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ENABLING TECHNOLOGIES FOR
ECONOMICAL MANUFACTURING OF COMPOSITES

FINAL REPORT

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Dr. George S. Springer

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13. ABSTRACT (Maximum 200 words) Two separate contributions to the state-of-the-art in on-line (out-of-autoclave) consolidation of thermoplastic matrix composites have been developed under this program. First, an induction consolidation sensor has been developed which appears capable of detecting differences in consolidation state without physical contact. The consolidation defects detected are in the category of "kissing unbonds" (KUB's). Second, in collaboration with Lockheed and the Air Force Phillips Lab, in-situ consolidation of thermoplastic composites by means of direct electrical heating has been demonstrated at realistically high winding speeds. A version of the "top-of-component electrode" which is capable of off-axis laydown and fabrication of cross-ply tubular laminates has been constructed and tested. Reasonably good consolidation has been obtained thus far, though quality assessment is still in progress.				
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1. Consolidation monitoring and detection of "kissing unbonds" during in-situ fabrication of composite laminates

Prof. Alan K. Miller
Mr. Robin Van den Nieuwenhuisen

PROBLEM AND RELEVANCE

To enable the utilization of advanced composites in a wider range of applications, new methods for fabrication of composite components are being developed by industry. One of these, in-situ (on-line) consolidation of thermoplastic composites, is intended to be an economical out-of-autoclave fabrication method particularly well-suited to the production of large composite structures such as submarines, large space launch vehicles, spacecraft optical structures, and terrestrial vehicles and static structures. During on-line full consolidation, an important need is monitoring of the level of consolidation between the top ply and the underlying plies at the point of material laydown, for quality assessment and real-time process control. A particular challenge that existing methods generally do not meet is the detection of "kissing unbonds" (KUB's) in which the two adjacent plies are contiguous but not well bonded to each other. A second challenge is detection of defects without physical contact to the composite (which is hot during in-situ processing).

APPROACH

To try to meet this need, Prof. Alan K. Miller and Mr. Robin Van den Nieuwenhuisen have developed a new non-contacting technique for detecting ply-to-ply consolidation (or lack thereof) during fabrication of graphite-fiber polymer-matrix composites. In the new method, a specially-shaped induction coil is placed near the workpiece, a pulse of high-frequency power is put through the coil, and the temperature rise on the external surface at a pre-specified location with respect to the coil is measured by an infrared sensor. The magnitude of the temperature rise in a given time indicates the degree of consolidation.

PROGRESS TO DATE

To test the concept, three Gr/PEEK (polyetheretherketone) specimens containing controlled localized KUB's were fabricated (with the assistance of Lockheed Missiles and Space Company). The localized KUB's were introduced under the top ply by placing spots of "Frekote" mold release agent between a single prepreg ply and a pre-fabricated laminate, and then bonding them together in a hot press. The fiber orientation in the single prepreg ply was at an angle to that in the adjacent layer of the laminate, as would generally be the case in real structures. For the most recent tests, all specimens had the same thickness, indicating that any defects are truly KUB's and not open voids. These three specimens were interrogated with the new induction technique using the 450 kHz. induction power supply at Stanford, measuring the rate of temperature increase at a given level of induction power applied for a short time (a few seconds).

The three NDT data sets from the new technique fall in the same order of defect level as that expected from the specimen preparation procedures (Table 1) indicating that the new sensor is producing useful information.

To help interpret the data and identify the physical mechanisms governing the sensor's response, a physical model was developed. In this model, heat is generated by induction within both the underlying laminate and the top prepreg layer. The heat is conducted to the top of the specimen through the solid composite and the internal interface in question. The model predicts the temperature at the surface as a function of the magnitude of heat generated at the two locations and also of the thermal conductance at the interface. The induction coil is designed so that it generates heat as a "global electrical current loop" encompassing both the top ply and the underlying laminate. The

concept of the sensor is that if the top ply is in good electrical contact with the underlying layers, a strong current loop will be formed and heating will be relatively rapid. Poorer contact will produce a weaker current loop and less heating. Because the electrical conductivity at the interface is quite sensitive to the degree of fiber-fiber contact across the interface, the strength of the global current loop is expected to be a sensitive indicator of the degree of bonding at the interface. The degree of interfacial bonding also affects the thermal conductance at the interface, but probably in a less sensitive manner than the electrical conductance because thermal conduction can be bypassed by other heat transfer paths.

