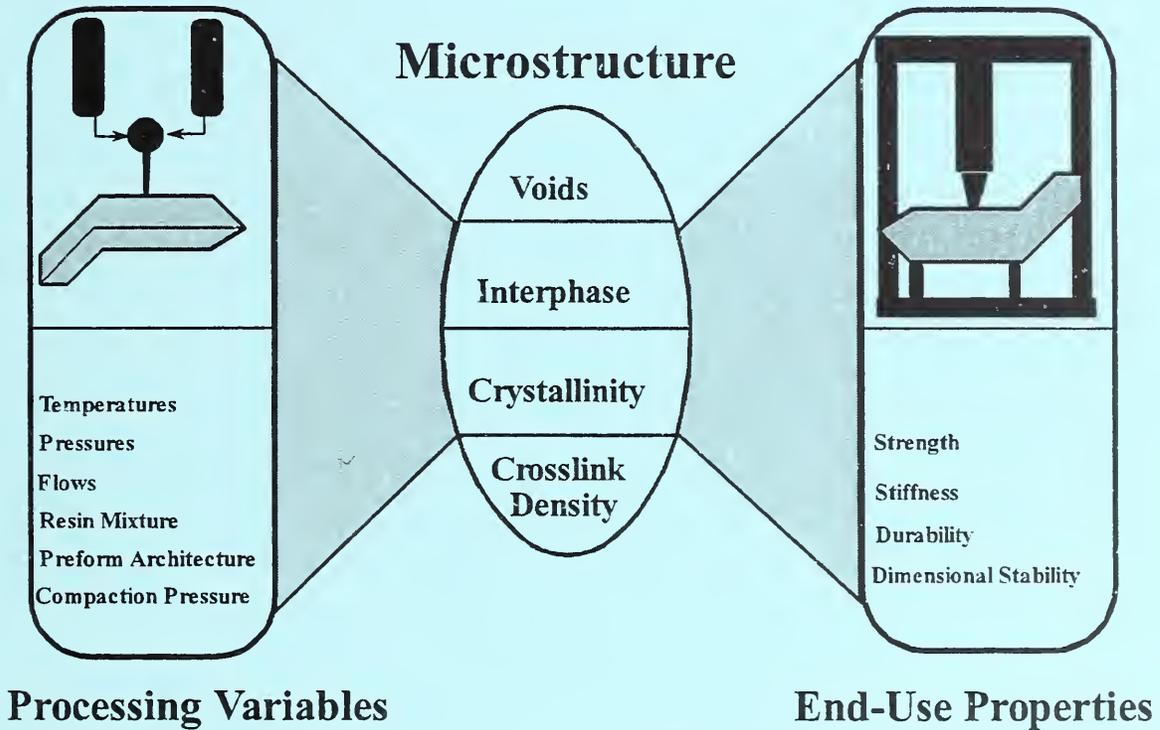




Report on the  
**Industry Workshop for  
On-line Composite Process Monitoring**  
April 3 & 4, 1996



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## EXECUTIVE SUMMARY

The Industry Workshop for Online Composite Process Monitoring was organized by NIST to stimulate inter-industry discussion and to guide the internal research program in the Polymers Division on sensors and control in composite fabrication. To facilitate the discussion a diverse group of 15 people, including 11 from the automotive, aerospace, electronics, and construction industries, were invited and attended the meeting. The meeting was purposefully restricted to a carefully chosen very small group because of the extremely diverse nature of the topic and the concurrent desire to keep the discussion focused tightly enough to provide maximum guidance to the internal research effort.

The most important conclusion of the workshop is that industry wants, foremost, technology capable of monitoring and controlling both the resin cure state and the residual porosity because these microstructural properties are difficult to control in practice, yet strongly affect a broad range of end-use-properties. The aerospace and automotive industries, however, emphasized resin cure and porosity differently due to their differing requirements and manufacturing processes. For example, the rapid cycle time and lower performance requirements of the automotive industry led to a higher emphasis on void content than on resin cure state. Concurrently, the automotive industry also requires more robust and rapid response sensors than the aerospace industry to make the required measurements.

Factors that were considered most important in selecting a sensor for manufacturing composites were accuracy and cost, with durability also considered important. A number of sensors were discussed in the workshop, with conventional pressure transducers and temperature sensors ranked highest (tied), and the relatively new fiber optic sensors ranked second. The commercially available ultrasonic and dielectric sensors were ranked substantially lower. Although many interpretations of this result are possible, it may be reasonable to conclude that industry demand exists for less expensive, more versatile sensor technologies. During the workshop discussions, the potential for fiber optic sensors to provide fundamental measurements in an unintrusive manner clearly excited the participants with the possibility that such sensors could meet the accuracy, cost, and durability goals simultaneously.

Once the broad topics were introduced and the preliminary discussions occurred, important follow up discussions focused on the expectations of the industry participants for sensor and control technology. Briefly, the responses were almost evenly divided between the necessity to reduce part defects and to optimize processing, i.e. minimize cycle time. These two responses are indicative of the differing business environments of the performance driven aerospace industry and rate driven automotive industry. Thus, the “best” sensor and control structures may be substantially different in different industries that use and manufacture composite parts.



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## **OBJECTIVES**

The Polymer Composite Group at NIST organized this focused workshop, held on April 3-4, 1996, to bring together a small group of experts and interested researchers to discuss the role of sensors and process control in composite fabrication. The workshop addressed the applicability of recently developed sensors and process control methods in fabricating high quality composite parts. In addition, the meeting discussed the role of classic non-destructive evaluation (NDE) techniques in meeting current processing needs. Although the technology in the areas of sensors and process control has been advancing rapidly, this has often occurred without a clear picture of the opportunities and needs in industries utilizing composites in structural applications. To discuss these issues the workshop considered the automotive and aerospace industries as contrasting examples. Automotive applications typically require high-speed, low-cost processing with the production of preforms in rapid cycle time. In contrast, the overriding concerns in aerospace applications are often high performance structural requirements. Therefore, stringent control of microstructure is critical. The workshop used these industries as a baseline to discuss the broad spectrum of composite applications.

The objectives in the meeting were to determine the role of sensors and process control in improved fabrication of structural composite parts and to identify the technical hurdles restricting the widespread use of these technologies. To accomplish this, the workshop sought to determine the essential end-use properties that are not effectively controlled with existing technologies, relate these end-use properties to critical microstructural properties, develop a clear understanding of the industrial environmental conditions in which in-line sensors must function, and determine the effect manufacturing speed has on sensor, NDE and process control technologies. The results of this workshop will also guide the development of a NIST research program in this effort. In addition, it is hoped that participants would gain insight into this area for their own companies and also explore collaboration opportunities with the NIST research effort.

## **BACKGROUND**

In 1988 and 1990, NIST organized two industry workshops [1,2] to identify the most important technical and scientific barriers to the implementation of composite materials in commercial applications. The participants concluded that cost-effective processing was the most important problem. Liquid Composite Molding (LCM) was identified as the processing method with the most potential to address the fabrication issue for structural parts. The second most cited issue was uncertainty about long term performance (durability). In response to these challenges, NIST organized a major in-house research program. The initial focus was processing science for LCM, but an effort on durability was subsequently added. To update and redefine critical issues relating to specific aspects of processing science, NIST has subsequently organized or co-sponsored three additional workshops. These meetings included not only industry researchers but also government and academic scientists. The

workshops [3] have discussed reinforcement/resin interactions during flow, preform architecture and permeability, and heating and rheokinetic effects during flow. In addition to flow related issues, high speed processing and the production of high quality preforms in rapid cycle times emerged as critical issues in the automotive industry.

An important thrust in NIST's in-house program is the development of flow models. In recent years, the NIST modeling program has developed a simple model, for uni-directional composites made by LCM, that describes the generation of voids and porosity during mold filling. More sophisticated models, employing the Lattice Boltzmann method,[4] are currently being formulated. During the last 3 years, the composite processing program has focused on linking composite processing with the mechanics and durability of the final composite part. The key to this link is a knowledge of the microstructure that is generated during processing. The relevant microstructural features are believed to be, but not limited to, voids, residual porosity, the resin/fiber interface, the state of the resin chemistry, and the degree of crystallinity in those resin systems that crystallize. The previously mentioned durability program, using primarily the single fiber fragmentation test and the microdrop test, was implemented to address issues relating to the resin fiber interface. This program focuses on adhesion at the resin/fiber interface, the durability of the interface to water and common automotive solvents, and the relationship of single fiber fragmentation results to full composite durability research.

Another important research area in the NIST program is process monitoring sensors. The NIST focus is on the development of a versatile optical fiber sensor for measuring resin cure, and other microstructural properties. This research program is an important key to developing the link between composite processing and the mechanics and durability of the final composite part. The sensor aspect of the NIST composites program has now developed to the point where it is appropriate to seek input and direction from industry. This is a major goal in this workshop.

## INFORMATION SOUGHT

To achieve the objectives on page 1, the workshop format was formulated to address the following questions:

- (1) Which end-use properties are both desirable and difficult to control?
- (2) Which microstructural properties are critical to end-use performance?
- (3) What are the industrial environments in-line sensors must tolerate?
- (4) What are the effects of manufacturing speed on the requirements for sensors, NDE, and controls (i.e. what is the role of this technology in industries like automotive where speed is critical)?

These questions evolved from informal industry contacts who suggested that there was a critical need for fast methods to assess voids, porosity and interface properties. It was also proposed by these informal contacts that the measurement methods for on-line monitoring, control, and optimization of composite fabrication by rapid processes, *e.g.* LCM, must be rugged and fast. Since fast and rugged methods often compromise the quality of the measurement, it was noted that the development of slower and more accurate techniques was also desirable for calibration of the rapid methods and for use with slower fabrication processes. Hence, the information sought in this workshop was structured to prioritize the important microstructure features, determine the environmental factors that may inhibit the successful implementation of sensors in rapid processes, and understand the impact of processing speed on sensor requirements.

## COMPOSITION OF THE WORKSHOP

A small group of experts from industry and academia were assembled to address the above questions. To insure significant input from industry the selection process was targeted toward industrial representatives who were experts in the area of rapid composite processing, microstructural defects, and the implementation of sensors in the industrial environment. Hence, 11 industry representatives were chosen from automotive, aerospace, electronics, controls, and equipment manufacturing. To augment this group 4 representatives from academia and research organizations were chosen based on their accomplishments in pertinent areas. A full list of attendees is given on pages 7-8.

Several NIST personnel were selected to attend the workshop based on their affiliation with the topics to be discussed. In addition, these personnel were selected to insure the accurate recording of issues and solutions arising from from the workshop during the presentations and discussion sections.

