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Non-Polluting Composites Repair and Remanufacturing for Military Applications: Induction-Based Processing

by Bruce K. Fink, Nicholas B. Shevchenko, James M. Sands, Shridhar Yarlagadda, and John W. Gillespie, Jr.

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Abstract

The development of induction-based processing of carbon-fiber (CF) thermoplastic-matrix composites and accelerated cure of thermosetting adhesives has the potential to provide nonautoclave processing technology for manufacturing and repair of polymer-matrix composites (PMCs). In this report, the results of recent tests demonstrating bonding of composites using commercial off-the-shelf thermal-cure adhesives that are heat cured via an induction field using an inductive susceptor are discussed. This method of cure utilizes heat generation within metal screen-based susceptors to cure the adhesive matrix via a heat transfer mechanism. The mechanical performance of these bonds is presented in comparison with autoclave and thermally cured baselines. No substantial loss of mechanical lap-shear strength is observed in adhesive bonds processed by induction. In addition, an example of induction welding of a thermoplastic-impregnated carbon fiber (AS4) is presented. In order to successfully demonstrate induction welding for manufacture of CF composites, the degradation of the polymer in the laminates is also investigated. No measurable degradation of the polymer, either by dielectric or thermal breakdown when heated by induction, was observed.

Acknowledgments

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1. Introduction

The U.S. Army is currently pursuing the development of low-cost nonautoclave manufacturing technologies for polymer-matrix composites (PMCs) that will allow the U.S. military and contractors to prepare high-performance composites for armor, munition, and vehicle applications with reduced costs, reduced environmental impact, and increased efficiency. The authors have previously introduced the concept of induction-based processing and the environmental impacts for Army applications [1]. The initial repair demonstrations involved heat generation using a metallic (electrically conductive) mesh to translate electromagnetic energy into thermal energy at an adhesive bond line. In this report, the background and physics of induction processing in carbon-fiber thermoplastic laminates is reviewed and the successful development of induction heating that allows suitable repair of many composite structures is discussed. Specifically, the adhesive bonding using susceptor-based induction heating, susceptorless induction-based carbon-fiber laminate consolidation, and development of process models for electromagnetic heating of carbon-fiber composites, including heat generation by carbon fibers, metal-mesh susceptors, and magnetoresistive particles is discussed.

2. Induction-Based Repair and Remanufacturing

The search for cost-effective environmentally friendly manufacturing methods has led to the study of induction heating for bonding and processing of composites [1]. Electromagnetic cure methods involve using induction or electrical resistance heating focused directly at the material to be cured. Induction heating occurs when a current-carrying body, or coil, is placed near another conductor, the susceptor material. The magnetic field caused by the current in the coil induces a current in the susceptor. This induced current causes the susceptor to heat due to Joule heating, and, in the case of a ferromagnetic material, due to hysteresis losses. Carbon-fiber reinforcement in composite materials can function as the susceptor. For other material systems, the susceptor is a metallic mesh or magnetic particles. Energy can be introduced into the precise region to be cured both in the plane of the structure and at the specific depth required [2].

The ability of induction heating to rapidly process carbon-fiber-based thermoplastic composites is a significant environmental asset. Assuming that the thermoplastic composite meets the performance and quality requirements of an equivalent thermoset counterpart, one can replace the limited shelf-life thermoset with the unlimited shelf-life thermoplastic. This completely eliminates hazardous raw material wastes at the production level resulting from shelf-life expiration and raw-material overages associated with thermoset-based composites production.

Other advantages of induction include reduction of volatile organic compounds (VOCs) and nitrous oxide (NO_x) emissions by processing out of the autoclave, localizing processing controls for repair and remanufacturing, and eliminating processing steps. In addition, induction offers internal noncontact heating; the possibility of a moving heat source (the coil) to heat large areas; high efficiency; control of the heat generation by coil design or by susceptor design; and powerful, portable, and easy-to-operate units [3, 4].

2.1 Induction-Based Repair of Multifunctional Composite Armor

This research is motivated by the expanding use of multifunctional hybrid materials in military ground vehicles and the increasing need for field-expedient and depot-level repair procedures for these thick-section components.

The critical issue in adhesive-based repair of composites is the application of sufficient heat and pressure at the bond line. It is highly desirable that thermal generation be localized at the bond line and be evenly distributed (taking into account thermal conductive losses). This is especially important with the increasing use of multifunctional hybrid composites, such as composite armor. These composites typically have several layers, each serving a different function, as shown in Figure 1. Repair of such a thick-section composite will require heating locally at the appropriate bond lines; one such method is induction heating [5, 6]. In addition, due to the noncontact nature of induction heating, it may be possible to bond several layers at the same time, which reduces hazardous waste, energy consumption, and repair times for the part. Conventional repair techniques (e.g., heat blankets) will require bonding of one layer at a time, resulting in multiple potential hazardous waste streams such as trim, consumables, and VOC emissions.

While the induction-based repair procedure has the potential to reduce hazardous waste, it is essential that the repaired part meet the performance requirements dictated by the application. Hence, the initial work done under the program focuses on evaluating the performance of induction-based repair procedures. The goal is to obtain performance similar to that achieved with conventional repair procedures.

Recent studies [1, 7, 8] have shown similar properties for induction-heated adhesive bonds compared to baselines for room-temperature adhesives. Electrically conductive mesh susceptors and epoxy-based adhesives were used.

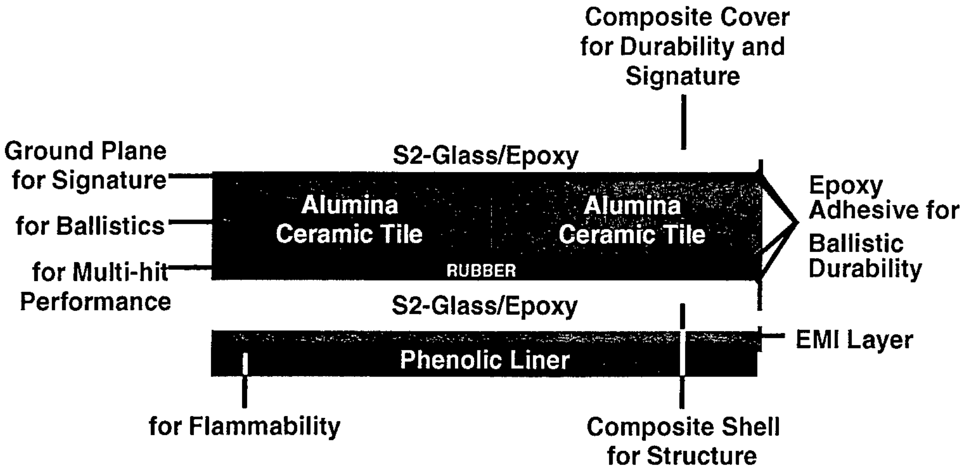


Figure 1. Multifunctional composite armor.

A methodology that enables cure cycles to be related to degree-of-cure predictions for accelerated curing of adhesives was established.

The present work extends this effort to high-temperature-curing adhesive systems because most military applications require high-performance adhesives, which typically are high-temperature-curing systems (250 °F and 350 °F thermosetting adhesives). A carbon/epoxy substrate system was used to study susceptorless induction bonding and assess the effect of the mesh or eddy-current susceptor on bond strengths. A glass/vinyl-ester substrate was also used for bond strength tests, due to the increasing use of such systems in military vehicles. The glass/vinyl-ester composite was fabricated by vacuum-assisted resin-transfer molding (VARTM), which utilizes fiber preforms and resin systems and has no shelf-life requirements.

2.1.1 Material Systems

Three adhesive systems were considered in this study: SW-2214 and EC-1386 from 3M, and FM300K film adhesive from American Cyanamid. Recommended cure cycles are 60 min at 250 °F (SW-2214) and 60 min at 350 °F (EC-1386 and FM300K). Recommended pressures during the cure are approximately 25 psi for SW-2214 and EC-1386, and 40 psi for FM300K.

Two composite systems were chosen for this study: HG-Cycom 919GF carbon/epoxy prepreg, and E-glass/vinyl ester fabricated via VARTM. Carbon/epoxy panels consisting of 16 plies were laid-up and cured in the autoclave based on the manufacturer's recommendations. The thickness of the cured panel was approximately 4.0 mm. The glass/vinyl ester was fabricated by VARTM at room temperature and post-cured at 250 °F for 1 hr to ensure complete cure of the resin system.

The susceptor material used in this study was a stainless steel mesh with a density of 30 × 30 boxes per square inch and a wire diameter of 0.0075 in. The mesh density and wire diameter were chosen based on prior testing and experience [7–10]

2.1.2 Induction Bonding Setup

The initial effort demonstrated the ability to bond one lap-shear specimen at a time, as compared to American Society for Testing of Materials (ASTM) Standard D1002 [11] methodology which calls for bonding of two laminates and subsequent machining to lap-shear test specimen dimensions. In this effort, the U.S. Army Research Laboratory (ARL) Strategic Environmental Research and Development (SERDP) program induction setup [1] was modified by mounting the induction coil on a robotic head. This allowed for three-dimensional (3-D) programmed coil motion, at a specified velocity resulting in the bonding of two composite panels at a time rather than a single lap-shear specimen. The substrate was cut into pairs of panels 7 in × 4.5 in. The bonded panels were subsequently machined to six lap-shear specimens. Figure 2 shows the specimen configuration during induction bonding.

