan resolution was 0.13 cm in step size and the images were 140 x 140 pixels. Spacers were used to offset the glass reflector from the sample so only the glass reflector return was digitized. The transducer, composite sample, spacers, and glass reflector plate were immersed in a water tank.

The longitudinal velocity was measured for a wave propagating in the direction perpendicular to the fibers. By measuring only this component of the velocity, ply orientations should have minimal effect on the propagation because the wave will always be polarized perpendicular to
the fibers and thus no knowledge of the ply lay-up was required. The longitudinal wave velocity was measured using a phase spectrum technique [33] where a spectral phase difference is measured. This phase difference corresponds to a time difference or a shift in time. This can be seen by observing the glass plate reflection. Because the reflection off the glass plate is consistent and a good representation of the initial pulse the glass plate reflection signal was chosen for digitizing. The digitized signal represented 200 sampled data points. The sample rate was 100 mega samples per second.

The phase difference is measured by first scanning the glass reflection plate and storing the data as a reference. This will also allow any nonuniformities in the glass plate to be removed from the measurement. A representative waveform of the reflected signal is shown in figure 3.12. The introduction of the sample between the glass reflection plate and transducer causes a shift in the propagation time of the echo signal. This is shown in figure 3.13. The ultrasound propagation through the sample is faster than through water and therefore a decrease in the propagation time is measured when the sample is scanned. A discrete periodic Fourier transform was implemented to obtain the magnitude and phase information. The digitized signal was padded with 56 points to implement the Fast Fourier Transform algorithm. The magnitude response
Figure 3.12: Ultrasonic pulse return off of glass plate.

Figure 3.13: Shifted ultrasonic pulse return off of glass plate caused by composite sample.
of reference and shifted ultrasonic waveforms is shown in figure 3.14. The phase response of reference and shifted ultrasonic waveforms is shown in figure 3.15. The difference in phase corresponds to the propagation time decrease. This time decrease can then be used to calculate the phase velocity of the composite sample using equation (1) below.

\[
V_{\text{long}}(f) = \frac{(2 \cdot L) \cdot 2\pi f}{\phi_1 - \phi_2}
\]

(1)

Where \(L\) is the thickness, \(f\) is the frequency, \(\phi_1\) is the reference phase and

![Comparison of Magnitude Response](image)

Figure 3.14: Magnitude of reference and shifted ultrasonic waveforms.
Comparison of Phase Response

Figure 3.15: Phase response of reference and shifted ultrasonic waveforms.

Phase Velocity Spectrum

Figure 3.16: Phase velocity variation as a function of frequency.
\( \phi_2 \) is the shifted phase. Since the ultrasonic signal traverses twice through the sample the longitudinal phase velocity is calculated using twice the measured thickness. Because a reference waveform was used, the phase velocity was directly calculated from the measured time delay and energy flux deviations were ignored [23]. An example of phase velocity variation as a function of frequency is shown in figure 3.16. This plot indicates a uniform velocity and thereby indicates little dispersion.

The magnitude plot of figure 3.14 indicates that most of the echo energy peaks around 3 megahertz. Because of this, the longitudinal velocity was computed by averaging the phase velocities out to 4 megahertz. The imaged longitudinal velocity results are shown in figures 3.17 and 3.18. Again the grid areas correspond to the destructively tested areas. Figure 3.19 shows the destructively tested velocity images with a spatial sample size of 1.27 x 1.27 cm. These images were calculated using a single averaged thickness value for a given test plate. The velocity images in figure 3.20 shows the results of the destructively tested regions with the thickness correction using the corresponding thickness images of figure 3.3. Plotting the destructively determined FVF with the measured velocity shows a decrease in velocity as the FVF increases. This indicates that variations in the FVF are being overshadowed by the presence of
significant porosity levels. Because of this, the destructively determined porosity is plotted as a function of velocity and the Smith model [32] with porosity is fitted to the data. The ultrasonic measurement uncertainty is approximately +/- 1 percent and the destructive testing error is about +/- 1 percent. The fit parameter used is the matrix modulus of elasticity. The fitted model with the data is shown in figure 3.21. The matrix modulus of elasticity value obtained from the fit was $1.5 \times 10^{11}$ Dyne/cm^2. This value was used in the model to generate measured FVF and porosity results. The other values used to calculate a measured FVF and porosity are given in Table 3.
0.4945 cm/μsec  
60-A