## WORKSHOP PROGRAM

The agenda of this workshop is given on page 9, and began with an introduction by Dr. Donald L. Hunston. The introduction was followed by presentations designed to evoke participation in the discussion sessions which followed each topic area. The topic areas were designed to answer one or more of the questions targeted in the information sought section of this report. The presentation and discussion synopses, which follows the summary, reflect the important aspects discussed in each topic area. Where appropriate, information has been added by the authors of this report to enhance or clarify understanding of some of the main issues. The slides used in each presentation are included in the appendix (page 25).

In addition to the presentations and discussion section, a questionnaire, designed to determine the views of the participants, was distributed. The results of the questionnaire are on pages 17 -19.

## SUMMARY

The Workshop first recognized that sensor and process control needs vary not only from one industry to another but even for different parts within the same application. Nevertheless, the attendees felt that they could identify general trends that run through the majority of applications within a given industry. In the area of high performance structural applications that typically occur in aerospace, the workshop concluded that the cure state of the resin and void/porosity content are often the most important microstructural features. The discussion mentioned delamination resistance and dimensional tolerance as important properties affected by these features. It was stated that void/porosity formation is difficult to control. Factors which have been shown to affect void/porosity formation are (1) presence of moisture-humidity, (2) solvent content, (3) air entrapment, (4) incomplete fiber wetting, and (5) poor tool design. In practice, void/porosity formation is minimized during processing by controlling humidity, pre-preg tack, the pre-preg film process, and autoclave pressure. Porosity has been shown to affect interlaminar shear strength and compression strength of composites (see Figures 19-21, on pages 46-48). In addition, voids/porosity affect micro buckling and longitudinal compression, since these properties are dependent on the stiffness of the matrix (see pages 50-51).

In manufacturing processes employing rapid composite processing techniques, *e.g.*, automotive, voids were considered the single most important microstructural feature that must be controlled. Although, the manufacturing of structural parts in the automotive and aerospace industries is driven by strength or stiffness factors, aerospace generally gives a higher priority to weight savings. This additional requirement necessitates the optimization of a structural composite to a higher level than is required in the automotive industry. Therefore the resin cure state in automotive composite manufacturing processes is not as critical as in aerospace applications. In the automotive industry cost per composite part is a primary driving factor in determining the feasibility of utilizing a composite part. Hence, rapid cycle times are critical to the manufacture of cost effective composites. As pointed out in the presentation on "Critical Composite Properties", the required liquid composite molding cycle time is in the range of 1-3 minutes.

With respect to microstructural properties and related features, the workshop rated residual cure stresses, rate of cure, and moisture content equivalent in importance and ranked third behind void/porosity content and degree of cure. In addition to the above features, the dielectric properties of the resin, the presence of foreign materials, consolidation, fiber wetting, and percent reinforcement and orientation were also mentioned as important composite properties.

There was no clear preference with respect to off-line characterization techniques. Of the seven techniques ranked by the participants, the ASTM void content procedure, spectroscopic characterization techniques of raw materials and ultrasonic techniques were slightly favored over the characterization of raw material processing characteristics by rheological measurements, x-ray, and thermomechanical techniques. With respect to on-line

sensors, temperature and fiber optic, dielectric, and ultrasonic sensors were all considered useful.

Ultrasonic sensors fall in the broad class of techniques used for non-destructive evaluation (NDE), i.e. post processing and in-service inspection for flaws and defects. Other methods included in this class are acoustic emission and various thermal, optical, and X-ray measurements. NDE technology has been developed over many years and can contribute to the process monitoring field in two ways. First, NDE experience can provide insight into important microstructural features. Process monitoring often seeks to detect the same features (i.e. flaws, defects, etc.) that are the focus in NDE. The objective in process control, however, is to identify these features as they are formed during fabrication so the processing cycle can be adjusted to minimize or eliminate them. The second area for input from NDE is the measurement techniques themselves. Since NDE methods often measure the same features of interest in process control, there is the potential to adapt NDE sensors for use as process monitoring tools. For example, the presentation entitled “A Manufacturing View of Adaptive Process Control” reported the implementation of an ultrasonic sensor in a commercial fabrication facility. An adaptive process control system was used with the sensor, and the resulting system significantly reduced the costs of producing compression molded composite parts.

In the presentation on the “Impact of Preforms on Sensors” and subsequent discussions, it was concluded that sensors which could determine the type of fibers, the fiber orientation, and the fiber volume fraction at specific locations within a part would be extremely useful for the production of preforms and composite parts. This is true across a wide range of industries. In aerospace, the high performance requirements can only be achieved if the fiber content and orientation is tightly controlled. In automotive applications, on the other hand, the allowable variations are significantly greater, but these limits must be achieved at very high fabrication rates. For example, the present goal in the manufacture of complex automotive parts like a structural cross-member is to reduce the fabrication time from 6 hours to 6 minutes.

The factors considered most important in selecting a sensor were accuracy, precision and cost. Second tier factors were response time and sensitivity. Several participants indicated that durability was also an important factor in the selection of a sensor. Of those participants who ranked this factor, it ranked number one ahead of accuracy & precision and cost. Most participants viewed the role of sensors in a manufacturing process as being two fold. The responses were equally divided between reducing defects and optimizing the process. Consistent with this result, it was stated in the presentation on “High Speed Processing of Composites” that the role of sensors in the manufacturing regime is to (1) improve process performance, (2) expand automation, (3) assure component quality, (4) minimize value added scrap, (5) reduce costs, and (6) provide documentation. It was also noted that for sensors to be successfully implemented in the manufacturing process they must (1) survive the manufacturing environment, (2) have response times of < 1 minute, (3) require low maintenance, and (4) be simple to operate.

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**Agenda  
for  
Industry Workshop for On-line Composite Process Monitoring**

April 3, 1996

8:00am - 8:30am	Registration
8:30am - 8:45am	Introduction Don Hunston NIST, Polymer Composites Group
8:45am - 9:00am	Survey
9:00am - 9:30am	Topic: Critical Composite Properties Ken Kendall Ford Motor Company
9:30am - 10:00am	Topic: Microstructure Thomas M. Donnellan Northrop-Grumman Corporate Research
10:00am - 10:30am	Coffee Break
10:30am - 12:00noon	Discussion - John Maguire Southwest Research Institute
12:00noon - 1:00pm	Lunch
1:00pm - 1:30pm	Topic: The Industrial Manufacturing Environment Foster Lamm United Technologies Research Center
1:30pm - 2:30pm	Discussion - John Fildes Basic Industry Research Laboratory
2:30pm - 3:00pm	Coffee Break
3:00pm - 3:30pm	Topic: Role of NDE Boro Djordjevic The Johns Hopkins University
3:30pm - 4:30pm	Discussion - Robert E. Green The Johns Hopkins University
4:30pm - 5:30pm	NIST Lab Tour
6:30pm -	Dinner Banquet, Courtyard Marriott

April 4, 1996

8:00am - 9:00am	Poster Session
9:00am - 9:30am	Topic: High Speed Processing Bruce Greve Budd Company
9:30am - 10:00am	Topic: Impact of Preforms on Sensors Daniel T. Buckley American GFM Corporation
10:00am - 10:30am	Coffee Break
10:30am - 11:30am	Discussion - Richard Parnas, NIST
11:30am - 12:00noon	Conclusion
12:00noon - 1:00pm	Lunch

## SYNOPSIS OF TOPIC PRESENTATIONS AND DISCUSSIONS

This section briefly summarizes what was said during the meeting. The agenda was organized around six topics that relate to the four questions mentioned earlier. For each topic, one or more presentations were given to stimulate thinking and then the meeting was open for discussion with everyone given the opportunity to contribute. The first topic, Critical Composite Properties and Microstructure, came from questions one and two while the second topic, The Manufacturing Environment, relates to question three. The topic, High Speed Processing of Composites, was taken from the fourth question. The final two topics, Role of NDE, and Impact of Preforms on Sensors, are related to question four but are broader than just high speed processing. The meeting concluded with a general discussion and a summary of the results from the questionnaire given to each participant. A highlight of the meeting was the description of a real success relevant to the topic of this meeting. It was included in the presentation by Foster Lamm. He described the development of a sensor based process control system and the transfer of this system to the manufacturing environment.

### *Critical Composite Properties & Microstructure*

A particularly crucial issue for the workshop was the identification of properties most important for process control and the microstructural features that control these properties. This topic which relates to the first two questions in the list was discussed first.

- (1) Which end-use properties are both desirable and difficult to control?
- (2) Which microstructural properties are critical to end-use performance?

Presentations were offered by Ken Kendall of Ford (see Figures 1-11 on pages 26-37) and Tom Donnellan of Northrop-Grumman (see Figures 12-53 on pages 38-80) while John Maguire of Southwest Research Institute moderated the discussion. Because the presenters represented automotive and aerospace companies, the discussion was able to contrast the needs and priorities for the two industries. This is useful since these requirements generally bracket those for most other commercial applications of composites.

### *Automotive*

With respect to composite manufacturing in the automotive industry, the importance of sensors is to help reduce cost. This is accomplished currently by reducing cycle time. In automotive applications, the materials requirements are stiffness driven. For structural parts produced by rapid processing, voids and the resin cure state are the primary microstructural properties that must be controlled to meet the stiffness requirements. Voids can have a detrimental effect on the stiffness and durability of a composite part. Existing technology indicates that void formation can be minimized by application of pressure at the appropriate time during the curing cycle. As a result, for complex parts where the pressure and

temperature may change dramatically throughout the part and during curing, continuous monitoring of these variables would be very useful. In addition to thermocouples and pressure transducers, dielectric sensors have been used in composite processing to monitor the cure process. Because of the rapid cycle times, 1-3 minutes, in Liquid Composite Molding (LCM), process control algorithms adjust process variables on subsequent parts as opposed to making adjustments on the current curing part. Therefore, raw material stability and control of the processing environment are important. In high volume processes, such as LCM, there is a push to ensure product quality by guaranteeing the process. Advocates of this viewpoint suggest that a cure standard, hence the need for appropriate sensors, and not a time standard be adopted. Current methodologies, attempt to minimize the impact of cure on the final composite part by post curing out of the mold and altering the cure kinetics when possible.