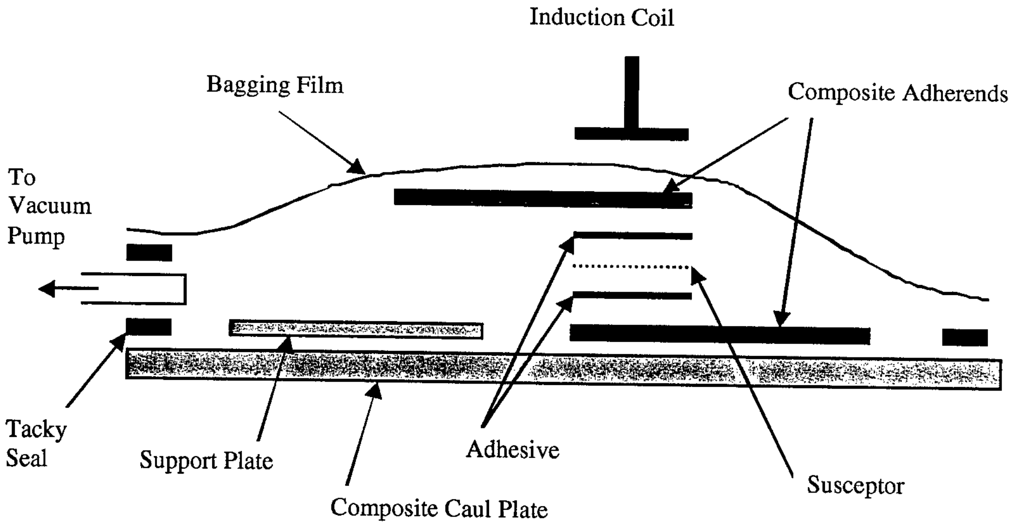


Figure 2. Specimen configuration during induction bonding.

Figure 3 shows the coil motion direction on the bagged specimen. The motion pattern was programmed into the robot. The velocity of the robot head was a variable that could be adjusted to achieve thermal uniformity along the motion path. For this effort, a velocity of 10 mm/s was chosen after initial trials on thermal uniformity. Figure 4 shows a typical infrared (IR) temperature profile

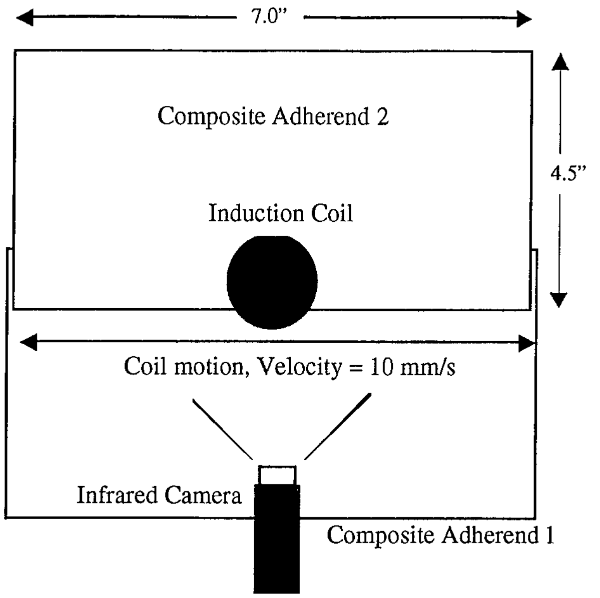


Figure 3. Induction coil motion direction and configuration.

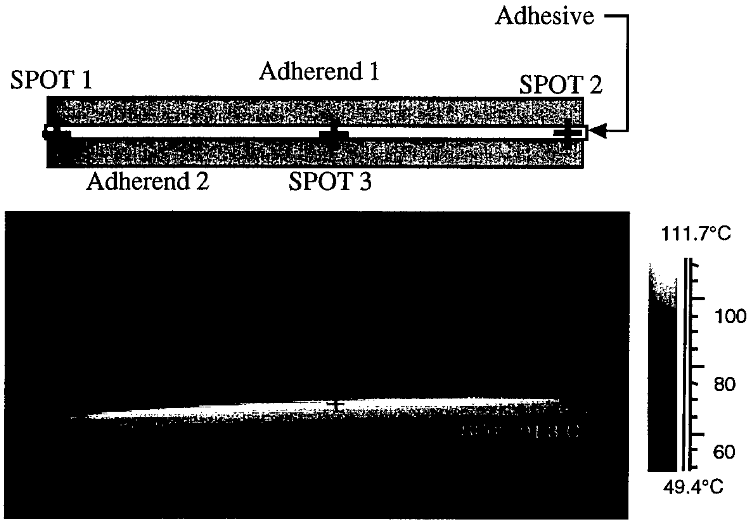


Figure 4. Typical bondline temperature profile along the coil motion direction.

along the motion direction of the coil, and differentials of up to ±10 °C were noted based on coil location. The temperatures shown are steady-state surface temperatures of the substrates or adherends. Material performance tests with these temperature differentials will help establish process windows for induction bonding. For the carbon/epoxy system, surface temperatures did not differ significantly from bond line temperatures due to the good thermal conduction of carbon fibers; this was confirmed by thermocouple measurements. For glass/vinyl ester, differentials of up to ±10 °C between the bond line and surface were noted. Further refinements to determine the ideal motion velocity and pattern for minimal temperature gradients are in progress.

For a carbon/epoxy substrate or adherend, no susceptor is necessary. However, bonding tests were performed for both cases (i.e., with and without the mesh susceptor) to assess the presence of the mesh on bond strengths. For the carbon/epoxy substrate, all three adhesive systems were induction bonded, with and without the mesh. For the glass/vinyl-ester system, the SW-2214 adhesive was induction bonded with the mesh, as the cure cycle of the other systems (350 °F) could cause degradation in the substrate. All the induction-bonded specimens were fabricated under vacuum pressure. Table 1 lists the baselines; Table 2 lists the test cases.

Table 1. Baselines for comparison with induction bonding.

Case	Substrate	Number of Specimens	Adhesive	Cure Cycle
A B	Carbon/Epoxy	6	SW-2214 SW-2214 + mesh[a]	250 °F, Vacuum 60 min
A B	Carbon/Epoxy	6	EC-1386 EC-1386 + mesh	350 °F, Vacuum 60 min
A B	Carbon/Epoxy	6	FM300K FM300K + mesh	350 °F, Vacuum 60 min
E F			FM300K FM300K + mesh	350 °F, 40 psi 60 min
G	Glass/Vinyl Ester	6	SW-2214 SW-2214 + mesh	250 °F, Vacuum 60 min

[a] Mesh = SS304, 30 × 30, 0.0075-in wire.

Baseline lap-shear specimens were fabricated according to the manufacturer's recommended cure cycles. For the FM300K adhesive system, two pressures were considered: vacuum (~15 psi) and 40 psi. For all other autoclave specimens, vacuum pressure was used. This allows for direct comparison with induction-bonded lap-shear tests, which were performed under vacuum pressure.

Test cases of single lap-shear (SLS) specimens were fabricated as described in Table 2. The adhesive bond-line thicknesses for the specimens were measured by means of a traveling microscope. In all cases where no susceptor or mesh was

Table 2. Test cases for induction bonding.

Case	Substrate	Number of Specimens	Adhesive	Cure Cycle
C D	Carbon/Epoxy	6	SW-2214 SW-2214 + mesh[a]	250 °F, Vacuum 60 min
C D	Carbon/Epoxy	6	EC-1386 EC-1386 + mesh	350 °F, Vacuum 60 min
C D	Carbon/Epoxy	6	FM300K FM300K + mesh	350 °F, Vacuum 60 min
H	Glass/Vinyl Ester	6	SW-2214 + mesh	250 °F, Vacuum 60 min

[a] Mesh = SS304, 30 × 30, 0.0075-in wire.

used, the thickness of the bond line was consistently about 0.002 in. The mesh caused an increase in bond-line thickness to about 0.013 in. These values were very consistent and did not appear to vary greatly between specimens. The effect of bond line thickness variation was not considered in the analysis, although it is recognized that this variable may be significant.

2.1.3 Mechanical Performance

All SLS specimens were tested to failure in an Instron universal testing machine. The mean nominal shear strengths and the associated error bars are shown in Figures 5, 6, and 7 for SW-2214, FM300K, and EC-1386, respectively. Six specimens were tested for each case, and in all cases cohesive failure of the adhesive layer was obtained. Degree of cure for each case was determined by differential scanning calorimetry (DSC) and was greater than 95% for all observed specimens.

In general, for all the adhesive systems, no loss in mechanical performance is noted, between induction-cured and autoclave baseline specimens. In some cases (Figures 5 and 7), the induction-cured specimens exhibit higher mean shear strengths, which may reflect the thinner bond line. The same trend is observed for the glass/vinyl-ester system (cases G and H in Figure 5). It is also interesting to note the relatively similar scatter in strength data between the induction-cured specimens and the autoclaved baselines, despite the temperature differentials during induction cure. For the FM300K film adhesive system, higher pressure during the cure cycle (40 psi) causes a significant increase in the bond strength but not in the presence of a mesh. However, the presence of the mesh does not seem to affect performance in vacuum-processed specimens. For the EC-1386 and SW-2214 paste adhesives, the mesh causes a significant drop in strength.