The model was used to ascertain whether the data was best explained by (1) variations in the strength of the global current loop (due to variations in electrical conductance at the interface) or (2) variations in the interface thermal conductivity. Variations in both of these parameters were made, and values of the parameters which best fit the three sets of data were selected by least-squares fitting. The three best-fit values thereby determined are characterized by a relatively large difference in induction heat generation among the three specimens, and a relatively small difference in thermal conductance (Table 1). Thus the modelling studies appear to confirm that the operating mechanism of the new sensor is variation in the electrical conductivity across the ply-to-ply interface. This is useful because, as explained above, this electrical conductivity is expected to be a sensitive measure of the degree of bonding, even for "kissing unbonds."

Lockheed personnel also conducted acoustic imaging examinations of the same specimens. The acoustic imaging results (Table 1) fall in the same order as both the induction consolidation monitor data and the expectations from the Frekote application, helping to confirm the capabilities of the induction consolidation technique for detection of defects in composites.

Specimen number	Amount of Frekote applied at interface	Specimen thickness	Heating rate with induction sensor	Electrical heat generation in top ply ¹	Thermal conductance at interface ¹	Defect level by acoustic imaging
II	least	0.043"	highest	highest	same	fewest
VIII	middle	0.043"	middle	middle	same	middle
IV	most	0.043"	lowest	lowest	same	most

TABLE 1 -- DATA ON PERFORMANCE OF INDUCTION CONSOLIDATION SENSOR IN DETECTING INTENTIONALLY-INTRODUCED "KISSING UNBONDS" IN GR/PEEK COMPOSITES. THE SENSOR'S OUTPUT CORRELATES WITH BOTH ACOUSTIC IMAGING DATA AND EXPECTATIONS OF DEFECT LEVELS FROM THE SPECIMEN PREPARATION, AND THE RESULTS ARE BEST EXPLAINED BY VARIATIONS IN THE ELECTRICAL CONDUCTIVITY AT THE INTERFACE.

SUMMARY OF MOST IMPORTANT RESULTS

1. The induction consolidation sensor appears capable of detecting differences in consolidation state without physical contact. This can be very useful for on-line process quality control.
2. The consolidation defects detected are in the category of "kissing unbonds" (KUB's) which are ordinarily very difficult to detect.
3. The physical mechanism governing the response appears to be the effect of consolidation state upon the inter-ply electrical contact resistance.
4. Quantitative differences in signal response are small, indicating that signal-to-noise ratio considerations could limit the applicability of the technique in actual composites processing.

¹ Determined by modelling studies

2. In-situ consolidation of thermoplastic composites by means of direct electrical heating

Prof. Alan K. Miller

Mr. Robin Van den Nieuwenhuisen

Work conducted in collaboration with Lockheed Missiles and Space Company and
US Air Force Phillips Laboratory at Edwards Air Force Base, CA

PROBLEM AND RELEVANCE

Autoclave processing of composites is an extremely costly, error prone, time consuming and laborious process which by its very nature inhibits the use of composite structures in many applications. In addition, the fabrication of very large composite structures will simply not be feasible if composite fabrication techniques are limited solely to autoclave processing. The specific goal of our work is the development of alternative, low-cost, out-of-autoclave composite fabrication techniques for thermoplastic matrix composites. Instead of depositing all of the material in one operation and then processing all of it in a subsequent step (autoclave processing) our approach employs in-situ processing, in which the prepreg material is gradually applied to a shaped mandrel and is locally melted and fully consolidated to the previously-applied layers as it is applied. At the end of the deposition, the part is complete. This approach is termed "in-situ fabrication". As the result of the incremental nature of material build-up during processing, this proposed fabrication technique will be as equally attractive to thin structures (spacecraft, aircraft) as it is to thick ones (submarines, ground vehicles, static structures).