The variation of temperature and pressure during LCM was demonstrated on an experimental plaque mold (see Figure 1, page 27) fitted with temperature, pressure, and dielectric sensors located at strategic positions in the mold. The importance of time constants, response time, and measurement accuracy of a sensor was underscored by a comparison of the temperature response curve from a “twisted wire” thermocouple and a “metal sheathed” thermocouple (see Figure 2, page 28). In this comparison the “metal sheathed” thermocouple exhibited a much lower maximum temperature and different temperature profile than the “twisted wire” thermocouple. Other issues concerning sensor selection were mentioned briefly in the 1st LCM workshop report [3]. Also, the through-thickness temperature variations in the experimental plaque are significant (see Figure 3, page 29) and dependent on the location of the sensor within the mold. Hence, the cure state of the matrix and matrix sensitive composite properties may not be uniform throughout the composite part. In addition, the thermal and pressure histories measured in the plaques made by resin transfer molding (RTM, see Figure 6, page 32) or structural reaction injection molding (SRIM, see Figure 8, page 34) were found to depend on the location of the thermocouple within the plaque. During RTM the temperature rise due to the reaction of a polyurethane resin occurs at the plaque vent at least 15 seconds before the rise at the plaque gate (see Figure 5, page 31).

In RTM the mold pressure is also dependent on the location of the pressure transducer during the mold fill stage. However, during “pump-up” the pressure throughout the mold became more uniform and by the end of the injection the pressure distribution through the mold is uniform (see Figure 6, page 32). The pressure due to liquid resin expansion is highest near the inlet (see Figure 7, page 33). Since variations in pressure and temperature throughout the mold can affect void formation and the cure state of the resin, the discussion and presentation highlighted the need for having unobtrusive sensors in the manufacturing process to control the quality of the finished composite part.

## *Aerospace*

Aerospace composite applications are currently focused in the following applications: (1) Highly loaded, primary structures and (2) Large and/or contoured and/or integrated structures. The current process approach tends to be all experience based (trial and error).

Therefore, making a composite part with a new material and/or part shape requires the development of a new composite process. Unlike the automotive industry, composite technology in the aerospace industry is focused on high temperature materials (e.g. bismaleimides and polyimides (condensation)), tough materials (e.g., multi-phase prepreg and materials which phase separate during the curing process). In addition, there is a driving force toward low cost materials which meet the above requirements. As a result, the manufacturing processes for these materials should be low cost (w.r.t. Fiber placement, RTM, etc.) and integrated.

The current fabrication induced composite defects that are of concern to the aerospace industry are: (1) porosity (voids), (2) delamination, (3) cure state, and (4) dimensional tolerance. Since high temperature resins with complex cure kinetics are normally used in aerospace structural composites the resin cure state may be more critical than the presence of voids. Undercuring the resin results in a reduction in hot, wet properties and overcuring the resin results in a decrease in toughness. The cure profile of the composite resin can be affected by the variability in chemistry and age of the starting material as well as by variations in tooling and autoclave loading. The cure profile of the composite resin is controlled by process design, system thermal profiles, and material quality control. An example of the effect of the cure rate on the gel temperature for a 16 ply 3501-6/T300 composite is shown in Figure 34, page 61. For this composite, reducing the heating rate from 1.8 °F/min. to 0.5 °F/min. resulted in a reduction of 30 degrees in the gel temperature of the resin. The effect of the change in the cure cycle on the compression after impact (CAI) strength of an aerospace composite is shown in Figure 39, page 66.

Void formation is a concern because it affects resin dominated and fiber dominated properties. Interlaminar shear strength, a resin dominated property, is adversely affected by the presence of voids (see Figure 19-20, page 46-47). In addition, laminate compressive strength, normally considered a fiber dominated property, is also adversely affected by the presence of voids (see Figure 21, page 48). Longitudinal compression and microbuckling, also considered to be fiber dominated properties, are tied to the dependence of the critical lengths for shear failure and microbuckling in the composite matrix (see Figure 22-24, page 49-51). Since the matrix modulus is sensitive to void content, fiber dominated properties are also affected by voids. Void formation is difficult to predict. The factors which contribute to void formation are (1) moisture-humidity, (2) solvents, (3) entrapped air, (4) degree of fiber wetting, and (5) tooling. Although voids are a problem, most of the composites are made by autoclave processes and the appropriate application of pressure can usually minimize the presence of voids. In addition, voids are also minimized by controlling humidity, pre-preg tack, and the pre-preg film process.

Delaminations are considered a material specific effect. Because of its relationship to buckling stability, delaminations have a large impact on compression dominated designs. Delamination is typically controlled by good quality control, good tooling concepts, and by controlling the process rates. The impact of single and multiple delaminations on compression failure strain is shown in Figure 28, page 55. For example a single delamination causes a larger reduction in compression failure strain than 2% porosity.

## *The Manufacturing Environment*

The requirements placed on a sensor and control system by the need to operate in a real manufacturing environment were discussed in this topic which came directly from question three in the list (see page 2):

(3) What are the industrial environments in-line sensors must tolerate?

The discussion was initiated with a presentation by Foster Lamm of United Technologies Research Center (Figures 54-66 on pages 81-94) and moderated by John Fildes from Basic Industry Research Laboratory. A major conclusion from the session was summarized when Foster Lamm stated, "From a manufacturing view any new technology must impact the financial side of the business. It is not sufficient to be *Good Technology*."

There are two types of sensor configuration utilized in on-line process monitoring (1) internal probes and (2) extrinsic (including non-contact) probes. The presentation in this section focused on an ultrasonic sensor that was external to the processing environment (see Figure 64 on page 92). This configuration resulted in the development of a reliable cost effective sensor design. The presentation centered on the successful implementation of a cost effective and supportable adaptive process control system to minimize the cost of producing compression molded composites. In the conventional compression molding operation the process requires a significant amount of process knowledge. In addition, the process is 'set point' controlled and rheology must be run on every lot to confirm the state of the process.

In the adaptive process control scheme, control of the process is based on the current condition of the curing resin. The condition of the curing resin is monitored using an ultrasonic sensor whose output is calibrated to viscosity changes in the curing resin. To effect adaptive process control, the information from the sensor is analyzed using a rules based approach. However, adaptive process control cannot make up for bad tooling and bad starting materials.

Dielectric sensors are used in the development of structural parts for aerospace applications. These sensors are also external to the process. The selection of the appropriate sensor will depend on what is to be measured, the chemistry of the resin system, the curing temperature, or the pressures used in fabrication the composite part. Once the appropriate sensor has been selected, making good connections between the information provided by the sensor and the properties of the part to be controlled is typically the major concern with all types of sensors.

## *High Speed Processing of Composites*

For industries where production volumes are large, like automotive, the need for high speed fabrication is essential, and this has consequences for the use of sensors. This topic relates to question four in the list:

- (4) What are the effects of manufacturing speed on the requirements for sensors, NDE, and controls (i.e. what is the role of this technology in industries like automotive where speed is critical)?

Thinking in this area was stimulated by a presentation from Bruce Greve of the Budd Company (see Figures 86-111 on pages 115-141), and the discussion was moderated by Richard Parnas of NIST.

Currently SMC products, such as hood, trunk lids, etc., have the highest yearly volume. However, these parts tend to be of very low structural complexity. Liquid molding parts (*e.g.*, passenger compartment body frames, pick up truck cabs, floor parts, and body side operatives) span the range of structural complexity, but have low yearly production volumes. The market is currently moving toward high structural complexity liquid molded composite parts with a significant increase in yearly volume.

The role of sensors in this manufacturing regime is to (1) improve process performance, (2) expand automation, (3) assure component quality, (4) minimize value added scrap, (5) reduce costs, and (6) provide documentation. For sensors to be successful, they must (1) survive the manufacturing environment, (2) have response times of 1 minute or less, (3) require low maintenance, and (4) be simple to operate. Sensors are typically used to monitor fabrication equipment and the component that is being fabricated. With respect to the fabrication equipment, sensors are commonly need to monitor pressure, vacuum, temperature, component ratios, and cure time. Component monitoring, which has been more widely discussed at this workshop, involves the measurement of the following parameters: (1) weight, (2) void content, (3) glass content, (4) degree of cure, (5) dimensional accuracy, and (6) assembly accuracy.

Although esthetic quality requirements are more stringent for appearance parts than for structural parts, there is a need for void detection sensors in foam core structures. In addition, there is a critical need for sensors that can inspect preforms and determine the fiber orientation and the amount of fiber present.

## *Role of NDE*

The field of NDE is well established, and this experience has much to offer the area of on-line process monitoring. Consequently, this topic is appropriate for this meeting. It relates to the fourth question in the list but is considerably more general:

- (4) What are the effects of manufacturing speed on the requirements for sensors, NDE, and controls (i.e. what is the role of this technology in industries like automotive where speed is critical)?

The discussion began with remarks from Boro Djordjevic (see Figures 67-85 on pages 95-114), and the discussion was moderated by Robert Green, both from The Johns Hopkins University.

Classic NDE focuses on characterizing flaws and other defects in a part after fabrication and during its service life. In recent years, however, the distinction between this and on-line monitoring has begun to blur because the information sought and the measurement techniques used for the two applications strongly overlap. Historically, NDE has used a broad range of sensors including ultrasonics, acoustic emission, X-ray, and optical or thermal methods. More recently, studies have added laser ultrasonic, x-ray tomography, holography, etc. (see Figure 69 on page 98 for other NDE techniques). The NDE technologies are particularly adept in detecting defects such as delaminations, porosity, voids, misoriented fibers & plies, etc. (see Figure 68 on page 97 for other examples). Parameters and properties of organic matrices that are amenable to process control are shown in Figure 70, page 99. Devices that have been targeted for organic matrices are shown in Figure 71, page 100. One important consideration for any measurement technique is that the detection sensor should cause minimal interference in the fabrication process. A second critical issue in process monitoring is what features in the material are important and which process variables can be controlled.

A promising technique for process monitoring is embedded acoustic sensors. They fall into three classes (1) bulk wave, (2) surface wave, and (3) guided wave sensors. The latter is dependent on the material geometry. Another promising technology is fiber optics using evanescent wave absorption (see in Figures 72 & 73, page 101 & 102.). Other emerging techniques that have the attractive feature of non-contact measurements are ultrasonics with laser generation and detection, gas coupled ultrasonics, and microwave sensors (see Figures 79-84. on pages 108-113). Recall that in the previous presentation the ultrasonics sensor utilized in that process was a non-contact ultrasonics sensor.

## *Impact of Preforms on Sensors*

Preform manufacturing is a critical issue not only for high speed fabrication but for the entire range of applications with LCM, RTM, and structural reaction injection molding (SRIM). Since sensors could have an important role in this topic, it was discussed at the meeting. A presentation by Daniel Buckley of American GFM (slides not available) introduced the area, and the discussions were moderated by Richard Parnas of NIST.

The technology developed by the aerospace industry to meet stringent performance criteria in composites is being applied to the manufacture of sporting goods equipment. Current automotive technology in the making of structural cross-members requires 6 hours and 3 people. Because this industry is cost driven, the present goal is to make this part in 6 minutes.