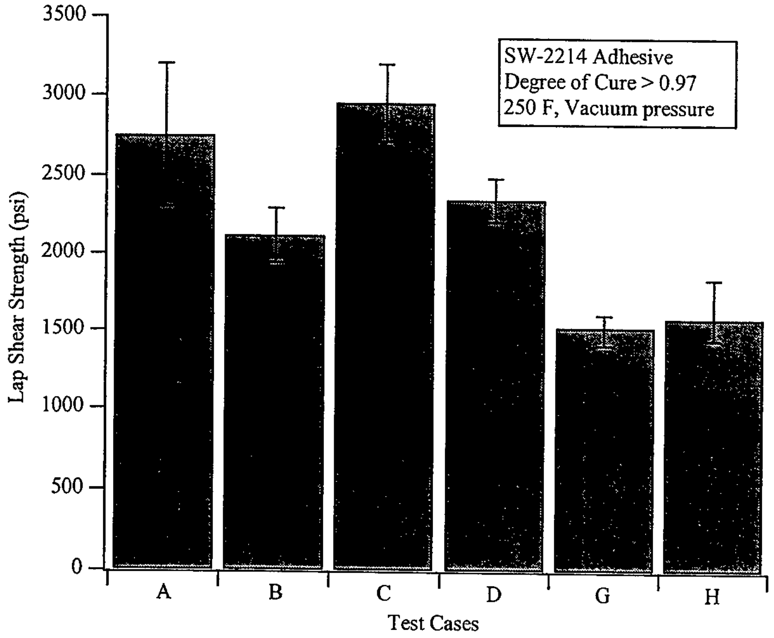


Figure 5. Mechanical performance for SW-2214 adhesive system cured at 250 °F. (A–H are defined in Tables 1 and 2).

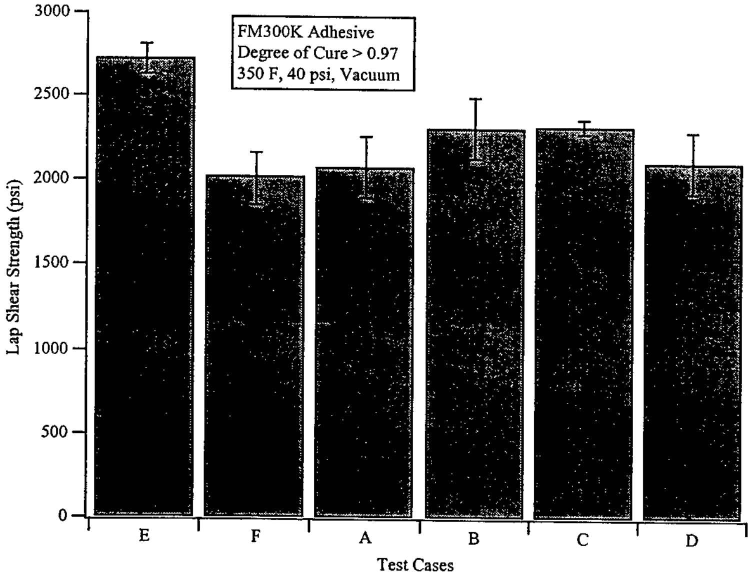


Figure 6. Mechanical performance for FM300K adhesive system cured at 350 °F.

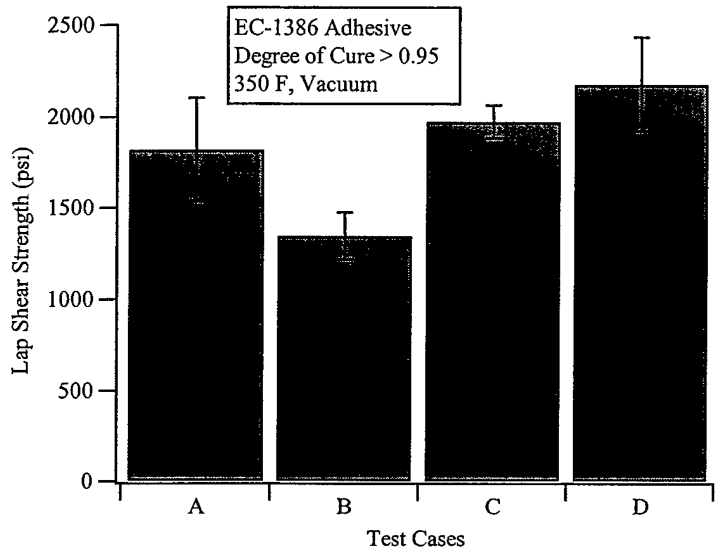


Figure 7. Mechanical performance for EC-1386 adhesive system cured at 350 °F.

2.1.4 Conclusions to Induction-Based Adhesive Bonding

Following the methodology established in the previous work [9] on thermoset adhesive cure, performance studies of high-temperature curing adhesive systems, due to induction cure, have shown no loss in bond strengths between baselines and induction processing. This study, along with the previous effort [9], validates induction cure as an alternative method for processing thermally curable adhesives while retaining baseline bond strengths. Advantages of induction cure stem from reduction of VOCs and NO_x emissions by processing out of the autoclave and an increased cure rate due to through-thickness heating. It also provides a unique ability to perform multimaterial repairs (as in composite armor) in a single step, thus eliminating process steps and corresponding hazardous waste.

2.2 Induction-Based Remanufacture of Thermoplastic Composite Laminates

The Department of Defense (DOD) has increased use of carbon-fiber-reinforced thermoset prepregs, resulting in a potentially significant hazardous waste stream due to shelf-life expiration and VOC emissions during processing [1]. It is estimated that the raw material requirements (i.e., prepreg) for one particular application will exceed one million pounds annually when that weapon system goes into full-scale production, and the potential waste is estimated at 20% of the total [1]. This problem can be avoided by the replacement of thermoset-based carbon-fiber composite laminates with carbon-fiber-reinforced thermoplastic prepregs, due to their infinite shelf life. In addition, VOC emissions during thermoplastic processing are insignificant compared to thermosets [1].

However, a major barrier to the use of thermoplastics is their ability to meet both performance and throughput requirements (100+/day). Induction processing of carbon-fiber thermoplastics offers a potential solution to this problem by enabling rapid volumetric heating of the thermoplastic prior to consolidation as described in the previous report [1]. This technology will enable reduction in cycle times, while maintaining quality, compared to conventional compression molding processes.

The key thermal requirement of induction processing for lamination applications is rapid, uniform heating of the composite for maximum throughput and quality. This requires an understanding of the heating mechanisms during induction processing, followed by optimization of the critical process parameters. It is also essential to determine possible degradation mechanisms and process windows due to the rapid heating requirement.

2.2.1 Heating Mechanisms for Carbon/Thermoplastics

Induction heating for carbon-fiber-reinforced thermoplastic composites is based on the fact that eddy-current flows along conductive paths within the composite subjected to an alternating magnetic field generated by the induction coil (Figure 8). The frequency and the intensity of the magnetic field penetrating the composite as well as the loop area of the conductive path determine the electromotive force (emf) or induced voltages, which, in turn, govern the heating of the composite.

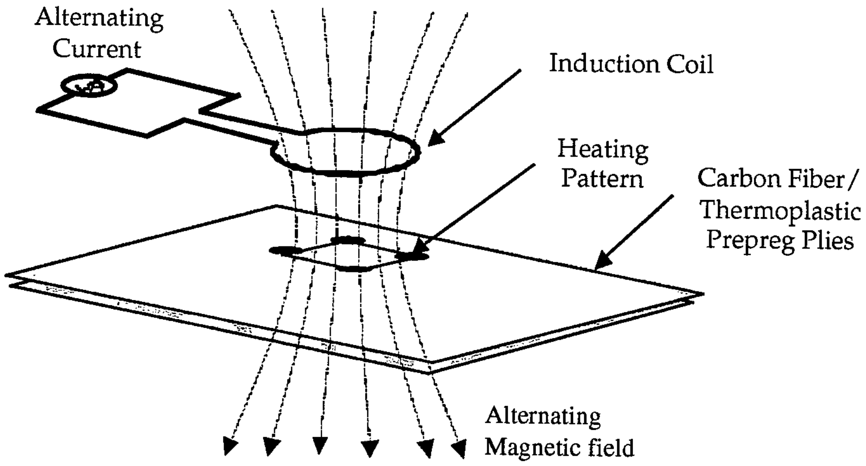


Figure 8. Schematic of the induction heating process for carbon/thermoplastic composites.

The primary objective of this study is to investigate the heating mechanisms of carbon/thermoplastics and then determine the relationship between the induction coil and material parameters and heating. Once this relationship is determined, it will be possible to perform parametric studies using the major process variables in order to optimize and meet the thermal requirements for the potential production of thermoplastic-based composite structures.

2.2.1.1 Theoretical Heating Model

Alternating magnetic field lines intersecting the laminate induce emf's within each conductive loop are governed by Faraday's Law of Induction [12]. Loops are formed between adjacent plies through the junctions, where fibers overlap each other. As a result, the induced current flows along the carbon fibers and either through the polymeric region or by direct contact of fibers, into its adjacent ply, as shown in Figure 9. Generally the emf induced in a circuit is directly proportional to the time rate of change of magnetic flux through the circuit and is calculated from

$$emf = \omega A B_0 = 2\pi f A B_0, \tag{1}$$

where B_0 is the maximum value of the magnetic field normal to the area of the conductive loop, A is the area of the conductive loop, and f is the time rate of change of magnetic flux.

Once the emf values for all conductive loops in the calculation domain are obtained, Kirchoff's voltage and current conservation laws are applied to the network of conductive loops. Kirchoff's voltage law (KVL) [12] requires that the algebraic sum of all voltages around the loop should be zero while Kirchoff's current law (KCL) means that current is conserved at each node. In mathematical terms, they can be expressed as follows:

$$\begin{aligned} &\sum \text{Voltage Drop} = \text{Induced emf} && \text{; Kirchoff's Voltage Law} \\ &\sum (\text{Incoming Current - Outgoing Current}) = 0 && \text{; Kirchoff's Current Law} \end{aligned} \tag{2}$$

Three heating mechanisms are possible within the composite:

- Joule heating due to the inherent electrical resistivity of the carbon fibers.
- Dielectric heating of the polymer at the fiber junctions.
- Contact resistance at the fiber junctions.