0.2550 cm/μsec  
60-B

60% 16 ply velocity image avg = 0.3661 cm/μsec, sd = 0.015

0.4816 cm/μsec  
65-A

0.2929 cm/μsec  
65-B

65% 16 ply velocity image avg = 0.3408 cm/μsec, sd = 0.019

Figure 3.17: Ultrasonic velocity images of 60 and 65 percent composite test samples.
Figure 3.18: Ultrasonic velocity images of 70 percent 16 and 32 ply composite test samples.
Figure 3.19: Ultrasonic velocity images of areas destructively tested with spatial sample size is 1.27 x 1.27 cm.
Figure 3.20: Ultrasonic velocity images of areas destructively tested with thickness variations corrected, spatial sample size is 1.27 x 1.27 cm.
Figure 3.21: Model estimation of porosity.

Table 3: Material property values used in ultrasound model, matrix modulus

<table>
<thead>
<tr>
<th>PROPERTY</th>
<th>FIBER</th>
<th>MATRIX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transverse Elastic Moduli Dyne/cm²</td>
<td>$1.69 \times 10^{11}$</td>
<td>$1.50 \times 10^{11}$</td>
</tr>
<tr>
<td>Longitudinal Elastic Moduli Dyne/cm²</td>
<td>$2.21 \times 10^{12}$</td>
<td>$1.50 \times 10^{11}$</td>
</tr>
<tr>
<td>Longitudinal Poisson's Ratio</td>
<td>0.20</td>
<td>0.35</td>
</tr>
<tr>
<td>Transverse Poisson's Ratio</td>
<td>0.25</td>
<td>0.35</td>
</tr>
<tr>
<td>Transverse Shear Moduli Dyne/cm²</td>
<td>$6.76 \times 10^{10}$</td>
<td>$5.56 \times 10^{10}$</td>
</tr>
<tr>
<td>Longitudinal Shear Moduli Dyne/cm²</td>
<td>$9.21 \times 10^{11}$</td>
<td>$5.56 \times 10^{10}$</td>
</tr>
<tr>
<td>Transverse Bulk Moduli Dyne/cm²</td>
<td>$1.13 \times 10^{11}$</td>
<td>$1.67 \times 10^{11}$</td>
</tr>
<tr>
<td>Longitudinal Bulk Moduli Dyne/cm²</td>
<td>$1.23 \times 10^{12}$</td>
<td>$1.67 \times 10^{11}$</td>
</tr>
<tr>
<td>Reinforcing Factor</td>
<td>N/A</td>
<td>0.25</td>
</tr>
<tr>
<td>Density grams/cm³</td>
<td>1.85</td>
<td>1.30</td>
</tr>
</tbody>
</table>
of elasticity found from fit to data other values from [32, 40].
It is interesting to note that the difference between the matrix modulus of
elasticity and the transverse fiber modulus of elasticity is very small
thereby indicating that this method is more suited for porosity
measurements.

The X-ray measurements used in this study are described. Two
techniques were used, a digital Reverse Geometry X-ray™ technique using
a scintillation detector and conventional X-ray film technology. X-ray
techniques have potential to detect FVF variations because of the
differences in the absorption coefficients and density values of the fiber and
matrix at low energy levels. Two setups were used to perform the X-ray
measurements. The first setup used is Digiray's Reverse Geometry X-
ray™ system which uses a unique scanned X-ray source and scintillation
point detector for radiographic imaging of materials and structures. The
Reverse Geometry X-ray™ configuration is shown in figure 3.22. The
scanned X-ray source with a fixed detector significantly reduces the
contribution of scattered X-rays in the resulting image. This reduces the
blurring in the image, improving visualization of details in the structure as
compared to conventional real time systems. The second set used was the
conventional film setup shown in figure 3.23. To obtain quantitative
results the exposed film was converted into digital 8 bit format using a film scanner.