Preforms tend to fall into three categories: (1) foam core, (2) shells - random fibers, and (3) woven fiber mats. A number of promising new techniques for preform manufacturing are under development. The conventional spray up method is labor intensive and does not provide good control of fiber orientation. An automated version sometimes called the directed fiber process is now being tested, and this should be a significant improvement. Another new method is the slurry process. In this approach, the reinforcement is suspended in the slurry and gets deposited on a screen when the slurry is forced through the mesh. Proper application of this method results in rapid production of a rigid preform with semi-random orientation. As with spray up techniques, the challenge is to obtain the proper density and orientation of the fibers in a preform with good dimensional control. If the final preforms must be trimmed, this adds cost. The automotive industry is pushing for net shape preforms.

Thermoforming is another fabrication method that is under study. In this technique, control of fiber location and orientation can be a problem if the shapes are complex. Much of the work to date has involved random orientation of the fibers, and this generally limits the preforms that are made to non-load bearing applications. A final example of a new fabrication method is called the "compform" process. It incorporates high speed ultrasonic cutting to generate pre-cut shapes that are placed into a compression tool. A binder that can be UV cured is partially reacted to produce rigid preforms. This process has been used with a variety of reinforcements, *i.e.*, glass, Kevlar, and carbon.

With any of these approaches, it is critical to assess and control the fiber type, density, and orientation at each location in the preform. A sensor and control technology which could do this would be a major advance in the field.

## *Results of the Questionnaire*

To get a clear view of the attendee's opinions on critical issues, a questionnaire was distributed to each participant (excluding the NIST attendees) at the beginning of the meeting. These forms were completed during the course of the conference, and a summary of the results was presented in the discussion period. The tabulated results which are given on pages 18 and 19 show that the participants represent a variety of industries. Among the attendees, 65% indicated that they are currently using sensors in some form. With regard to microstructure, a wide range of features were viewed as important but void content and degree of cure ranked highest. Off-line characterization tools were also widely used. Although ultrasonic inspection and spectroscopic examination of raw materials were highly cited, a broad range of tools were considered important. Not surprisingly, the workshop listed temperature and pressure probes as the most widely used on-line sensors. This undoubtedly reflects the current availability of these technologies, and the fact that they measure the variables that can be controlled. Nevertheless, the results also show a strong interest in sensors that measure the "state" of the material and not just the processing environment.

When asked what was most important in a sensor, the response indicated that the sensor must be rugged and provide reliable data. Without this, other factors such as speed, cost, and sensitivity do not matter. If a sensor meets the rugged and reliable criterion, cost was ranked as the next most important consideration. Reasons for using a sensor varied widely which probably reflects the diversity of problems associated with the broad range of applications for composites. Finally, when asked to indicate what part of the process they would most like to control, the attendees listed all aspects as important, but ranked raw materials quality and cure as the highest priorities.

**Results of Questionnaire for  
Industry Workshop for On-line Composite Process Monitoring**

- (1) What Industry Segment do you represent or would like to respond for?
- (1) Aerospace 38.4% (4) Construction 7.7%  
 (2) Automotive 23.1% (5) Other 23.1% Manufacturing; Controls; Research  
 (3) Electronics 7.7%

- (2) Do you or your Company use sensors or would like to use sensors?
- Yes We are Currently Using Sensors 75%  
 Yes We Would Like to 25%  
 No 0%

*Rank Numerically the responses in each of the following questions with 1 as most important*

*Tabulated values represent the average rank based on participant responses.*

- (3) What microstructural properties of the composite do you think are most important in your company's application? (*Please Rank*)

<u>2.3</u> Degree of Cure	<u>3.6</u> Moisture Content
<u>1.7</u> Void Content	<u>4.7</u> Temperature Profile during Cure
<u>3.4</u> Residual Cure Stresses	<u>6.0</u> Refractive Index
<u>3.6</u> Rate of Cure	<u>3.4</u> Other (Specify) <u>Viscosity, % Reinforcement, Consolidation, Customer Specs; Foreign Materials</u>

- (4) What Off line Characterization Techniques do you consider important? (*Please Rank*)

<u>2.9</u> ASTM Void Content Procedure	<u>2.1</u> Ultrasonics
<u>2.3</u> Spectroscopic Characterization of Raw Materials	<u>3.4</u> Digital Shearography
<u>4.1</u> Electronic Speckle Pattern Interferometry (ESPI)	<u>4.9</u> Dye Penetrant Techniques
<u>3.6</u> Thermal Techniques (e.g. SPATE & Thermal Conductivity)	
<u>      </u> Other (Specify) <u>x-Ray, Thermomechanical Analysis, Mechanical Testing, FTIR/Optical Time Domain Reflectometry, DSC, DMA</u>	

(5) What type of on line sensors do you think are most useful to the manufacture of composites in your company's application? (**Please Rank**)

<u>2.8</u> Dielectric Sensors	<u>2.3</u> Fiber Optics Sensors
<u>3.0</u> Ultrasonic Sensors	<u>2.0</u> Temperature Sensors
<u>2.0</u> Other (Specify) <u>Pressure Transducers</u>	

---

(6) In a sensor what do you consider important? (*Please Rank*)

<u>3.6</u> Response Time	<u>4.6</u> Sample Size
<u>2.9</u> Cost	<u>3.9</u> Sensitivity
<u>2.1</u> Accuracy & Precision	<u>4.8</u> Range
<u>2.0</u> Other (Specify) <u>Durability, Reliability; Survivability</u>	

---

(7) Why would you use a sensor in your manufacturing process? (*Please Rank*)

<u>1.6</u> Reduce defects	<u>2.0</u> Optimize Processing
<u>2.5</u> Reduce Scrap	
<u>3.3</u> Relax raw material specifications or overcome variability in raw materials	
<u>2.0</u> Other (Specify) <u>Process Control, Rate</u>	

---

(8) What part of the production process do you consider most important to control? (*Please Rank*)

<u>2.2</u> Quality and physical properties of raw material feed
<u>3.1</u> Controlling the production environment (humidity, temperature)
<u>2.2</u> Controlling the cure process
<u>3.8</u> Controlling the mold preparation step
<u>3.3</u> Controlling the mold filling step
<u>3.0</u> Other (Specify) <u>Microstructure, Dimensional Tolerance, Control Entire Process</u>

## Summary Discussion Session

The closing discussion addressed three issues in some detail. The format involved offering a question which elicited a series of responses, comments, and sub-questions from attendees.

**The first issue considered was Preforms.**

**Question: What do we want to measure?**

- ~ Permeability. This cannot be measured directly with an on-line sensor, but we can measure the factors that control or influence permeability such as
  - ~ Thickness of final preform
    - Comment:* Control of preform thickness is a tooling problem
  - ~ Preform deformations during injections
  - ~ Difference in volume fraction and orientation around curve
  - ~ Contour changes and shape changes (*i.e.*, sense geometry of preform)
  - ~ Ply orientation. This affects permeability but also directly controls important properties in the final part
    - Issue:* Do we have the right amount of material where we need it? Metal or tracer fibers in rovings can be used to follow the orientation.
    - Query:* Is orientation part of the process and more importantly is there variability in orientation?
      - Response:* Yes, but orientation is usually too complex to deal with so we often just try to over design the part and cross our fingers!
- ~ Preform integrity - are the fibers where we need them?
  - Comment:* Determination of biaxial or triaxial, orientation is a very complex problem. It is often circumvented by over designing the part.
- ~ Mold closure operation can be an issue since it can change fiber position and orientation

**Question:** If we measure one thing on a preform what would it be?

- ~ For virtually all applications we want to know fiber volume fraction.

**Comment:** In processes such as RTM, the overall fiber volume fraction is dictated by the mold and preform, but can still vary from one location to another. We need to know the fiber content in critical regions not just the overall average.

- ~ In high performance composites, fiber content is not adequate, orientation of the fibers is also critical.

**Comment:** The aerospace industry buys preforms and hopes they conform to specs. Unfortunately, preforms can be quite variable.

**Query:** Would 3-D image microstructure be useful ?

**Response:** Yes

## The second area discussed was the Molding Operation

**Question:** What end use properties of the composite are both desirable and difficult to control?

*In Aerospace these properties are:*

- ~ Compression strength. It depends on features like voids and porosity.

**Comment:** Post processing inspection for voids is well established. We can use C-scan, ultrasonics, and X-Ray tomography. On-line detection would be very useful.

Process variability can cause changes in porosity, and this is more difficult to detect. Advances here would be very desirable.

- ~ Fiber waviness in thick composite

**Comment:** Tracer fibers can be included and examined in post processing inspection, but fiber waviness cannot be detected if it is less than 2-3% in a 500 ply lay up!

**Query:** Is waviness a random event? Where is it critical? Where will it occur the most?

**Response:** Known sources include (1) corners where lots of plies come together, (2) areas with large flow gradients during processing, (3) poor manufacturing of a preform, and (4) deformation during mold closure.

~ Cure level

**In Automotive these properties are:**

~ Voids

~ Resin rich areas

~ Loss of architecture

**Comment:** The problems with aerospace and automotive are similar here.

**Query:** But what about processing speed?

**Response:** This increases the problem for applications like automotive but usually these applications also have less severe performance requirements.

~ Gel time control is critical because it controls when the mold can be opened and therefore cycle time.

~ Fiber wetting is particularly important in fast processes

**Comment:** Even in a slow process like vacuum infiltration molding, fiber wetting can be a concern.

~ Microstructure of interface

**Comment:** Drzal's work shows that fiber-polymer bonding is a function of many things: processing speed, sizings, etc.

~ Resin cure

~ Exotherm and temperature control is still a problem in thick composites.

## **The final issue discussed was the Industrial Environment**

**Question: What characteristics must a sensor have if it is to be used in an Industrial Environment?**

- ~ A sensor must be transparent to the process.
- ~ Embedded sensors usually appear to be inclusions (Hence, thin sensors must be developed)
- ~ Sensors are needed that have a strong application to physical properties

## REFERENCES

- 1 P. Beardmore and D. Hunston, "Polymer Composite Processing (An Industry Workshop)", NBSIR 87-3686, National Bureau of Standards, Gaithersburg, MD, February, 1988.
- 2 C. Johnson, S.S. Chang, and D. Hunston, "Polymer Composite Processing (2nd Industry Workshop)", NISTIR 4461, National Institute of Standards and Technology, Gaithersburg, MD, December 1990.
- 3 R.S. Parnas, A.J. Salem, K.N. Kendall, M.V. Bruschke, "Report on the Workshop on Manufacturing Polymer Composites by Liquid Molding", National Institute of Standards and Technology, Gaithersburg, MD, February 1994.
- 4 M. A. A. Spaid, F. R. Phelan, Jr., "Lattice Boltzmann Methods for Modeling Microscale Flow in Fibrous Porous Media", Submitted to Physics of Fluids.