In general, prepregs have nonuniform surface roughnesses, which makes it difficult to determine which mechanism is dominant at a certain region in the interface between plies. In addition, it is not easy to estimate the electrical contact resistance between carbon fibers of adjacent plies. The heating mechanisms are shown schematically in Figure 10 and described in detail hereafter.

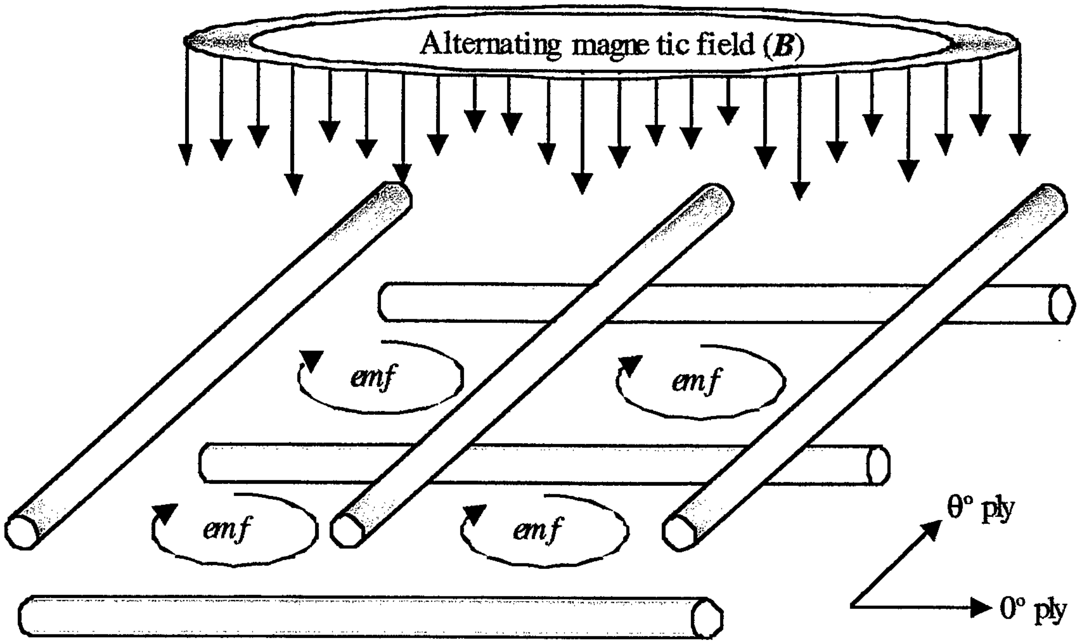


Figure 9. Schematic of induced voltage loops in the composite.

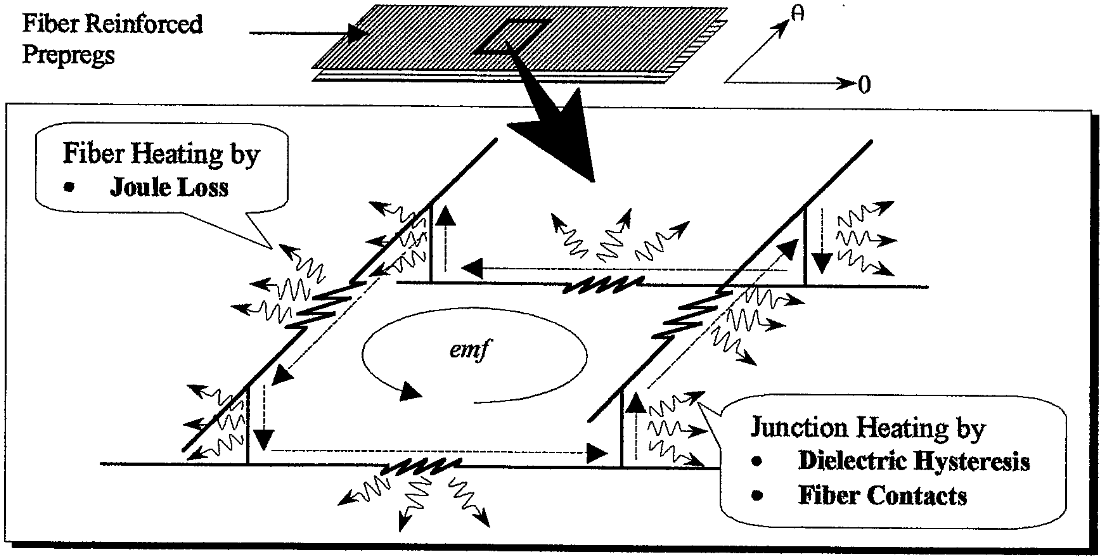


Figure 10. Heating mechanisms in each individual conductive loop.

2.2.1.2 Joule Heating of Carbon Fibers [15, 16]

Previous authors [13–16] have compared joule heating in the fiber and dielectric heating in the matrix and shown that dielectric heating is the dominant mechanism. The current effort includes the contact resistance mechanism and performs a parametric study of all three mechanisms for the process variables defined for thermoplastic laminates. Successful modeling will identify the key heating mechanism and optimize induction-based processing parameters to meet quality and performance requirements. This will enable transition from thermosets to thermoplastics, thus eliminating potentially large hazardous waste stream due to shelf-life expiration.

Each carbon fiber is treated as a resistor and the heat generated is calculated from

$$P_{fiber} = I^2_{fiber} R_{fiber}, \tag{3}$$

where I_{fiber} is the induced current flowing in the fiber and R_{fiber} is the resistance of the fiber, which can be expressed as

$$R_{fiber} = \rho_{fiber} \frac{l_x}{A_{fiber}} \text{ or } \rho_{fiber} \frac{l_y}{A_{fiber}}, \tag{4}$$

where A_{fiber} is the cross-sectional area of the fiber and l_x and l_y are the spacing distances between fiber intersections in the x and y directions, respectively. Note that l_x and l_y vary according to the mesh size as shown in Figure 10.

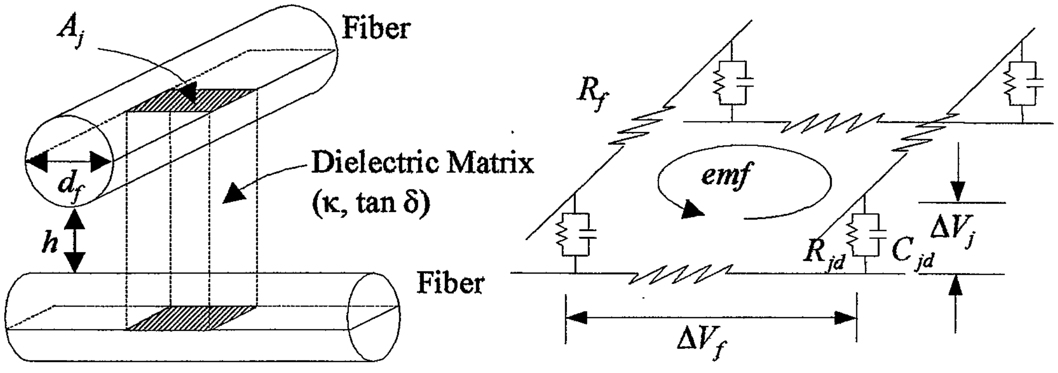
2.2.1.3 Dielectric Hysteresis Heating at Fiber Junctions [13, 14].

If the distance between the fibers at the junction is enough to form a capacitor, dielectric heating takes place, since the molecular dipoles in the matrix cannot rotate with the same frequency of the induced voltages in the fibers. The dissipation factor (tan δ), which is one of the electrical properties of the matrix, determines how much heat will be dissipated. The impedance of the capacitor is $1/(\omega C \tan \delta)$, where ω is the angular frequency of the alternating current and C is the capacitance of the material. Considering the configuration of the fiber junction shown in Figure 11, the capacitance of the dielectric material can be expressed as follows:

$$C = \kappa \varepsilon_0 \frac{A_P}{h} \tag{6}$$

where κ is the relative dielectric constant of the material and ε_0 is the permittivity of vacuum (8.85×10^{-12} f/m). A_p and h are the projection area and distance between the fibers at the junction, respectively. Therefore, the impedance of the capacitor (Z_C) can be written as

$$Z_C = \frac{h}{\omega \kappa \varepsilon_0 (\tan \delta) A_P}. \tag{7}$$



$$R_{jd} = \frac{h}{\omega \varepsilon_0 \kappa (\tan\delta) d_f^2}$$

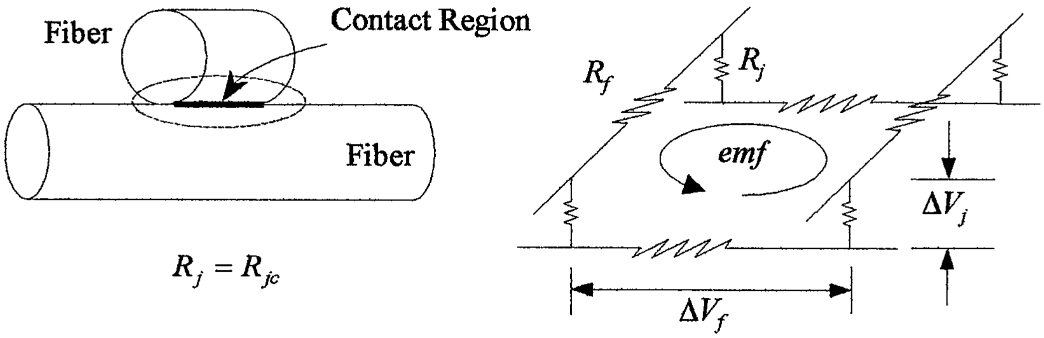
Figure 11. Circuit model for dielectric heating.