Figure 3.22: Reverse Geometry X-ray™ configuration.

Figure 3.23: Conventional X-ray film setup with digitizing scanner.
The Reverse Geometry X-ray™ system generated 16 bit images 1024 x 1024 in pixel size. To image differences in the fiber and matrix a low kilo-voltage level (37 kv) was used. Data acquisition was performed by averaging 256 images. A background image was also obtained to remove source unevenness. The conventional X-ray technique also used low kilo-voltage level of 30 kv. The exposure time was 3 minutes. The scanner digitized the exposed film at 8 bit depth resolution and pixel resolution of 140 dpi. The Reverse Geometry X-ray™ result is shown in figure 3.24. This image was produced by dividing the background to normalize the data. The outline of the sample is barely visible. In addition the .22 cm thick aluminum marker barely shows up. There seems to be quite a bit of noise and source unevenness present and because of this no analysis was performed on the images. The scanned film results are shown in figures 3.25 and 3.26. The destructively tested areas are marked. Quantitative results were not obtained using the digital scanner on the film results. This was because the contrast was enhanced during the capture and because of this no relative information was obtained from image to image.
Figure 3.24: Reverse Geometry X-ray™ image of 16 ply composite sample.
Figure 3.25: X-ray images of 60 and 65 percent composite test samples.
Figure 3.26: X-ray images of 70 percent 16 and 32 ply composite test samples.
Destructive testing was performed on the grided areas after all the NDE measurements and these results were used as the standard for comparison. The most widely used technique to determine FVF is by destructive matrix digestion. The procedure used in this study, standard D-3171-76, is described in the American Society for Testing and Materials (ASTM) Handbook [8]. All the measurements performed in this work will be compared to the destructive testing standard. This technique requires the removal of a small composite section to be destructively tested. The spatial sample size for this study was 1.27 x 1.27 cm. The destructive test result images of FVF and porosity are shown in figures 3.27 and 3.28 respectively. The total variation of the FVF from the destructive testing was from 56.0 to 70.5 percent. The total overall variation of porosity for all the destructive tests were 0.9 to 7.2 percent. The porosity was determined from equation 3.
Figure 3.27: Destructive FVF image results.
Figure 3.28: Porosity images obtained from destructive testing.
CHAPTER IV

QUANTITATIVE COMPARISON OF RESULTS

A comparison was performed based on the destructive test images being considered the known standard. A mean square difference metric was used to indicate how good the measured FVF images compared with the known standard destructive test images. This was calculated using the following equation:

\[
msd = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (X_i - Y_i)^2}
\]

where \(i\) is the pixel number, \(N\) is the total number of pixels within an image, \(X_i\) is the destructive test image and \(Y_i\) is the measured FVF image.

Based on this metric the best nondestructive technique to measure FVF was chosen. The results are given in figures 4.1 through 4.12 for the test images 60A through 70A2 respectively. This metric was calculated for thermally measured FVF, thermally measured FVF with localized thickness
correction, thermally measured FVF with thickness and destructive porosity correction, ultrasonically measured FVF, ultrasonically measured FVF with thickness correction, and ultrasonically measured FVF with thickness and destructive porosity correction. In addition since the ultrasonic measurement was dominated by the presence of significant porosity, this metric was also calculated between destructively measured porosity and ultrasonically measured porosity. The porosity imaging results are given in the figures 4.13 through 4.18 for the test images 60A through 70A2 respectively. No FVF calculations were performed for either X-ray method due to the low signal to noise and automatic contrast enhancement. A summary of the results are shown in Table 4 showing the calculated mean square difference values for the corresponding images and