# **Presentations**

**Process Monitoring for Composites**  
**Manufacturing in the Automotive Industry**

**Kenneth N. Kendall**

**Ford Motor Company**

# Experimental Plaque Mold

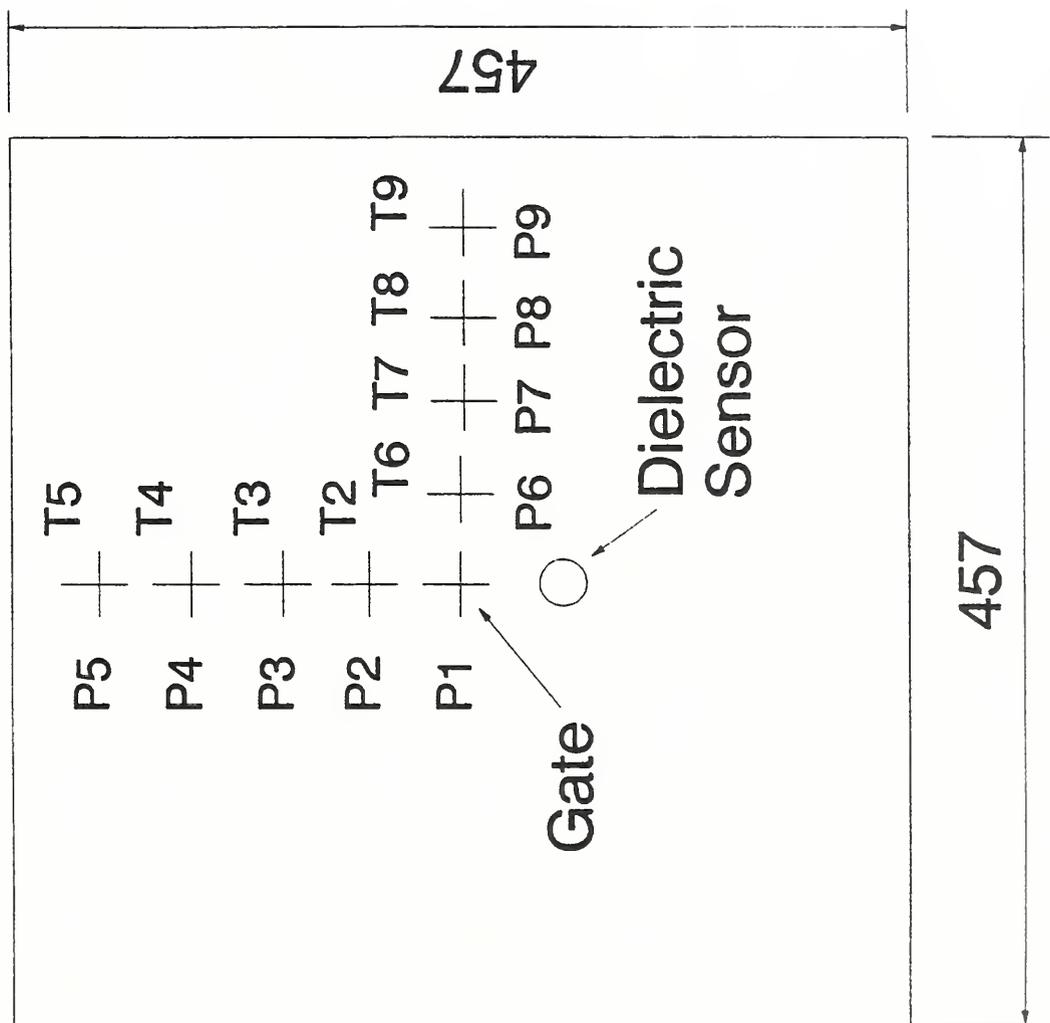
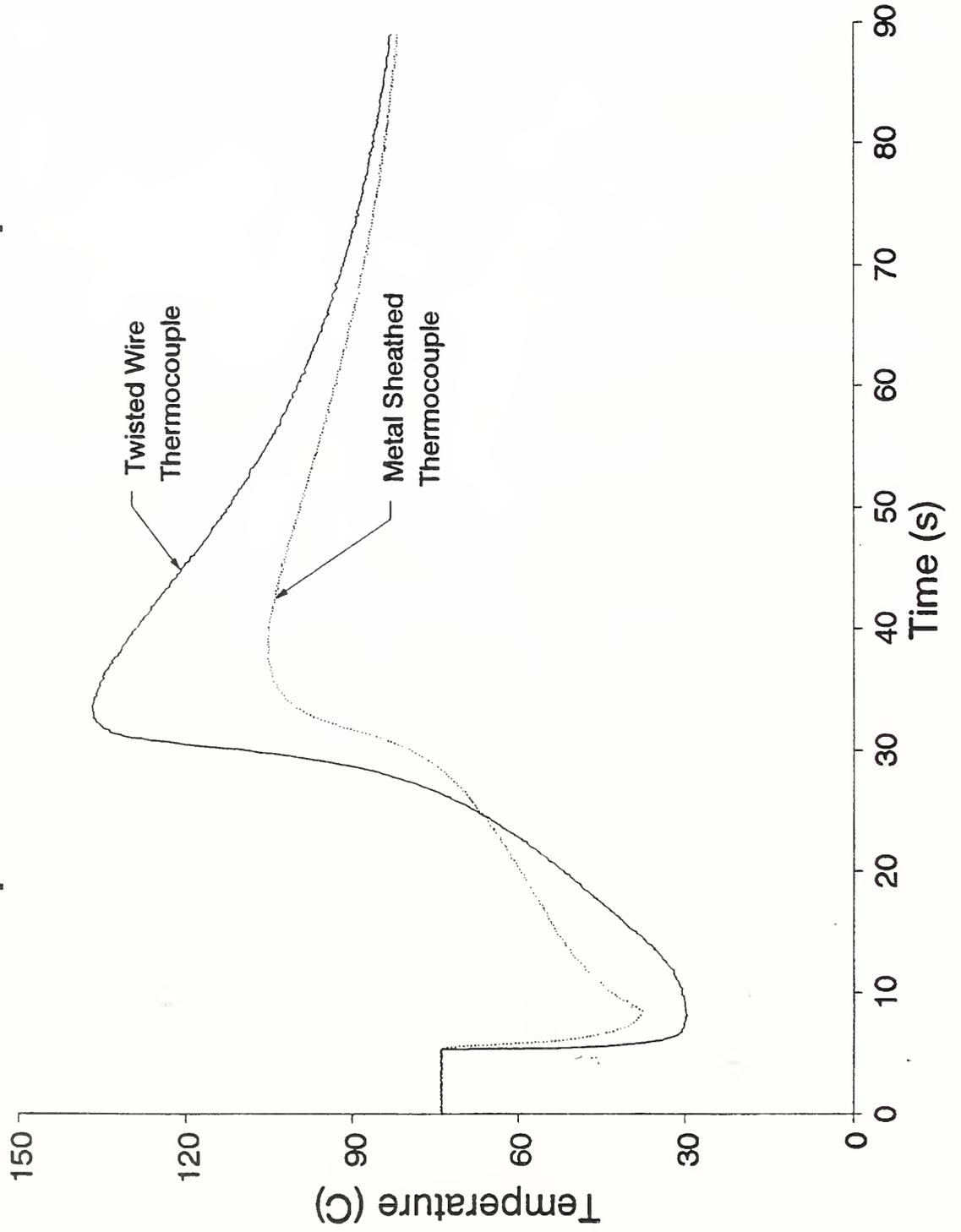