The heating generated by the capacitor is as follows:

$$P_{junction} = \frac{\left(V_{junction}\right)^2}{Z_C} = \frac{\omega \kappa \varepsilon_0 (\tan\delta) A_P \left(V_{junction}\right)^2}{h} \qquad (8)$$

2.2.1.4 Heat Generated by Fiber Contact Resistance

If the fibers at the junction are in contact or the distance between fibers is very short, heating can occur at the contact region due to contact resistance between the fibers. However, as mentioned previously, it is hard to quantify the contact resistance, as it is a function of surface roughness of prepreg and the laminate processing parameters. A simple resistor can model the fiber contact and the heating mechanism (Figure 12), and through parametric studies and experiments, the contact resistance is estimated.

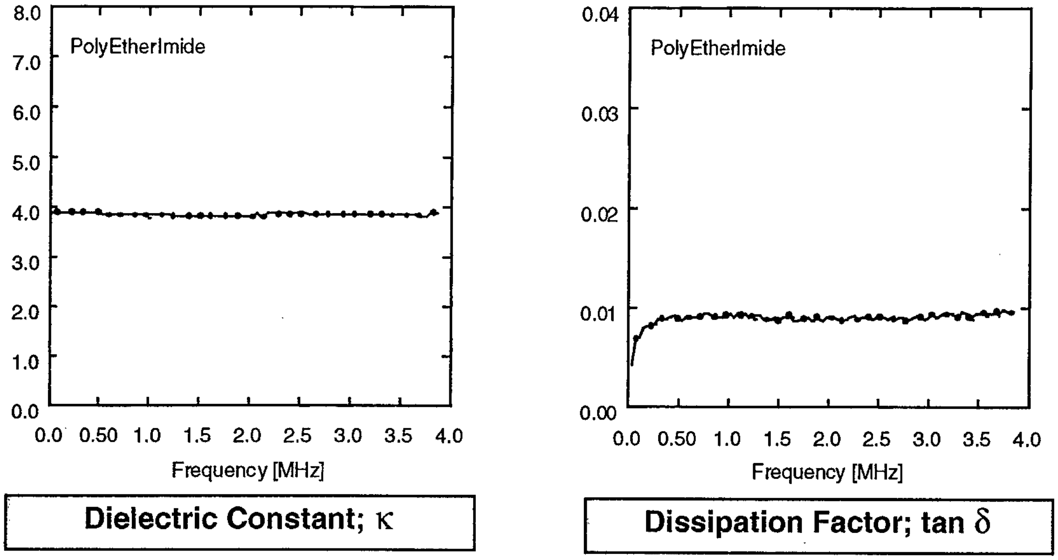


$$R_j = R_{jc}$$

Figure 12. Circuit model for heating by fiber contact resistance.

2.2.1.5 Material Properties

In this study, AS4 carbon fiber and polyetherimide (PEI) thermoplastic prepreg was selected as the test matrix because it serves as a model system for proposed military munitions manufacture and provides high-process temperature limits. The diameter of the carbon fiber was 8 μm, and the electrical resistivity was 15.3 μΩm. The dielectric constant (κ) and dissipation factor (tan δ) for the PEI were measured by experimental studies. The experimental results show that the dielectric constant and dissipation factor can be assumed to be constant for frequencies up to 4 MHz as shown in Figure 13.



Dielectric Constant; κ

Dissipation Factor; tan δ

Figure 13. Dielectric properties of PEI (ultem 1000, GE plastics).

2.2.1.6 Two-Ply Heating Model

Initial models focused on addressing the heating behavior of 2-ply prepreg stacks. This allows for a perform of a detailed parametric study, followed by experimental verification to assess the model's capabilities. A schematic describing the model procedure is shown in Figure 14.

Since carbon/thermoplastic prepregs, such as AS4/PEI or AS4/polyetheretherketone (PEEK), have approximately 20 fibers per 5-mil (127-μm) width, it can be estimated that about 790,000 fibers exist in a 1-m wide prepreg. Therefore, cross-ply or angle-ply prepregs are simplified by a conductive loop network using a finite number of fiber grids with an assumption that fibers and junctions within each conductive loop have the same resistance and current values. It is expected that this type of meshing will generate good qualitative results, and if the mesh becomes denser, more precise results, in the quantitative sense can be expected.

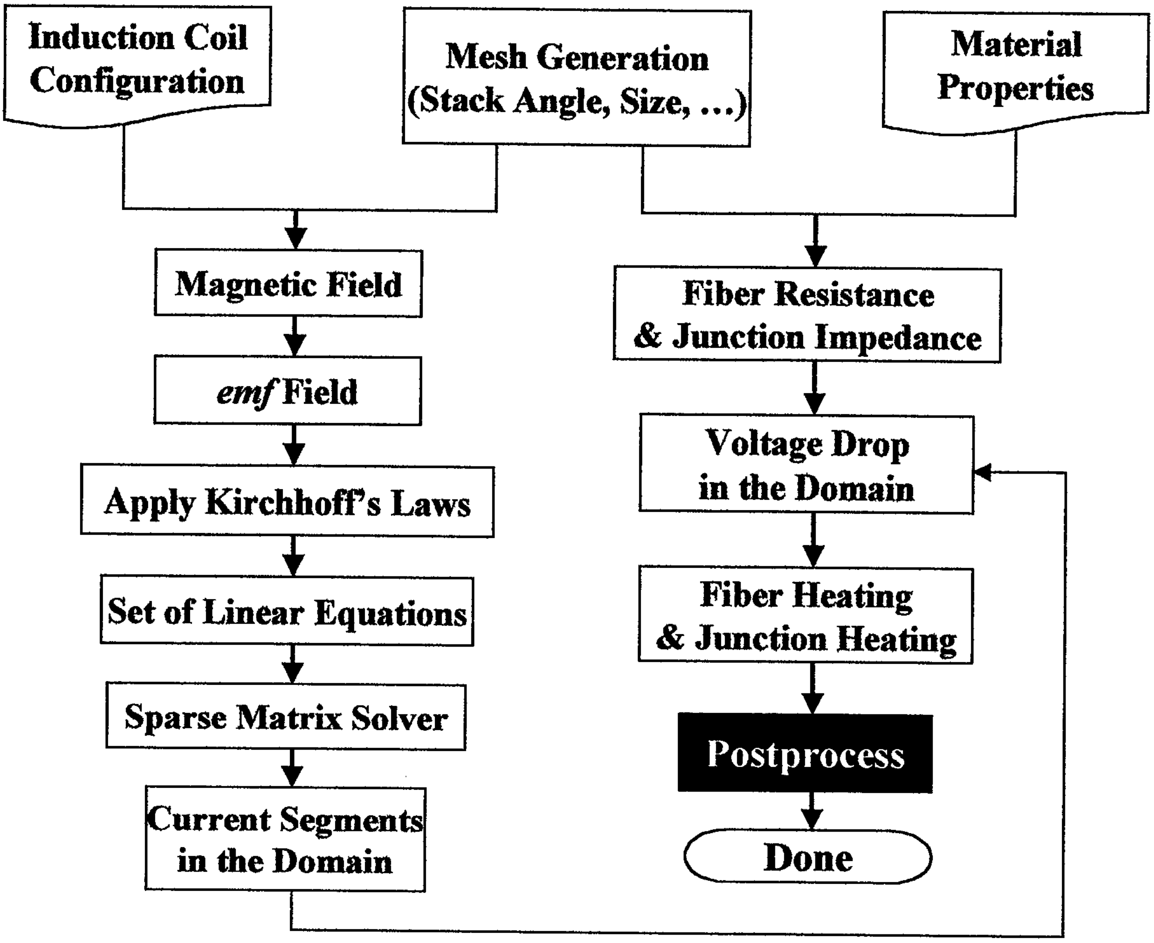


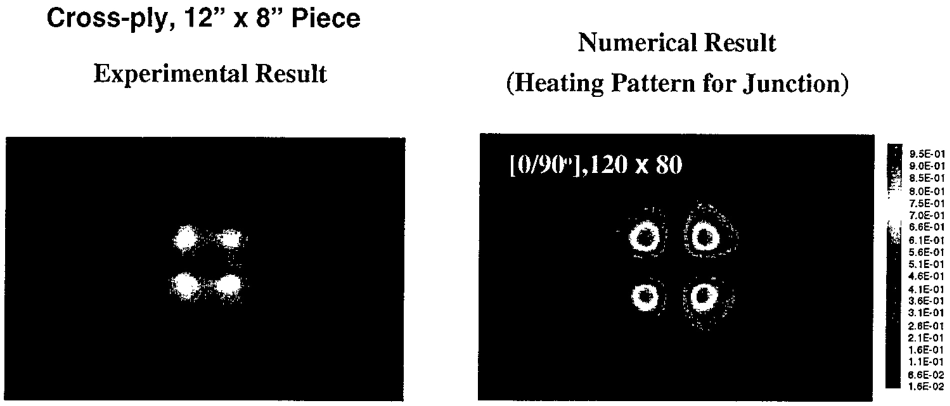
Figure 14. Schematic of a 2-ply heating model.

The process variables used in the model are: (1) coil type (pancake, conical, paper clip, and solenoid), (2) coil size (outer dimension, inner dimension, (3) number of turns, (4) spacing between turns), (5) distance between induction coil and composite, (6) frequency of the current in the induction coil, and (7) size and geometry of the composite.

Variables in the numerical model for parametric studies are mesh size and density, fiber-fiber distance at the interface of two plies, and fiber-fiber contact resistance or equivalent impedance for fiber junction.