<table>
<thead>
<tr>
<th>IMAGE</th>
<th>Thermal FVF +/ -</th>
<th>Thermal FVF thick +/ -</th>
<th>Thermal FVF thick/por +/ -</th>
<th>Ultrasound FVF +/ -</th>
<th>Ultrasound FVF thick +/ -</th>
<th>Ultrasound FVF thick/por +/ -</th>
</tr>
</thead>
<tbody>
<tr>
<td>60A</td>
<td>0.014</td>
<td>0.005</td>
<td>0.005</td>
<td>0.068</td>
<td>0.046</td>
<td>0.135</td>
</tr>
<tr>
<td>60B</td>
<td>0.020</td>
<td>0.006</td>
<td>0.005</td>
<td>0.063</td>
<td>0.051</td>
<td>0.058</td>
</tr>
<tr>
<td>65A</td>
<td>0.011</td>
<td>0.012</td>
<td>0.012</td>
<td>0.220</td>
<td>0.184</td>
<td>0.35</td>
</tr>
<tr>
<td>65B</td>
<td>0.013</td>
<td>0.025</td>
<td>0.025</td>
<td>0.290</td>
<td>0.32</td>
<td>0.42</td>
</tr>
<tr>
<td>70A</td>
<td>0.020</td>
<td>0.012</td>
<td>0.011</td>
<td>0.26</td>
<td>0.29</td>
<td>0.26</td>
</tr>
<tr>
<td>70A2</td>
<td>0.035</td>
<td>0.029</td>
<td>0.029</td>
<td>0.25</td>
<td>0.27</td>
<td>0.26</td>
</tr>
<tr>
<td>AVG</td>
<td>0.0188</td>
<td>0.0148</td>
<td>0.0145</td>
<td>0.192</td>
<td>0.194</td>
<td>0.247</td>
</tr>
</tbody>
</table>

Table 4: Calculated mean square difference values for the corresponding images and FVF measurement technology.
Table 5: Calculated mean square difference values for the corresponding images and measurement technology.

FVF measurement technology. A summary of the results are also shown in Table 5 for the ultrasonically measured porosity compared to the destructive porosity. Also included in Table 5 is a final result where the thermal and ultrasonic measurements were combined. The thermal measurements were used to measure FVF and the ultrasonic porosity measurements where used in the thermal model to account for the porosity effects on the thermal diffusivity. The imaged results are shown in figures 4.19 through 4.24. The numerical results are given in the last column of Table 5.
Figure 4.1: Comparison of thermally measured FVF for 60A sample.
Figure 4.2: Comparison of ultrasonically measured FVF for 60A sample.
Figure 4.3: Comparison of thermally measured FVF for 60B image sample.
Figure 4.4: Comparison of ultrasonically measured FVF for 60B image sample.
Figure 4.5: Comparison of thermally measured FVF for 65A image sample.
Figure 4.6: Comparison of ultrasonically measured FVF for 65A image sample.
Figure 4.7: Comparison of thermally measured FVF for 65B image sample.
Figure 4.8: Comparison of ultrasonically measured FVF for 65B image sample.
Figure 4.9: Comparison of thermally measured FVF for 70A image sample.
Figure 4.10: Comparison of ultrasonically measured FVF for 70A image sample.
Figure 4.11: Comparison of thermally measured FVF for 70A2 image sample.
Figure 4.12: Comparison of ultrasonically measured FVF for 70A2 image sample.
Figure 4.13: Comparison of measured porosity for 60A image sample.
Figure 4.14: Comparison of measured porosity for 60B image sample.
Figure 4.15: Comparison of measured porosity for 65A image sample.
Figure 4.16: Comparison of measured porosity for 65B image sample.
Figure 4.17: Comparison of measured porosity for 70A image sample.
Figure 4.18: Comparison of measured porosity for 70A2 image sample.
Figure 4.19: Comparison of thermal/ultrasonic measured FVF for 60A.
Figure 4.20: Comparison of thermal/ultrasonic measured FVF for 60B.
Figure 4.21: Comparison of thermal/ultrasonic measured FVF for 65A.
Figure 4.22: Comparison of thermal/ultrasonic measured FVF for 65B.
Figure 4.23: Comparison of thermal/ultrasonic measured FVF for 70A.
Figure 4.24: Comparison of thermal/ultrasonic measured FVF for 70A2.
A comparison of the imaging technologies investigated, thermal, ultrasonic, and X-ray, was completed for the imaging of FVF in T300/934 graphite epoxy plates. A theoretical model for each measurement technology was used to relate the physical property measurement (e.g. ultrasonic velocity, diffusivity) to the FVF. For practical applications to varied ply orientations, measurements were made where no a priori knowledge of the ply lay-up was required. Images were generated of the measured FVF and these results were compared to the destructive test FVF images using a mean square difference metric. The errors in this study were caused by two main sources random and fixed. The random errors were due to mainly to the analog/digital conversion and the associated electronics. The fixed errors were due to the model used. The ability of the models to accurately describe energy propagation is important. For example the shape of the voids was not taken into consideration. Also inaccurate values of material parameters can contribute to this type of error. This error was considered greater than the random errors.
On the basis of the mean square difference metric it was found that the thermal technique provided the best agreement, by a factor of 10, as compared to the ultrasonic velocity measurement. It was found the ultrasonic velocity measurement was more sensitive to porosity. The significant levels of porosity was introduced into the samples during prebleeding of the plies for higher FVF. The large errors resulting from the FVF measurement using ultrasonics was due to the insensitivity of the technique to measure FVF even when porosity and thickness was taken into account. Both X-ray technologies, Reverse Geometry X-ray™ and standard film technology did not provide any quantitative information in this study. The thickness corrections proved to be of some use, however, only benefiting the thermal technique slightly. Based on these findings the thickness mapping did not seem feasible for small variations in thickness considering the amount of effort required. A plot is shown in figure 5.1 comparing the different techniques to the destructive test results where no thickness mapping information was used. This is the case for most NDE applications where small variations of thickness is encountered. It is interesting to note that when combining the thermal FVF measurement with the ultrasonic porosity measurement only a small decrease in the mean square difference of +/- 0.004 was encountered.
Finally, the final important points of this study are stated below:

- Thermal measurement of FVF proved to be better by a factor of 10 over longitudinal velocity ultrasonics.

- Difference in transverse modulus between 934 resin and T-300 fiber was not significant enough to measure FVF ultrasonically.

- Because of the small difference in transverse modulus between 934 resin and T-300 fiber the ultrasonic velocity measurement proved more useful in measuring porosity.
• Mapping thickness variations of less than +/- 2 percent did not significantly prove worthwhile in reducing the mean square difference.

• For high porosity areas greater than 5 percent the ultrasonic correction into the thermal model did not significantly improve agreement with the destructive images indicating the thermal model is not sufficient for high porosity.

• The combined technique of using ultrasonic velocity to measure porosity and using that information into the thermal model proved best overall in measuring FVF for porosity levels less than 5 percent.

• Finally, curve fits were used to determine material properties for the matrix from the destructive data. The property values obtained from the fits are dependent on the models used and were considered approximate values.

Based on these findings, a combined thermal and ultrasonic nondestructive approach for mapping FVF proved best. Future work in improving this technique would involve several areas. The first area would be an independent measurement of the material properties of the
fiber and matrix. This would allow the determination of the best theoretical model to predict FVF. Another area would be to develop a more accurate thermal model. This may help improve agreement with destructive test results for higher porosity levels.

These results can benefit the Army. As new composite weapon systems are developed, structural testing will be required. New designs are often fabricated into prototypes using finite element modeling. Often, structural testing is combined with finite element modeling to determine model agreement. One example, for the use of this technology is the mapping of FVF and porosity as input into a finite element model to accurately simulate the composite’s performance. This information could be used for design optimization studies.
APPENDIX A

THERMAL RESPONSE

The thermal response of the composite was determined by applying flashed radiant heating at one surface \((x = 0)\) and negligible convection losses. The derivation for back surface temperature response is given as follows. The one dimensional heat flow is described by the following equation:

\[
\frac{\partial T(x, t)}{\partial t} = \alpha \frac{\partial^2 T(x, t)}{\partial x^2}
\]  

with boundary conditions:

\[
\frac{\partial T(x, t)}{\partial x} = \delta(t) \quad \text{for} \; x = 0
\]  

and
\[ \frac{\partial T(x,t)}{\partial x} = 0 \quad \text{for } x = 1 \] (3)

where \( l \) is the layer thickness, \( \alpha \) is the effective layer thermal diffusivity, and \( T(x,t) \) is the temperature. A solution for the temperature decay at the back surface due to an impulse input at the front surface \( (x = 0) \) is given below as:

\[ T(1, t) = \frac{Q}{\rho c l} \left( 1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp \left[ -\frac{n\pi^2}{l^2} \alpha t \right] \right). \] (4)