APPROACH

The Stanford work supported under this ARO program is building upon work done at Lockheed Missiles and Space Company, Research and Development Division by Dr. Alan Miller and colleagues during 1990-91. With in-situ consolidation, a key consideration is the heating method used to locally melt and fuse the composite as it is applied. Other investigators have employed hot gas, infrared, hot shoe, laser, and microwave heating. No method has demonstrated the ability to lay down composite with full consolidation, over complex shapes and laydown angles, at economically high tape-laying speed, and with minimal peak pressure so as to avoid damage to any sensors which may be embedded to make the composite component into a "smart structure."

With these goals in mind, the Lockheed work has been employing direct electrical heating of the graphite fibers near the laydown point as the heating method. Various means have been tried for introducing the electrical currents into the fibers, including induction (non-contact) and resistance (direct contact to electrodes). In the early Lockheed work, the effectiveness of both induction and high frequency resistance heating was found to be too low for practical tape-laying applications. Success was subsequently achieved using direct resistance heating with ordinary 60-Hz AC, but employing a new conformable "top of component electrode" configuration instead of the "mandrel electrode" tried by other investigators. With this electrode configuration, the internal electrical heat generation combined with the continuous motion of the prepreg relative to the tape head results in the desired material processing zone which keeps the material above melt while under pressure under the "top of component electrode".

PROGRAMMATIC OBJECTIVES

The Lockheed work demonstrated consolidation down to a void content as low as 1.4%, far better than had ever been achieved with direct electrical heating of composites. However at the time, Lockheed R&D had only a simple ring winder (3" diameter) operating at about 10"/min. lay-down rate. Edwards AFB (Composites Lab) had available a 5-axis computer-controlled En-Tech winding machine and was glad to accommodate visiting scientists. Accordingly the decision was

taken to build upon the Lockheed technical approach and the Air Force equipment availability for composites processing research within this ARO-sponsored program. In particular, the objective selected for the ARO-sponsored work is to fabricate more complex tubular components containing various and crossed fiber orientations by the direct electrical heating approach, and to employ more realistic laydown rates than the Lockheed work. Such shapes, layups, and laydown rates are of direct relevance in a number of applications.