2.2.1.7 Two-Ply Model Results

Initial experiments focused on evaluating numerical predictions qualitatively. This was done by heating 2-ply stacks at various angles—[0/90], [0/θ]—under a known magnetic field and comparing measured heating patterns, obtained using a calibrated thermal infrared camera, with the 2-ply model predictions. Results are shown for two select cases: a cross-ply stack [0/90] (Figure 15) and an angle-ply stack [0/45] (Figure 16). Qualitatively, there is excellent correlation between the predicted pattern and the actual measured heating profile.

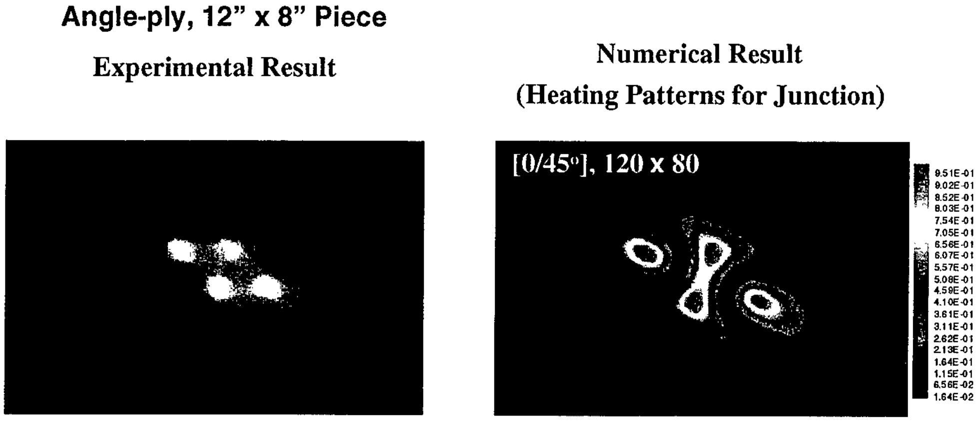


Cross-ply, 12" x 8" Piece

Experimental Result

Numerical Result
(Heating Pattern for Junction)

Figure 15. Comparison of heating patterns for [0/90] 2-ply stack.



Angle-ply, 12" x 8" Piece

Experimental Result

Numerical Result
(Heating Patterns for Junction)

Figure 16. Comparison of heating patterns for [0/45] 2-ply stack.

Predictions in these figures were obtained by parametric studies with the three heating mechanisms (fiber heating, dielectric, and contact resistance) in the 2-ply stack. These studies show that the junction-heating effects are greater than fiber heating by an order of magnitude. In other words, the primary heating in carbon/thermoplastics occurs at the junction and can be dielectric or contact resistance based, depending on the process variables.

Quantitative predictions are not as accurate and difficult to compare, because the model predicts heat generation rather than temperature. Work is continuing to optimize process variables and to extend the 2-ply model to multiply cases.

2.2.1.8 Through-Thickness Heating

The 2-ply model has shown that during induction processing, heating occurs predominantly at the junctions between fibers of adjacent plies or along the ply-ply interface. This raises the question of the uniformity of the temperature profile through the thickness of the composite. Figure 17 shows a typical heat-generation profile that can be expected for an 8-ply stack. As seen in the figure, heat generation occurs at the interface between any two plies that do not have the same fiber orientation.

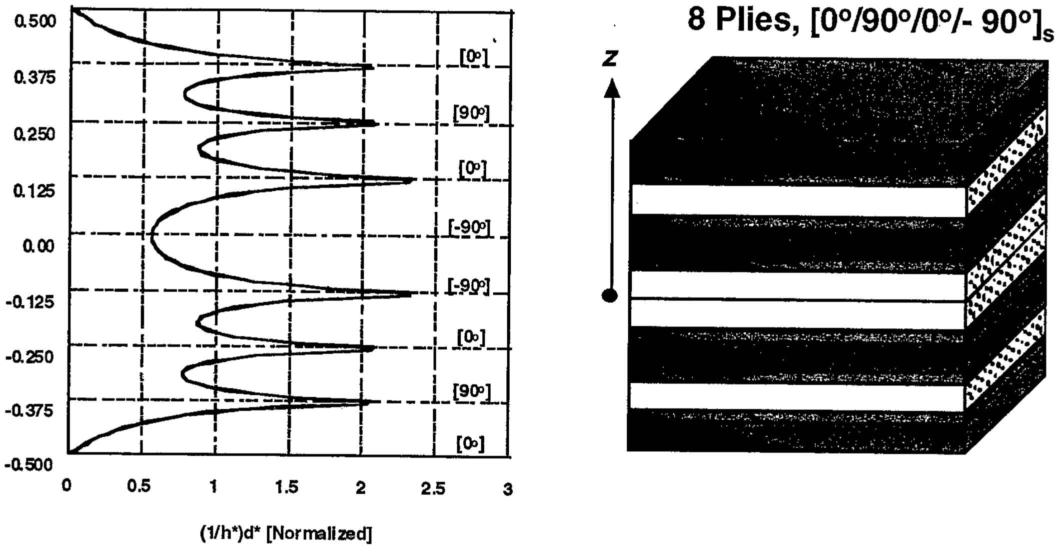
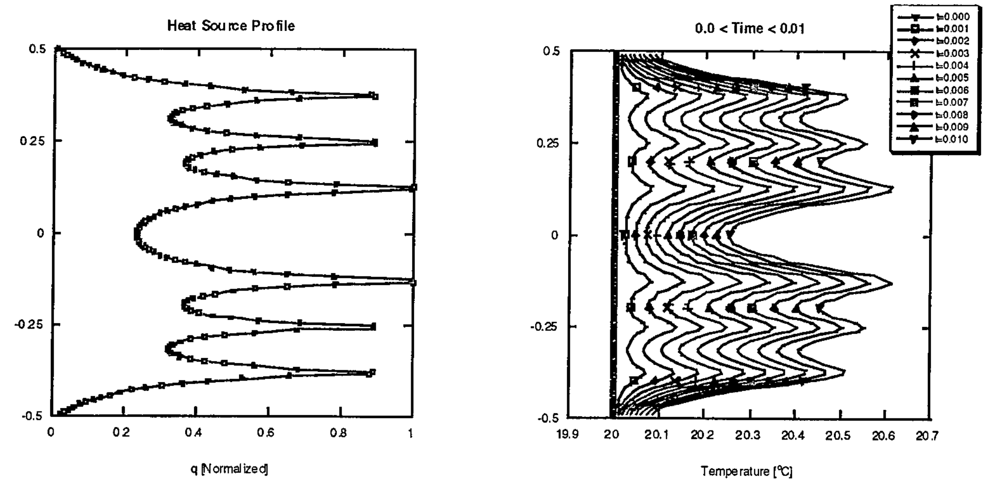


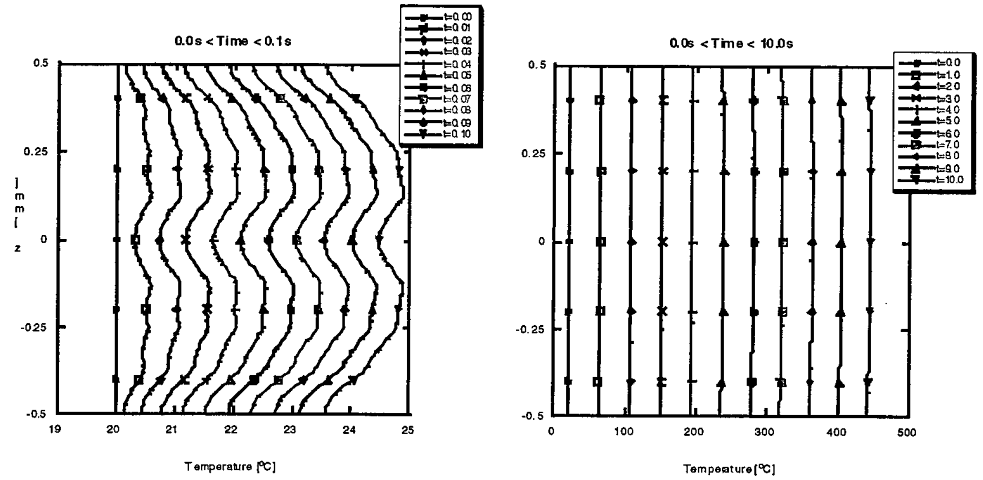
Figure 17. Model through-thickness heat generation of an 8-ply carbon-fiber laminate with orientation $[0/90/0/-90]_S$.

Since many structural laminates of interest to DOD are fabricated from 8-ply stacks similar to those shown in Figure 17 (or quasi-isotropic laminates built from such 8-ply stacks), it is necessary to identify not only surface heating profiles but also through-thickness heating profiles for quality and performance. It is essential that the temperature gradient across and through the thickness of the 8-ply stack is small during induction heating or significant performance degradation can result [17].

A one-dimensional (1-D) transient heat transfer model was formulated using a finite difference scheme to predict temperature profiles through the thickness, based on heat generation shown in Figure 17. Predicted temperature profiles are shown in Figure 18, showing transient, as well as steady-state, results. Results show that temperature variations across the thickness of the 8-ply stack are insignificant after 1 s, which is within the range of the process cycle time. This is expected due to the small thickness of each ply and has been borne out by experimental measurements.



Heat Source **0 s < t < 0.01 s**



0 s < t < 0.1 s **0 s < t < 10 s**

Figure 18. Predicted through-thickness temperature profiles at various time steps.

2.2.2 Degradation Studies

Electromagnetic induction processing of thermoplastic-based laminates is currently being evaluated and perfected to produce a 10-fold decrease in cycle times for production and a resultant decrease in production costs. This process requires that carbon-fiber-based composites be subjected to large alternating electromagnetic fields. In addition to the issues of degradation of the matrix in induction-based processing, in the EM gun program glass- and carbon-based polymer composite compulsator components experience very large fluctuations in electromagnetic energy during charging and discharging [18]. These systems are subject to potentially critical polymer degradation as a result of a little-studied phenomenon known as thermoelectric degradation [19]. When polymers degrade through any mechanism, they suffer significant losses in strength, stiffness, and durability.