Where \( Q \) is the energy per unit area and \( \gamma \eta \varphi_{\text{fiber}}^{\text{trans}} \rho c \) is the volumetric heat capacity. Normalization of the temperature data is usually done to reduce heating or emissivity variations [41]. Dividing by the maximum temperature gives the normalized heat up temperature response expressed from the previous equation as:

\[ T_N(1, t) = 1 + 2 \sum_{n=1}^{7} (-1)^n \exp \left[ -\frac{n\pi^2}{l^2} \alpha t \right]. \] (5)
For practical reasons the equation was summed to the first seven terms. The normalized equation is a function of diffusivity and the sample thickness. The diffusivity can be related to the FVF in a composite using a simplified model where the heat flow through a plate is perpendicular to the fiber and matrix. The composite's volumetric heat capacity can be stated from [42] using the law of mixtures as

\[
(\rho c)_{\text{avg}} = (V_f)(\rho c)_{\text{fiber}} + (1 - V_f - V_{\text{por}})(\rho c)_{\text{matrix}}
\] (6)

where \( \rho \) is the density, \( c \) is the specific heat, \( V_{\text{por}} \) denotes percentage volume fraction of porosity, and \( V_f \) denotes percentage volume fraction of fiber. Since the volumetric heat capacity of air is small it does not factor into the equation. In this study three main theories were compared to calculate an equivalent thermal conductivity for the effective composite thermal diffusivity value. These models were quantitatively compared to the destructive tests and the best model was chosen. These models were for heat conduction perpendicular to the fiber. The first model is the series model or stacked plate model (SPM). The simple series model case is for two different types of layered structures stacked in a laminated fashion. This is analogous to two resistors in series. The equation for this model is given from Nusslet [43] as:
\[
\kappa_{\text{equiv}} = \frac{\kappa_{\text{fiber}} \kappa_{\text{matrix}}}{\kappa_{\text{matrix}} V_f + \kappa_{\text{fiber}} (1 - V_f)}.
\] (7)

Where \( \kappa_{\text{fiber}} \) and \( \kappa_{\text{matrix}} \) are the thermal conductivity of the fiber and matrix respectively. The term \( V_f \) is the percent volume fraction of the fiber. The second model was derived by Springer and Tsai [44] and is known as the square packing array model (SPAM). This model takes into account the dispersed fibers within a matrix. The fibers are assumed to be square filaments. The equivalent thermal conductivity is then given below where the heat conduction is perpendicular to the fibers.

\[
\kappa_{\text{equiv}} = (1 - \sqrt{V_f}) \kappa_{\text{matrix}} + \frac{\kappa_{\text{matrix}} \sqrt{V_f}}{(1 - \sqrt{V_f}) (1 - \frac{\kappa_{\text{matrix}}}{\kappa_{\text{fiber}}})}.
\] (8)

The last model is the Composite Circular Assemblage model derived by Hashin and Rosen [4] and used by Charles and Wilson [39]. This model takes into account the circular geometry of the filament. The equation is given in (9).
\[ k_{\text{equiv}} = k_{\text{matrix}} \frac{k_{\text{matrix}} (1 - V_f) + k_{\text{fiber}} (1 + V_f)}{k_{\text{matrix}} (V_f + 1) + k_{\text{fiber}} (1 - V_f)} \] (9)

Since the thermal diffusivity is calculated from the equivalent thermal conductivity, the diffusivity is plotted versus FVF with the same material properties. This plot is shown in Figure 1 where the bottom line (SPM) is the Nusslet model, the middle is the Springer Tsai model (SPAM), and the top (CCA) plot is the Hashin and Rosen model. All three models were plotted with the same conductivity values. As expected, for all three models, as the volume amount of fibers increase the diffusivity also increases since the fibers are a better heat conductor relative to the resin.