PROGRESS TO DATE

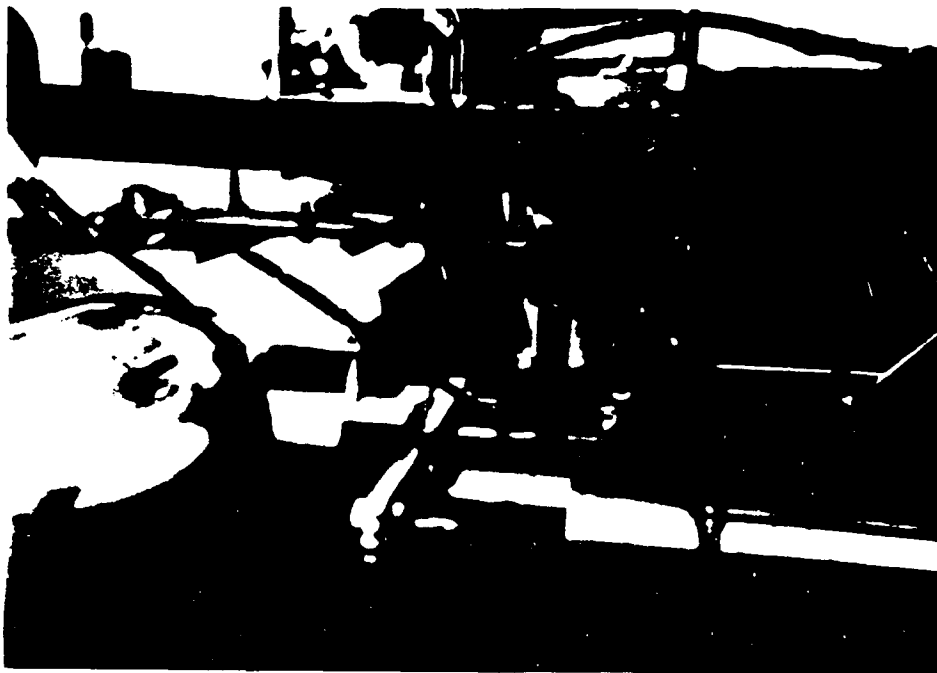
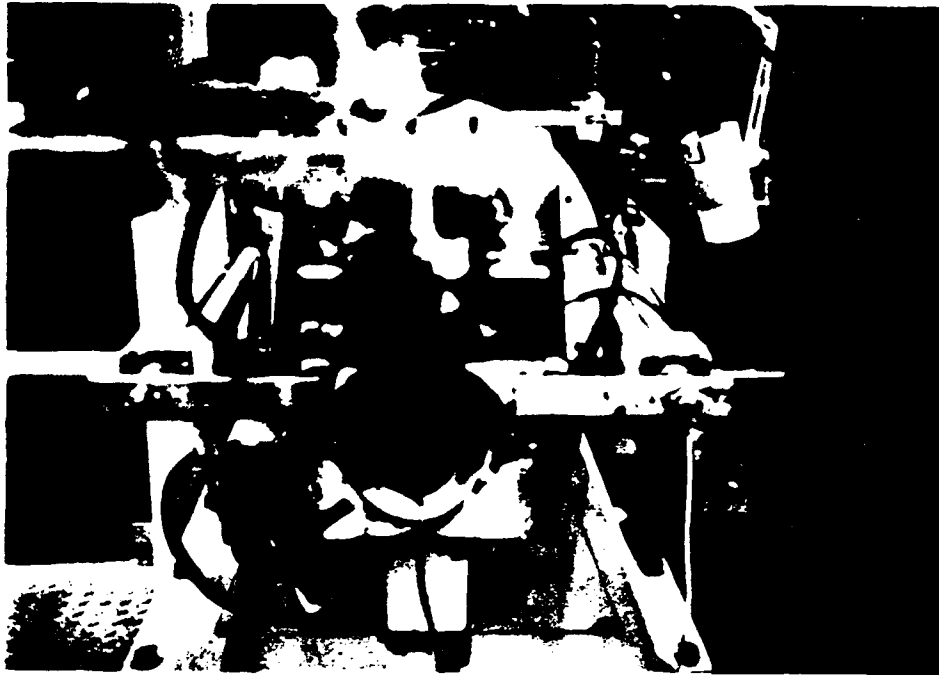
The research has been carried on at Edwards AFB by Stanford Ph.D. student Mr. Robin Van den Nieuwenhuisen, operating under the general guidance of Prof. Alan Miller and at Edwards AFB under the guidance of Mr. James Koury, and with financial support by this ARO program. Mr. Van den Nieuwenhuisen designed and had fabricated a top-of-component electrode which is capable of laying down composite tape at any laydown angle, on the existing 5-inch diameter mandrel of the En-Tech winder. Whereas the earlier Lockheed ring winder had a simple small pressure pad (under which the composite tape passes while it is hot, before it reaches the top-of-component electrode), the electrode in Mr. Van den Nieuwenhuisen's design is surrounded by an annular pressure pad, so that regardless of the direction from which the composite tape enters, it will receive pressure from the pressure pad before it passes under the electrode which withdraws the electrical current. (Current is introduced to the graphite fibers of the composite through a prepreg electrode.) Photos of the device are shown below.

During the research at Edwards, various challenges in apparatus refinement have been discovered, addressed, and met. A full test matrix of 32 trial runs (circumferential winding only) has been made, covering two values each of the electrical current density, prepreg electrode pressure, top-of-component electrode pressure, hot gas temperature, and laydown speed. The consolidated rings thus formed are being C-scanned and will be assessed for quality by densitometry and acoustic response. Based on the optimal conditions elucidated by the circumferential runs, cross-plyed tubular specimens will be fabricated, and the effects of additional process variables will be explored.

Since the funds and spending period of the ARO program expired in April 1992, the work is being carried on using Stanford University funds. Expected completion date is June 1993.

SUMMARY OF MOST IMPORTANT RESULTS

1. In-situ consolidation of thermoplastic composites by means of direct electrical heating has been demonstrated at realistically high winding speeds.
2. A version of the "top-of-component electrode" which is capable of off-axis laydown and fabrication of cross-plyed tubular laminates has been constructed and tested.
3. Reasonably good consolidation has been obtained thus far, though quality assessment is still in progress.