Figure 1

Figure 2

# Comparison of Thermocouples



# Through-Thickness Temperature Variations in LCM

Figure 3

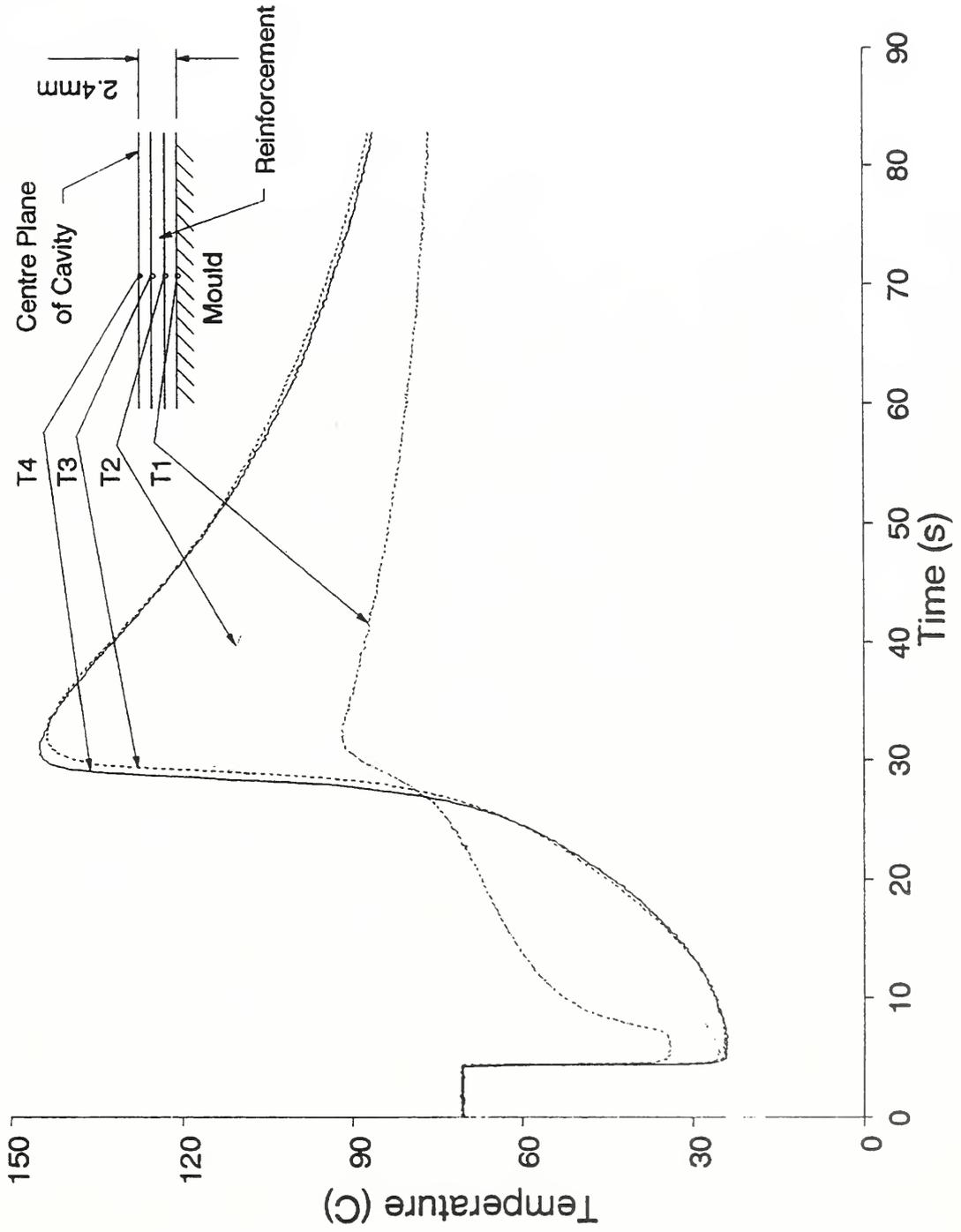
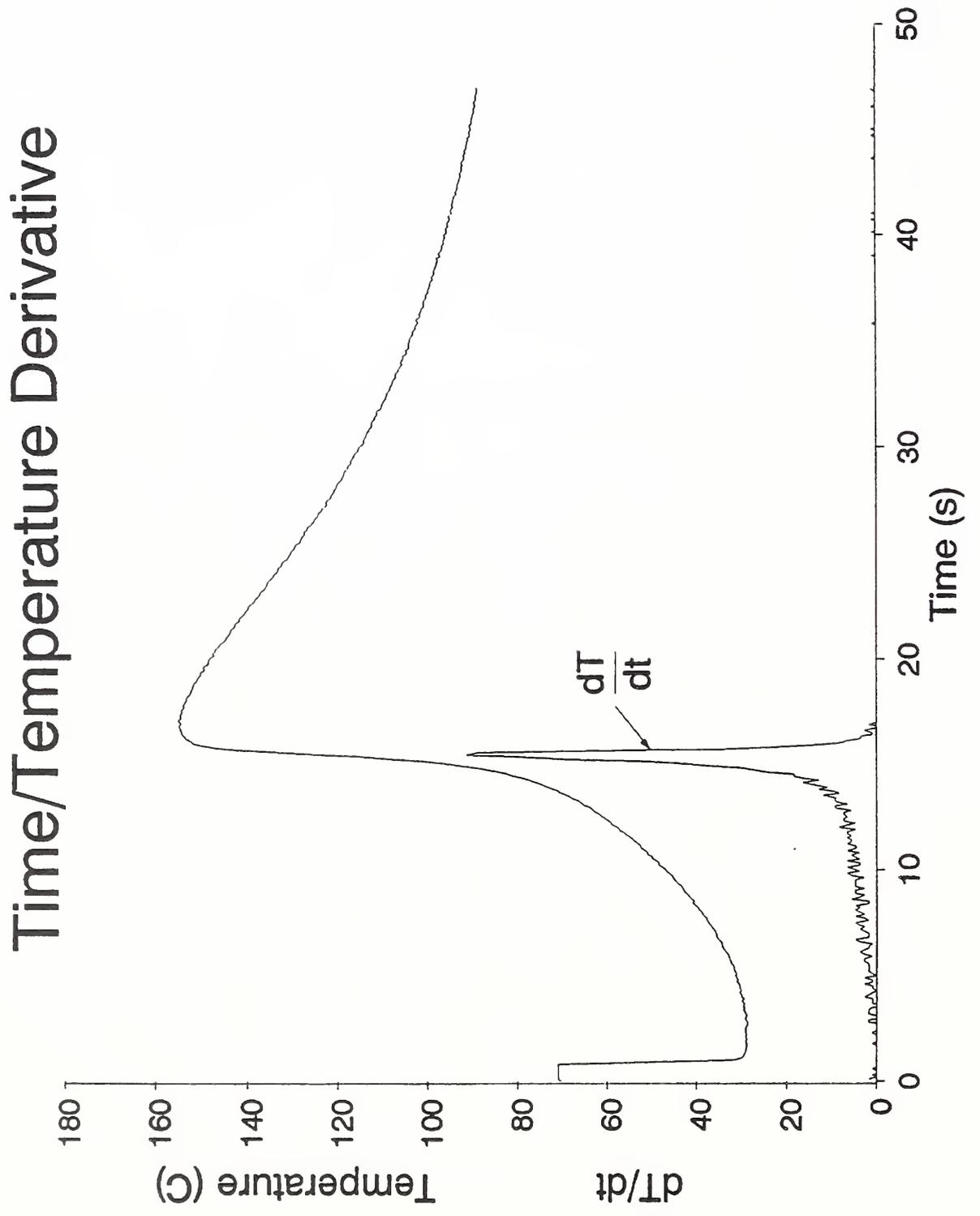


Figure 4



# Thermal History in LCM

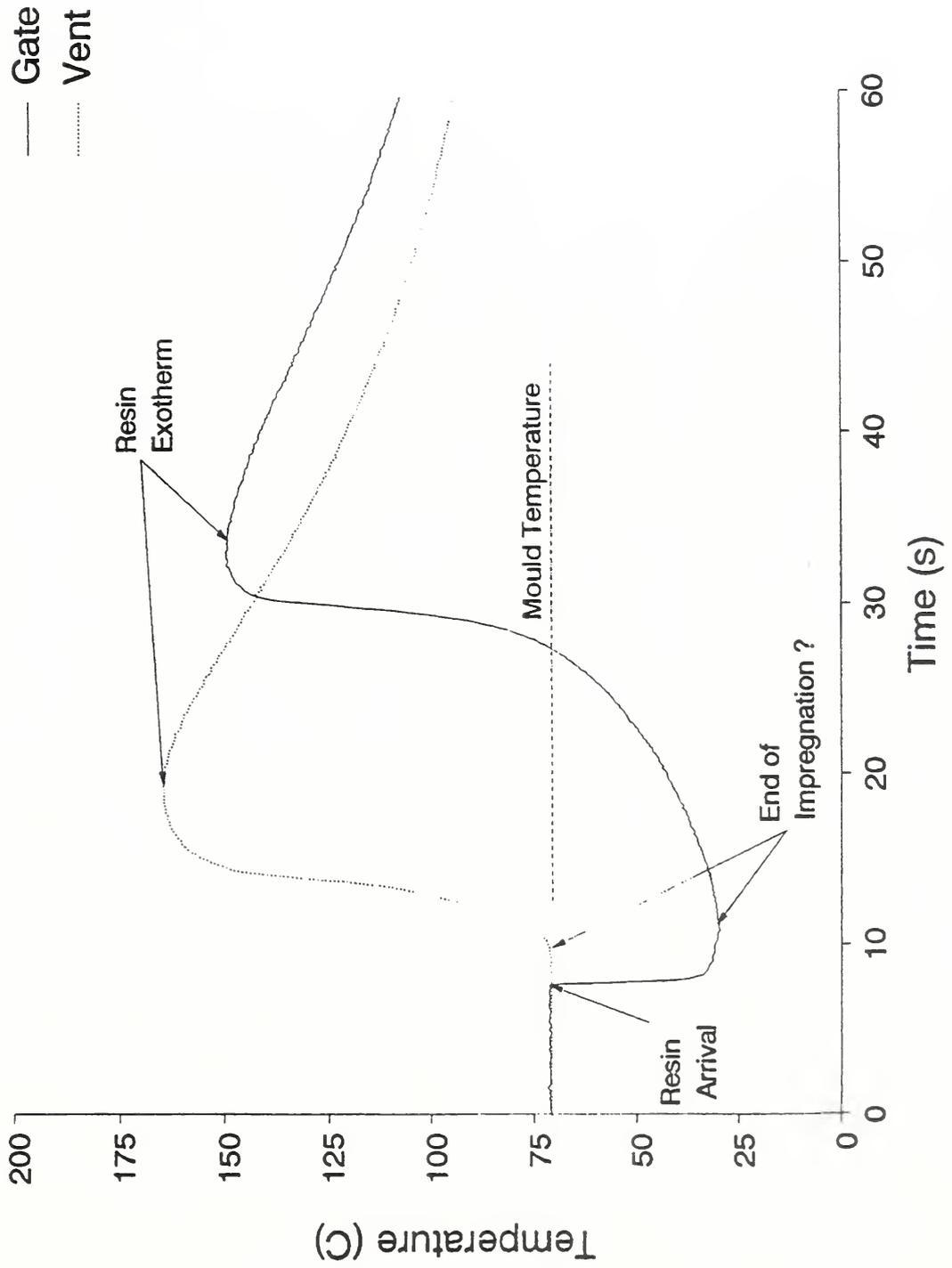
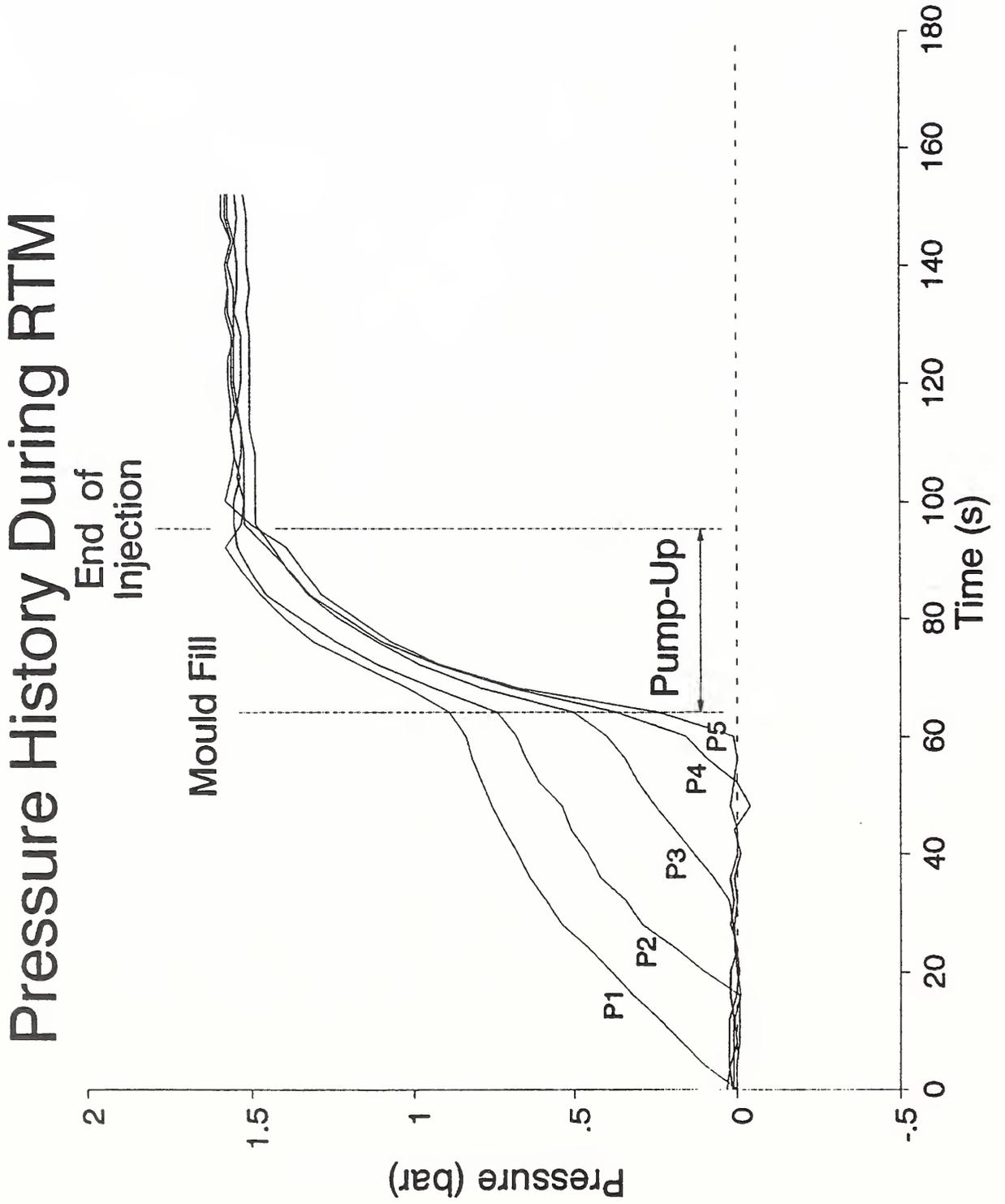