The focus of this effort is on identifying degradation mechanisms during electromagnetic induction processing and quantifying their effects on performance. There are two possible degradation scenarios associated with induction-based processing of carbon/thermoplastics: thermal degradation, and electrical degradation due to dielectric breakdown in the matrix.

2.2.2.1 Thermal Degradation Study

Weight loss and molecular weight (MW) measurements were used to characterize thermal degradation of both neat resin and prepreg. Thermogravimetric analysis (TGA) was used for weight loss measurements in both air and inert (nitrogen) atmospheres. Gel permeation chromatography (GPC) was used to obtain MW measurements. In addition, dissolution times for resins in a good solvent (methylene chloride) were also measured.

2.2.2.2 Weight Loss Measurements

TGA for both neat PEI and AS4/PEI prepreg indicates no measurable weight loss of the bulk material up to 500 °C, as shown in Figure 19. Isothermal TGA data also shows that no weight loss was observed at 350 °C, for up to 1 hr. Approximately 2% weight loss was observed at 450 °C after 30 min, which indicates significant degradation. Since the normal processing temperature is 330 °C, thermal degradation of the bulk material is expected to be minimal as long as the electromagnetic induction processed material does not exceed the processing window.

Neat PEI samples were exposed to various thermal histories using a TGA chamber, and the glass transition temperature was measured using DMA. Changes in resin color and dissolving time in a good solvent (methylene chloride) were also noted. As shown in Table 3, no significant changes in glass transition temperature were observed. However, the color of the resin changed from yellow to black, and the dissolving time increased significantly when temperature and time increased. Oxygen in the atmosphere also affects the color change and dissolving time in the solvent. In several cases (G≈K), there was some gel left over in the solution, which obviously indicates that crosslinking reactions occurred in the polymer.

The TGA study shows that weight loss alone is not sufficient to identify the degree of degradation of the polymer. The initiation of crosslinking in the polymer is a better measure of the onset of degradation. Crosslinking on a composite surface hinders diffusion of polymer chains during processing of complex parts and may result in poor bonding and performance.

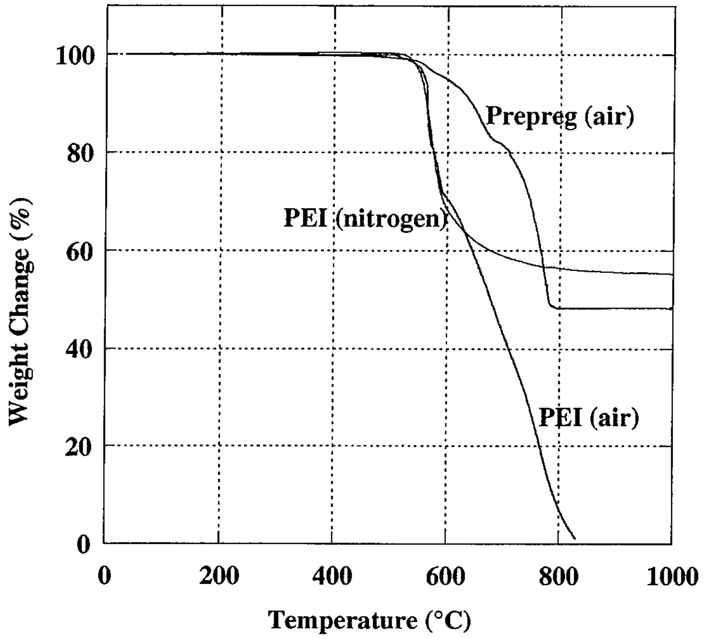


Figure 19. TGA weight loss profile for PEI resin and prepreg (heating rate = 15 °C/min).

2.2.2.3 Molecular Weight Characterization

GPC was used to assess the extent of crosslinking. According to the literature [20] and preliminary experimental observations, crosslinking reaction tends to be dominant over chain scission (breakup of chains) in the early stages of degradation of thermoplastic polymers (PEEK and PEI). Crosslinking reactions typically occur at lower temperatures (~350 °C) than the onset of weight loss observed by TGA (~450 °C). GPC is one of the most frequently used techniques to measure the polymer MW and its distribution (MWD). The MWD curves show the changes caused by crosslinking or chain scission. For example, if crosslinking is dominant over chain scission, there are higher MW chains; as a result, a broader curve will be obtained that has a peak intensity shifted to the left (shorter elution times) compared to a baseline polymer elution. If chain scission is dominant, the curve will be broadened to the right or longer elution times.

Most of the samples evaluated exhibited peak intensity shifts to shorter elution times, which indicates that crosslinking is dominant. All of the curves were recalculated after the intensity values were divided by the peak intensity. The normalized area under the curve can be used as a qualitative measure of the extent of crosslinking. The intensity normalized area increases with crosslinking.

Table 3. Experimental data of neat ultem 1000 resin under various heat treatments.

Sample	Condition of Heat Treatment	Atmosphere	T_g From DMA (°C)	Color Change	Dissolving Time in Solvent (Methylene Chloride)
A	No treatment		210	None	Less than 2 hr
B	330 °C, 30 min	Nitrogen	210	None	Less than 2 hr
C	330 °C, 30 min	Air	210	Mild	3 hr
D	350 °C, 30 min	Nitrogen	210	None	4hr
E	350 °C, 60 min	Nitrogen	210	Mild	5 hr
F	350 °C, 30 min	Air	210	Moderate	6 hr
G	350 °C, 60 min	Air	210	Moderate	Some left over as a gel
H	400 °C, 30 min	Nitrogen	210	Moderate	Some left over as a gel
I	400 °C, 60 min	Nitrogen	210	Moderate	Some left over as a gel
J	400 °C, 30 min	Air	210	Severe	Some left over as a gel
K	400 °C, 60 min	Air	210	Severe	Some left over as a gel

Results from GPC analysis are shown in Table 4. Neat resin and prepreg specimens were tested under various thermal histories in air, nitrogen, and vacuum atmospheres. All the GPC curve areas were intensity normalized with the neat resin case (sample A). In the neat resin study, no change is observed up to 350 °C for 30 min in nitrogen (A≈D). However, in the presence of air (atmospheric oxygen), significant increases in the GPC-curve areas are observed, indicating the presence of crosslinked polymer chains. Samples D and F show the effect of atmosphere, F and J show the effect of temperature, and J and K show the effect of time in a reactive atmosphere (air). The GPC area indicates the onset of crosslinking (and degradation) at lower temperatures than weight-loss tests and hence is a better tool to quantify degradation and establish process limits. For prepreg processed under vacuum conditions, some crosslinking occurs at 350 °C (1.13 compared to 1.08 baseline) while significant crosslinking degradation occurs at 400 °C. In comparison, specimens processed in air exhibit higher levels of degradation at both test temperatures, as expected.

Table 4. Degradation study of PEI and AS4/PEI prepreg using GPC.

Sample	Process	Temperature (°C)	Time (Min)	Atmosphere	Area Ratio Under the GPC Curve
A	Resin	No treatment	–	–	1.00
D	Resin	350	30	Nitrogen	1.00
F	Resin	350	30	Air	1.10
J	Resin	400	30	Air	1.13
K	Resin	400	60	Air	1.28
L	Prepreg	No treatment	–	–	1.07
M	Autoclave	330	20	Vacuum	1.08
N	Autoclave	350	60	Vacuum	1.13
O	Autoclave	400	60	Vacuum	1.60
P	Oven	330	20	Air	1.21
Q	Oven	350	30	Air	1.31
R	Oven	350	60	Air	1.39
S	Oven	400	30	Air	1.37
T	Oven	400	60	Air	1.48
U	Induction	309	1	Vacuum	1.07
V	Induction	319	1	Vacuum	1.07
W	Induction	330	1	Vacuum	1.08
X	Induction	387	1	Vacuum	1.05
Y	Induction	405	1	Vacuum	1.06

The induction-processed samples were subjected to high frequency magnetic fields for approximately 1 min, which was chosen based on design cycle times in electromagnetic-induction-based process for manufacture of laminates. Magnetic field parameters were selected to mimic process conditions in the manufacturing process. Preliminary tests performed under vacuum atmosphere indicate no measurable polymer degradations under these conditions, even though the composite laminate does reach the degradation temperatures of 380 °C and 400 °C (samples X and Y, respectively).

2.2.2.4 Electrical Degradation Study

Dielectric breakdown of polymers results in localized damage, which leads to deterioration of the mechanical properties of the composite [20]. Several mechanisms can occur and lead to breakdown, such as discharge breakdown and intrinsic breakdown. In this effort, the purpose is to identify electromagnetic parameters that produce breakdown; not to elucidate the mechanisms for this breakdown. Thus, it is show that dielectric breakdown is not likely to occur during electromagnetic processing of AS4/PEI.

Breakdown measurements were performed on neat PEI films of various thicknesses. The junction of fiber overlap is the region of expected breakdown and the thickness of PEI in these regions is small (submicron). Thin films, as small as 100 nm, were fabricated using a solvent-based spin-coating technique.

The dielectric breakdown voltages for neat PEI films of various thickness are presented in Figure 20. The breakdown voltage increases as the sample thickness increases. The voltage required for breakdown in a 100-nm film of PEI is ~350 V. The voltage drop in the induced current loops during electromagnetic processing is an order of magnitude smaller. For example, the induced voltage due to a three-turn coil with a 10-A current at a frequency of 10 MHz and affecting a 0.1-m square loop in the composite is only 40 V. It may be concluded that dielectric breakdown of the matrix is unlikely during electromagnetic processing of AS4/PEI composites.