**Figure 1:** Comparison of thermal diffusivity from different models.
The CCA and the SPAM models are very close whereas the stacked plate model predicts lower values. Chamis [40] shows that the thermal conductivity can include porosity by solving for the equivalent matrix thermal conductivity between the matrix and air voids. The CCA model is used in a similar manner to solve for the porosity effects on the matrix. Once solving for the matrix thermal conductivity with air voids, this can be used with the fiber thermal conductivity to find the thermal conductivity for the composite. By taking into account the voids, the effect is to lower the diffusivity since the air also doesn’t conduct heat as well as the fibers. A comparison plot of the effect of voids in thermal diffusivity is shown in figure 2 where the CCA model is used. The top plot is for 0.0 percent air voids, the middle is for 4.0 percent voids, and the bottom plot is for 8 percent voids. The diffusivity is lowered as the amount of voids increase. From the previous equation (5) the normalized temperature response
Figure 2: Porosity effect on diffusivity for FVF.

as a function of FVF using the desired thermal conductivity equation can then be stated as:

\[ T_N(1,t) = 1 + 2 \sum_{n=1}^{7} (-1)^n \exp\left[-\frac{n\pi^2}{1^2} \frac{k_{\text{equiv}}}{(\rho c)_{\text{avg}}} t \right] \]

This result shows the normalized temperature response as a function of time is dependent on the FVF.
APPENDIX B

ULTRASONIC LONGITUDINAL VELOCITY RESPONSE

The longitudinal velocity is calculated for a wave propagating in the direction perpendicular to the fibers. By measuring only this component of the velocity, ply orientations should have minimal effect on the propagation because the wave will always be polarized perpendicular to the fibers. The equation for the longitudinal velocity is given below:

\[ V_1 = \frac{\omega}{k} = \sqrt{\frac{c_{11}}{\rho}}. \]  

(1)

The theory used to relate the \( c_{11} \) elastic constant to FVF is found by combining the work of Hashin [31] who takes into account the effect of voids in the matrix with Martin [30] and Smith [32]. The derivations are determined using variational theorems of elasticity theory and are given in the references. The fibers are considered transversely isotropic for this theory. The equations relating the \( c_{11} \) elastic constant to the FVF and
porosity shown below are primarily obtained from Smith [32] and is given for reference.

\[ K_{m_o} = \frac{E_{m_o}}{3 (1 - 2v_m)} \]  
(2)

\[ G_{m_o} = \frac{E_{m_o}}{2 (1 + v_m)} \]  
(3)

For the matrix the bulk K and shear G modulii are given as follows without voids. Where \( K_{m_o} \) is the bulk modulus without voids, \( G_{m_o} \) is the shear modulus without voids, \( E_{m_o} \) is the modulus of elasticity without voids, and \( v_m \) is the matrix Poisson ration. The matrix is assumed to contain the voids (spherical) and the corresponding equations of the bulk \( K_m \), shear \( G_m \), and elastic \( E_m \) modulii with voids are given below as:

\[ K_m = K_{m_o} \left(1 - \frac{3 (1 - v_m)}{2 (1 - 2v_m)}P\right) \]  
(4)

\[ G_m = G_{m_o} \left(1 - \frac{15 (1 - v_m)}{(7 - 5v_m)}P\right) \]  
(5)
and

\[
E_m = E_{m_0} \frac{(3K_{m_0} + G_{m_0})}{[2(1-2\nu_m)(7-5\nu_m)-(51-75\nu_m)(1-\nu_m)P]} \frac{[2(1-2\nu_m)(7-5\nu_m)-(51-75\nu_m)(1-\nu_m)P]}{[2(1-2\nu_m)(7-5\nu_m)(3K_{m_0} + G_{m_0})-(1-\nu_m)P](9K_{m_0}(7-5\nu_m)+3G_{m_0}(1-2\nu_m))}
\]

(6)

where

\[
P = 1 - \frac{\rho_m}{\rho_{\text{comp}}}. \tag{7}
\]

\(P\) is the porosity calculated from \(\rho_m\) the matrix density and \(\rho_{\text{comp}}\) the composite density. Also \(K_{m_0}\) is the bulk modulus without voids, \(E_{m_0}\) is the modulus of elasticity without voids, and \(\nu_m\) is the Poisson ration of the matrix. The corresponding \(c_{11}^{\text{matrix}}\), \(c_{12}^{\text{matrix}}\), and \(c_{44}^{\text{matrix}}\) used to compute the overall \(c_{11}\) elastic constant is given below.