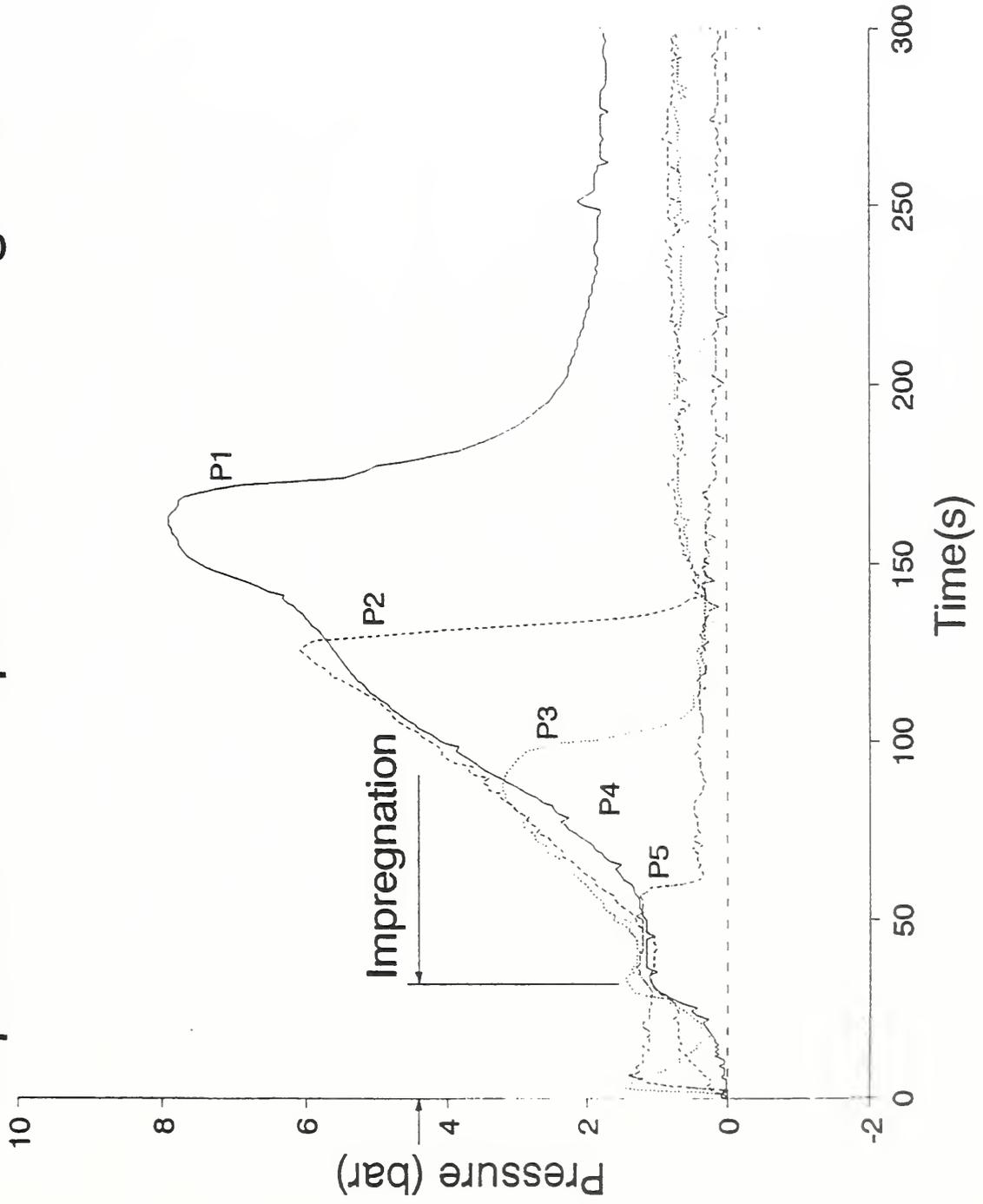
Figure 5

Figure 6



# Expansion of Liquid Resin During RTM

Figure 7



# Flow Progression in SRIM

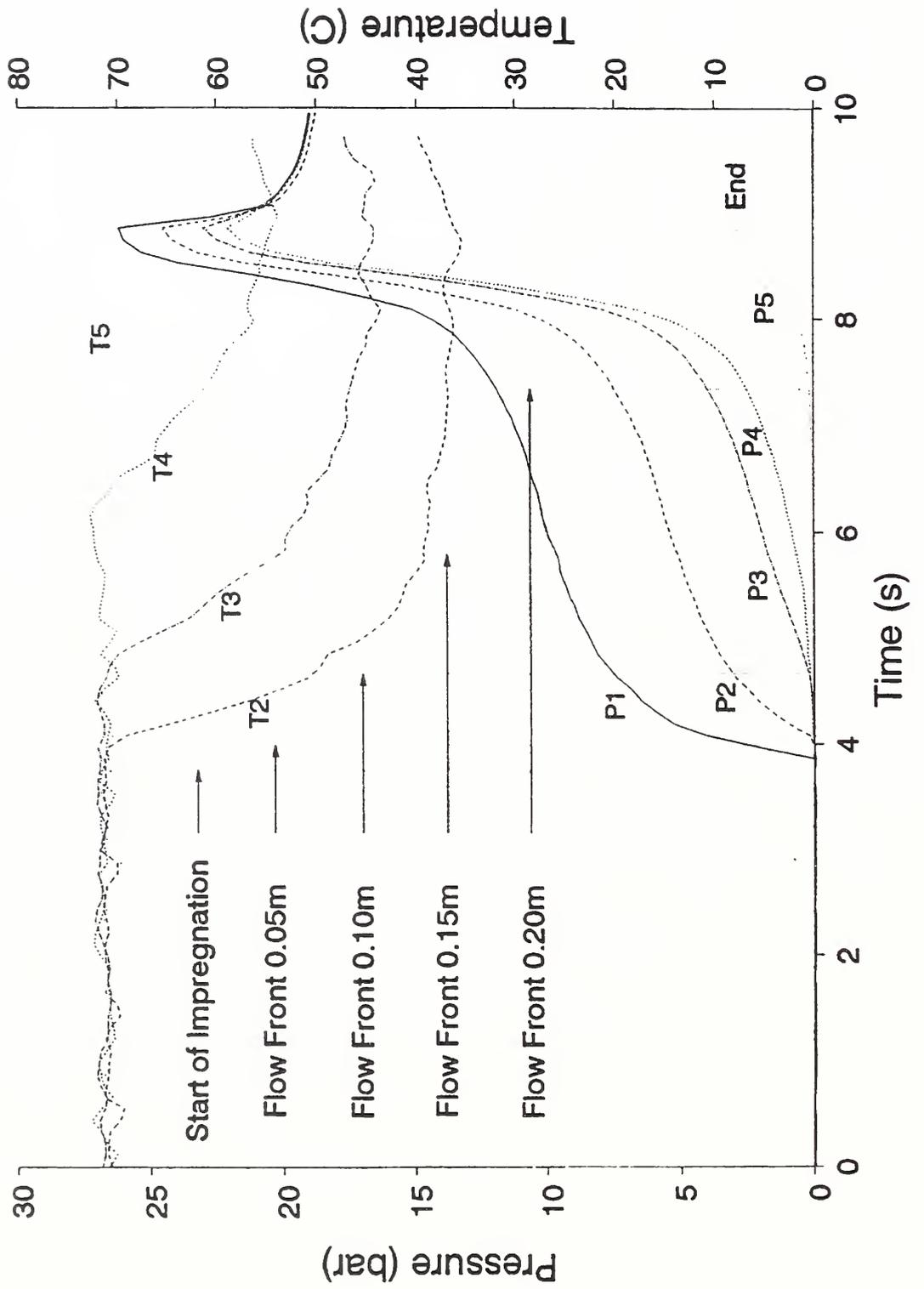


Figure 8

# Pressure History During SRIM

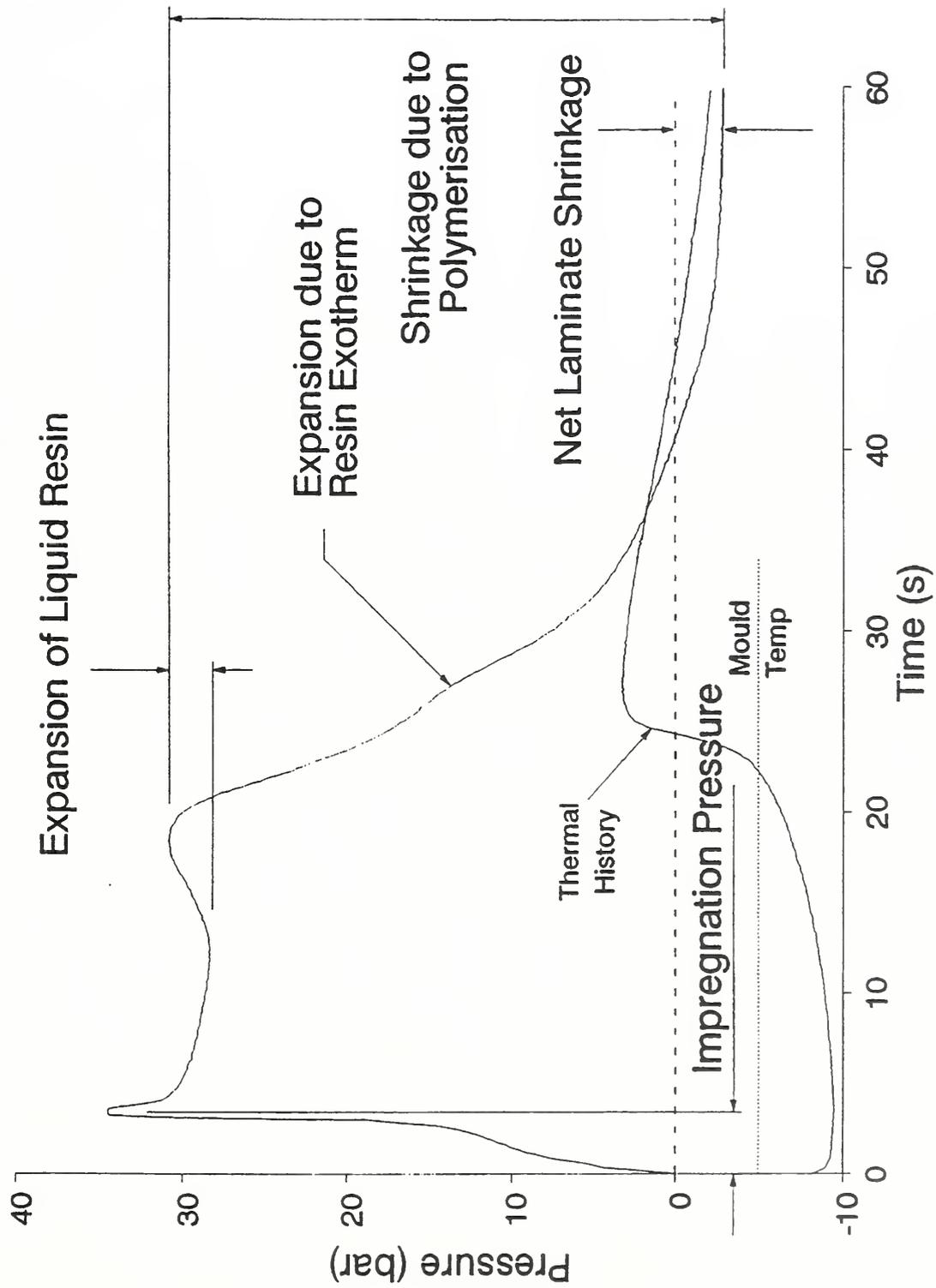


Figure 9

Figure 10