End view and side view of the "top-of-component electrode" mounted on the Air Force Phillips Lab's En-Tech 5-axis composites winding machine. The large outer annulus is the "pressure pad" while the inner region is the electrode. Current is introduced to the graphite fibers of the composite through a prepreg electrode (not visible). The device is shaped to permit laydown of composite material at any angle onto a cylindrical mandrel.

PARTICIPATING SCIENTIFIC PERSONNEL

Prof. Alan K. Miller

Mr. Robin Van den Nieuwenhuisen (received MS degree while employed on project)

Mr. James Koury (Air Force Phillips Laboratory, collaborating scientist)

1. Adaptive learning and the use of artificial intelligence for real-time progress control -- autoclave cure by an expert system.

George S. Springer
Peter Ciriscioli

The objective of this investigation was to develop an expert system which can be used both to select and control in real time the temperature and pressure inside the autoclave during the cure of parts made of thermosetting matrix composites. The advantages of such a system are

- there is no need to measure material properties prior to the processing,
- can be used for complex shaped parts,
- adjusts for variations in material properties,
- sets the "optimum" processing conditions in real time.

The system is based on rules. Thus, to perform the cure the following tasks were accomplished

- the rules were developed,
- a computer code was written for performing the necessary calculations in real time,
- sensors were developed,
- the entire system validated and tested.

Software and hardware were developed for controlling the autoclave. This system, designated as SECURE, was used to fabricate graphite epoxy laminates ranging in thickness from 0.1 to 6 inches. The data showed that the system controlled the temperatures extremely well and prevented significant temperature "overshoots" ("exotherms"). In addition, the properties of the material were found to be about the same as for laminates cured by conventional methods. Also, the cure time with SECURE was significantly lower than with conventional cure cycles.

P. Ciriscioli (Ph.D., 1990) and Q. Wang participated in this part of the project.

2. Effects of embedded sensors on composite strengths

G. S. Springer
S. Kim
M. Breslauer

In-plane compressive and three-point bend tests were performed to evaluate the strengths of composite laminates containing embedded sensors.

Composite plates were fabricated using unidirectional thermosetting or thermoplastic matrix prepreg tape. During layup a thermocouple or a simulated strain gauge was placed between two plies. After the layup was completed the thermosetting matrix plates were processed in an autoclave and the thermoplastic matrix plates in a press. After processing the plates were cut into 2.5 in x 1.0 in specimens for the in-plane compressive tests and into 4.5 in x 1.0 in specimens for the three-point bend tests.

The in-plane compressive test specimen was mounted in a specially built fixture in which the two short edges of the specimen were clamped. During the three-point bend test the plate was placed onto the three-point bend fixture. In each test the load was recorded at which the plate broke.

The data indicated that the embedded gauges had practically no effect on either the in-plane compressive or three-point bending strengths. This result was true for thermosetting as well as thermoplastic matrix composite specimens, regardless whether the specimens were made of unidirectional, cross-ply or quasi-isotropic laminates.

G.S. Springer, S. Kim and M. Breslauer participated in this project.

3. Sensor for detecting delamination damage

G.S. Springer
S. Kim
A. Segall

We developed a technique for detecting laminations in composite laminates.

The essential feature of this technique is the measurement of strain by sensors mounted on the surface as well as inside the composite. The undamaged structure is subjected to an in-plane compressive load, and the load versus strain is recorded for each sensor. Thereby a reference state is established under the proof load. To detect delamination introduced subsequently, the composite structure is again submitted to in-plane compressive loads and the outputs of the strain sensors are recorded. The strains thus obtained are compared with the reference values, and from these comparisons the magnitudes and the locations of the delaminations are deduced, as described below.

There can be many reasons for changes in the strain sensor outputs, including overall buckling, fiber breakage, matrix cracking and delamination. The present technique is addressed only to the detection of delamination, and identifies those strain responses which arise when delaminations open up under the applied in-plane compressive load. There may be delaminations present which do not open up under the applied load, and these are not indicated by the sensors. Unopened delaminations may be inconsequential within the applied load range. If it is deemed necessary, unopened delaminations may be examined by applying higher test loads.

Tests were also performed with graphite-epoxy plates containing either one, two or more delaminations. The data verified the technique, and demonstrated its usefulness in accessing delamination damage.

G.S. Springer, S. Kim and A. Segall participated in this phase of the program.