\[
c_{11}^{\text{matrix}} = \frac{E_m (1 - \nu_m)}{(1 - 2\nu_m)(1 + \nu_m)} \tag{8}
\]
\[
\begin{align*}
  c_{\text{matrix}}^{12} &= \frac{E_m v_m}{(1 - 2v_m)(1 + v_m)} \\
  c_{\text{matrix}}^{44} &= \frac{(c_{\text{matrix}}^{11} - c_{\text{matrix}}^{12})}{2} \\
  \text{The corresponding } c_{\text{fiber}}^{11}, c_{\text{fiber}}^{12}, \text{ and } c_{\text{fiber}}^{66} \text{ used to compute the overall effective } c_{11} \text{ elastic constant is given below.} \\
  c_{\text{fiber}}^{11} &= \frac{E_{\text{fiber}}^{\text{trans}}(1 - v_{\text{fiber}}^{\text{trans}})}{(1 - 2v_{\text{fiber}}^{\text{trans}})(1 + v_{\text{fiber}}^{\text{trans}})} \\
  c_{\text{fiber}}^{12} &= \frac{E_{\text{fiber}}^{\text{trans}}v_{\text{fiber}}^{\text{trans}}}{(1 - 2v_{\text{fiber}}^{\text{trans}})(1 + v_{\text{fiber}}^{\text{trans}})} \\
  c_{\text{fiber}}^{66} &= \frac{(c_{\text{fiber}}^{11} - c_{\text{fiber}}^{12})}{2}
\end{align*}
\]

Where \( E_{\text{fiber}}^{\text{trans}} \) is the fiber transverse elastic modulus and \( v_{\text{fiber}}^{\text{trans}} \) is the fiber transverse Poisson ratio. The equation used to calculate the effective \( c_{11} \) modulus from Smith [32] is given as:
\[
\begin{align*}
C_{11} &= \frac{1}{2} (C_{11}^{\text{matrix}} + C_{12}^{\text{matrix}}) + (C_{11}^{\text{fiber}} + C_{12}^{\text{fiber}} - C_{11}^{\text{matrix}} - C_{12}^{\text{matrix}}) V_f \\
&\quad - \frac{1}{2} \left( \frac{(C_{11}^{\text{fiber}} + C_{12}^{\text{fiber}} - C_{11}^{\text{matrix}} - C_{12}^{\text{matrix}})^2 V_f (1 - V_f)}{(C_{11}^{\text{fiber}} + C_{12}^{\text{fiber}} - C_{11}^{\text{matrix}} - C_{12}^{\text{matrix}})} + C_{44}^{\text{matrix}} \left( \frac{1 + \xi \eta V_f}{1 - \eta V_f} \right) \right) + C_{66}^{\text{fiber}} + \xi C_{44}^{\text{matrix}}.
\end{align*}
\]  

The term \( \eta \) is the FVF and \( \xi \) is the matrix reinforcing factor. The term \( \eta \) is given below as:

\[
\eta = \frac{C_{66}^{\text{fiber}} - C_{44}^{\text{matrix}}}{C_{66}^{\text{fiber}} + \xi C_{44}^{\text{matrix}}}
\]  

The \( \xi \) is the matrix reinforcing factor. These results show the longitudinal velocity dependency on FVF independent of lay-up. It can be shown from the model that the dependency of FVF on longitudinal velocity is heavily dependent on the difference between the transverse shear modulus of the fiber and matrix. Figure 1 shows the variation of longitudinal velocity as a function of FVF with 2, 5, 10 factor differences in the transverse shear modulus between the fiber and matrix. Figure 2 shows the variation of longitudinal velocity as a function of FVF and porosity for a factor of 7 difference between the transverse shear modulus of the fiber and matrix.
Figure 1: Factor effects on longitudinal velocity variation with FVF.

Figure 2: Porosity effect on longitudinal velocity and FVF.
NOTES


BIBLIOGRAPHY


VITA

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