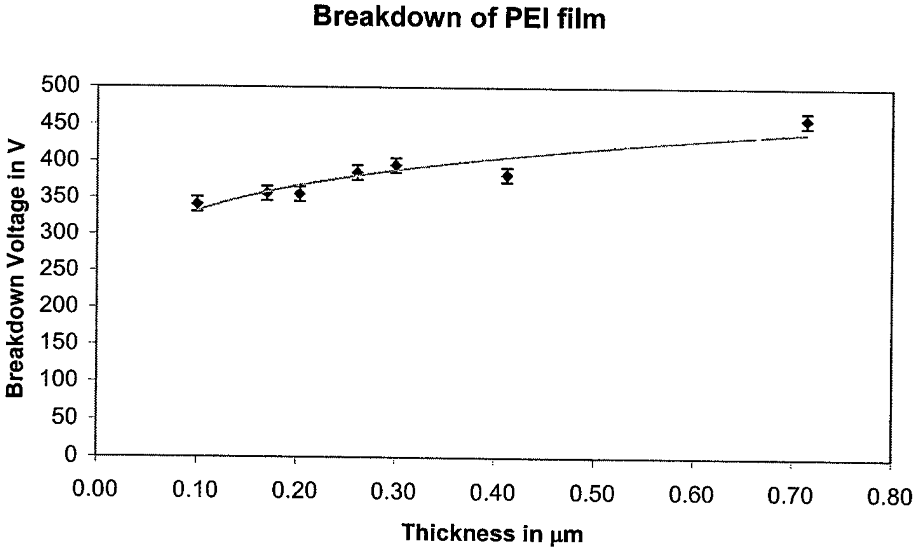


Figure 20. Breakdown voltage measurements for neat PEI films.

2.2.2.5 Mechanical Performance

Short beam shear (ASTM D 2344) [21] and compression (ASTM 695) [22] tests were performed with autoclaved and electromagnetic-induction-processed AS4/PEI specimens. These two properties are directly related to the matrix properties in the composite and are sensitive to matrix degradation. High pressure (75 psi) was used in order to eliminate the void content effect on properties. Measured void contents were less than 1% for both samples. The mechanical test results are shown in Table 5 and indicate no loss in performance due to electromagnetic induction-based processing of AS4/PEI.

Table 5. Comparison of mechanical properties.

Process	Apparent Shear Strength (psi)	Compressive Strength (ksi)
Electromagnetic-Induction Processing (A)[a]	1,1300 ± 500	120.1 ± 1.2
Autoclave (B)[b]	1,1500 ± 500	118.3 ± 1.8

[a]Process A: Autoclaved at vacuum; induction heated; autoclaved at 75 psi.
[b]Process B: Autoclaved at vacuum; autoclaved at 75 psi.

2.2.3 Induction Coil Design

A major advantage of induction heating technology is coil-design flexibility. The size and shape of an induction coil can be "fit," or matched, to the composite part that is to be heated, even for geometrically complex shapes. It is also possible to use a simple coil design and heat complex geometric shapes using programmed motion with a robot. Based on induction coil models, coil designs were developed for a 12-in wide laminate process. This involves lamination or consolidation of an 8-ply prepreg in the desired orientation into a consolidated laminate with specified quality. This is achieved by induction heating the prepreg stack up to process temperature, followed by consolidation under pressure.

2.2.3.1 Laminator Coil Design

The function of the laminator or the lamination stage is to fabricate 8-ply thermoplastic laminates at high throughputs (~20 ft/min) and desired quality. Thus, the induction heating stage of this process step has to uniformly and rapidly heat the incoming material (8-ply prepreg stack) up to the process temperature while allowing continuous material flow, as shown in Figure 21. The challenge is to handle incoming prepreg stacks of various orientations and still meet the rapid and uniform heating requirements.

Several different coil configurations were modeled and tested resulting in the selection of a rectangular (or paperclip shaped) coil for the laminator. The coil geometry and resultant temperature profiles are shown in Figures 22 and 23, respectively.

Work is in progress to optimize the rectangular coil geometry to further reduce temperature gradients and improve final laminate quality.

2.2.4 Conclusions to Induction-Based Thermoplastic Composite Lamination

Work to date has established that induction heating is a key technology component for the use of carbon/thermoplastics in Army composite structures.

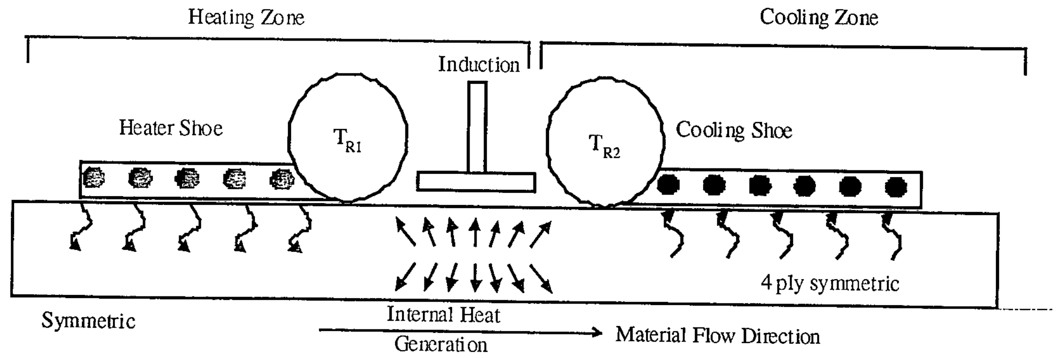


Figure 21. Process schematic of lamination stage in a production line.

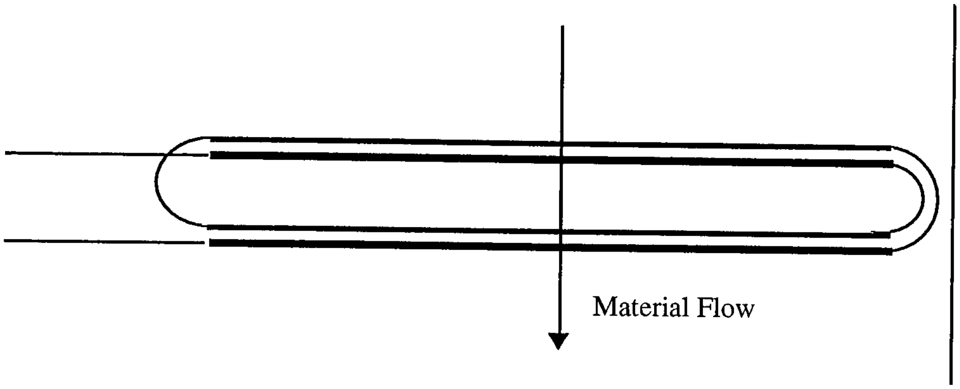


Figure 22. Schematic of rectangular coil for lamination stage.

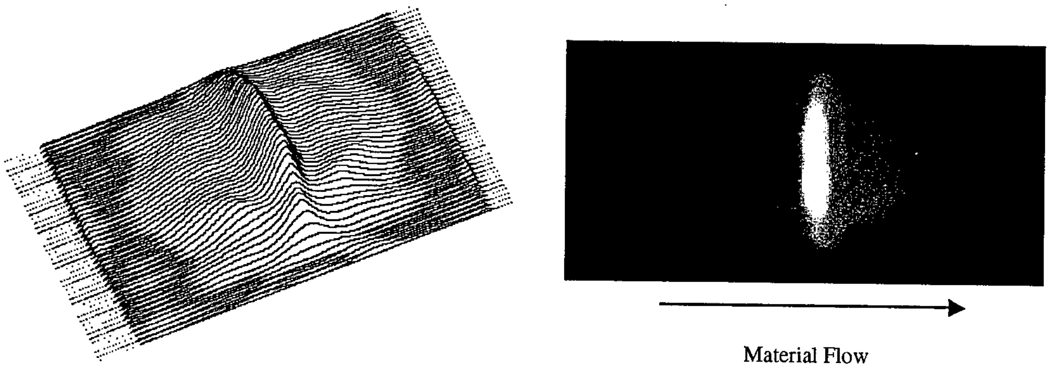


Figure 23. Temperature profiles of 8-ply stack with rectangular coil.

The ability to fabricate laminates from thermoplastic polymers while meeting performance and throughput requirements will allow future designs to replace environmentally hazardous thermoset-processing techniques with low-impact alternatives and potentially eliminate a substantial volume of hazardous waste production for the Army and its contractors.

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13. ABSTRACT*(Maximum 200 words)*

The development of induction-based processing of carbon-fiber (CF) thermoplastic-matrix composites and accelerated cure of thermosetting adhesives has the potential to provide nonautoclave processing technology for manufacturing and repair of polymer-matrix composites (PMCs). In this report, the results of recent tests demonstrating bonding of composites using commercial off-the-shelf thermal-cure adhesives that are heat cured via an induction field using an inductive susceptor are discussed. This method of cure utilizes heat generation within metal screen-based susceptors to cure the adhesive matrix via a heat transfer mechanism. The mechanical performance of these bonds is presented in comparison with autoclave and thermally cured baselines. No substantial loss of mechanical lap-shear strength is observed in adhesive bonds processed by induction. In addition, an example of induction welding of a thermoplastic-impregnated carbon fiber (AS4) is presented. In order to successfully demonstrate induction welding for manufacture of CF composites, the degradation of the polymer in the laminates is also investigated. No measurable degradation of the polymer, either by dielectric or thermal breakdown when heated by induction, was observed.

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