# Pressure Distribution During SRIM Impregnation

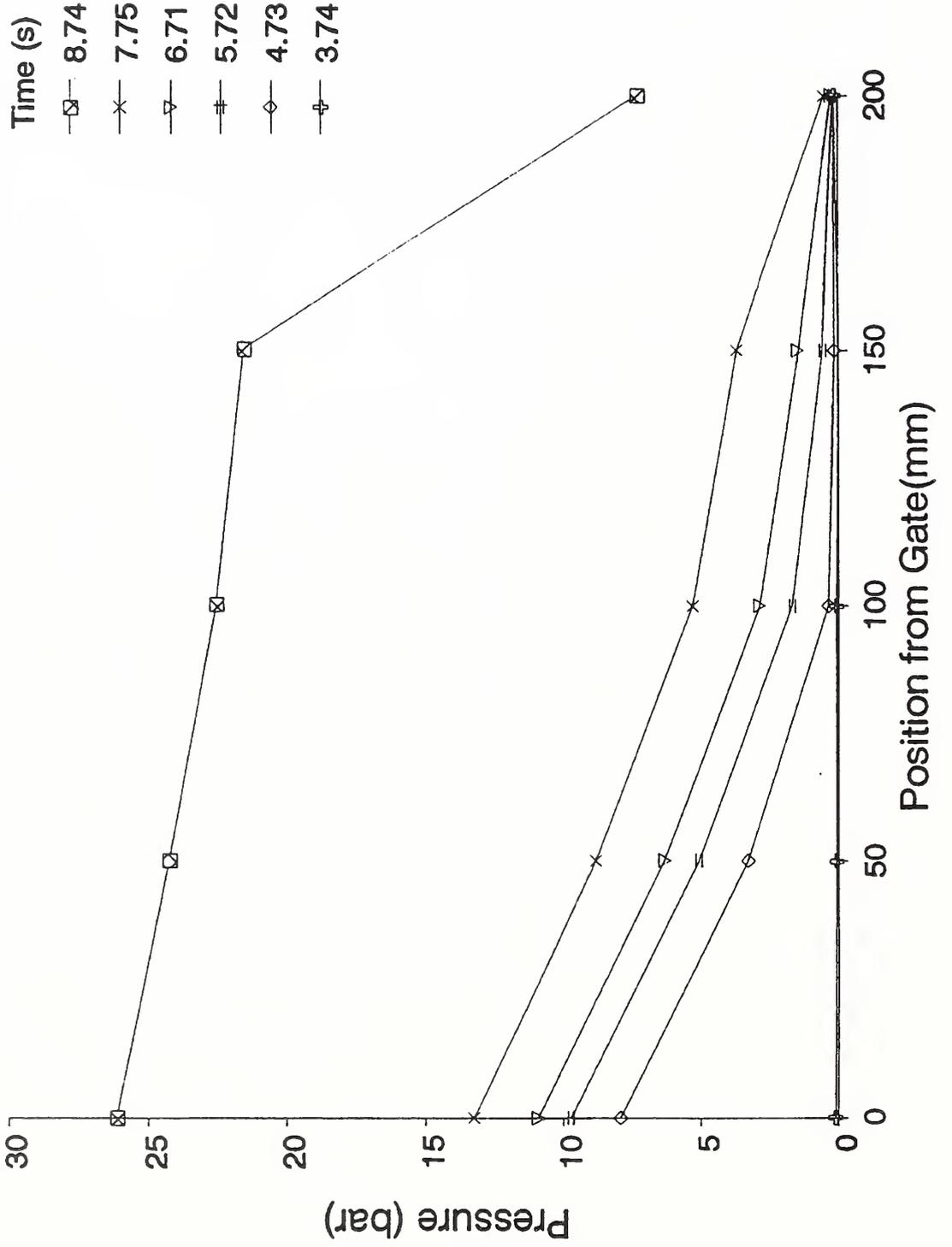
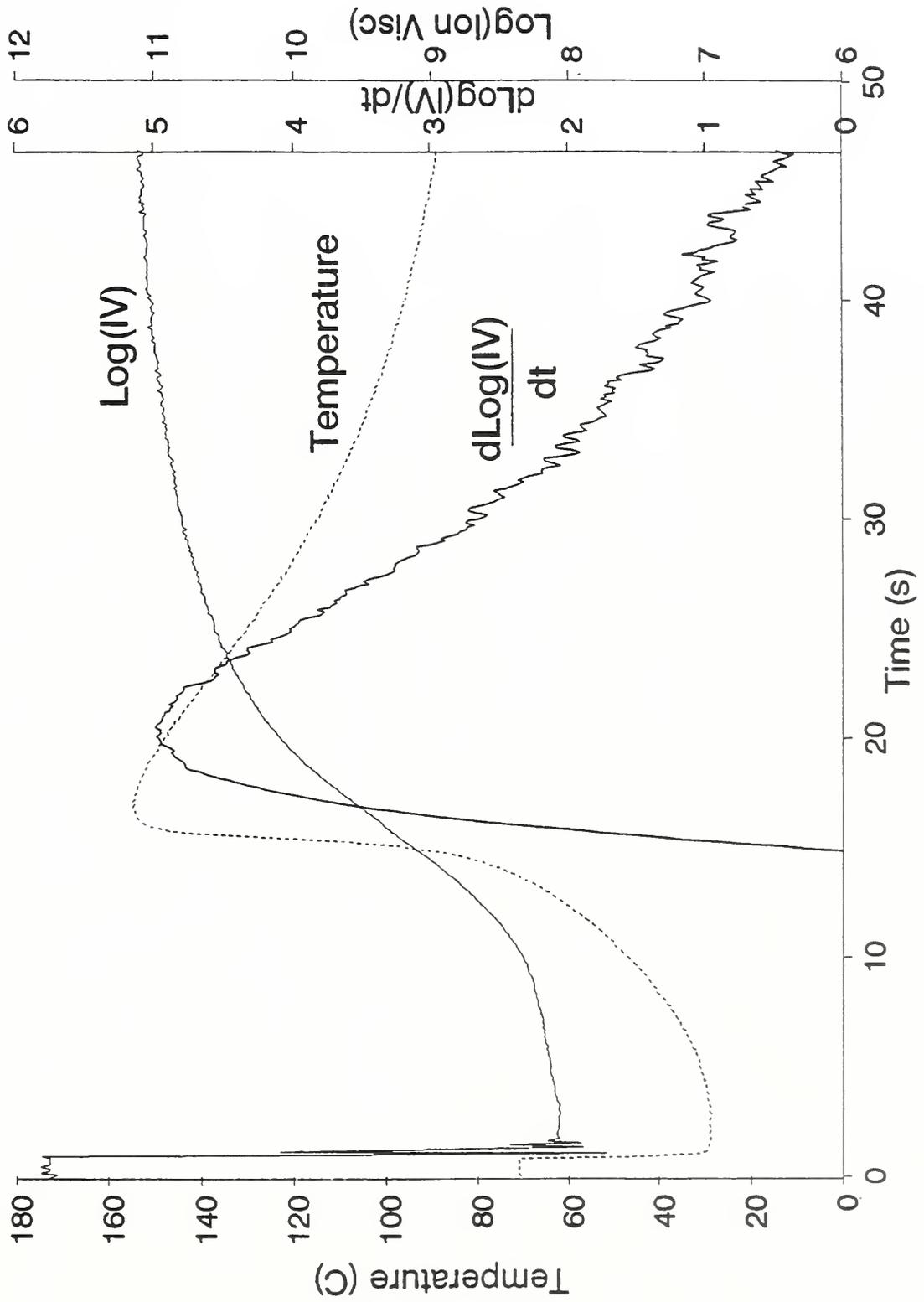


Figure 11

# Dielectric Measurements in SRIM



**FABRICATION DEFECTS IN COMPOSITES**

**T. M. DONNELLAN**

**INDUSTRY WORKSHOP FOR ON-LINE  
COMPOSITES PROCESS MONITORING**

**APRIL 3 - 4, 1996**

**AT&T**

**NORTHROP GRUMMAN**

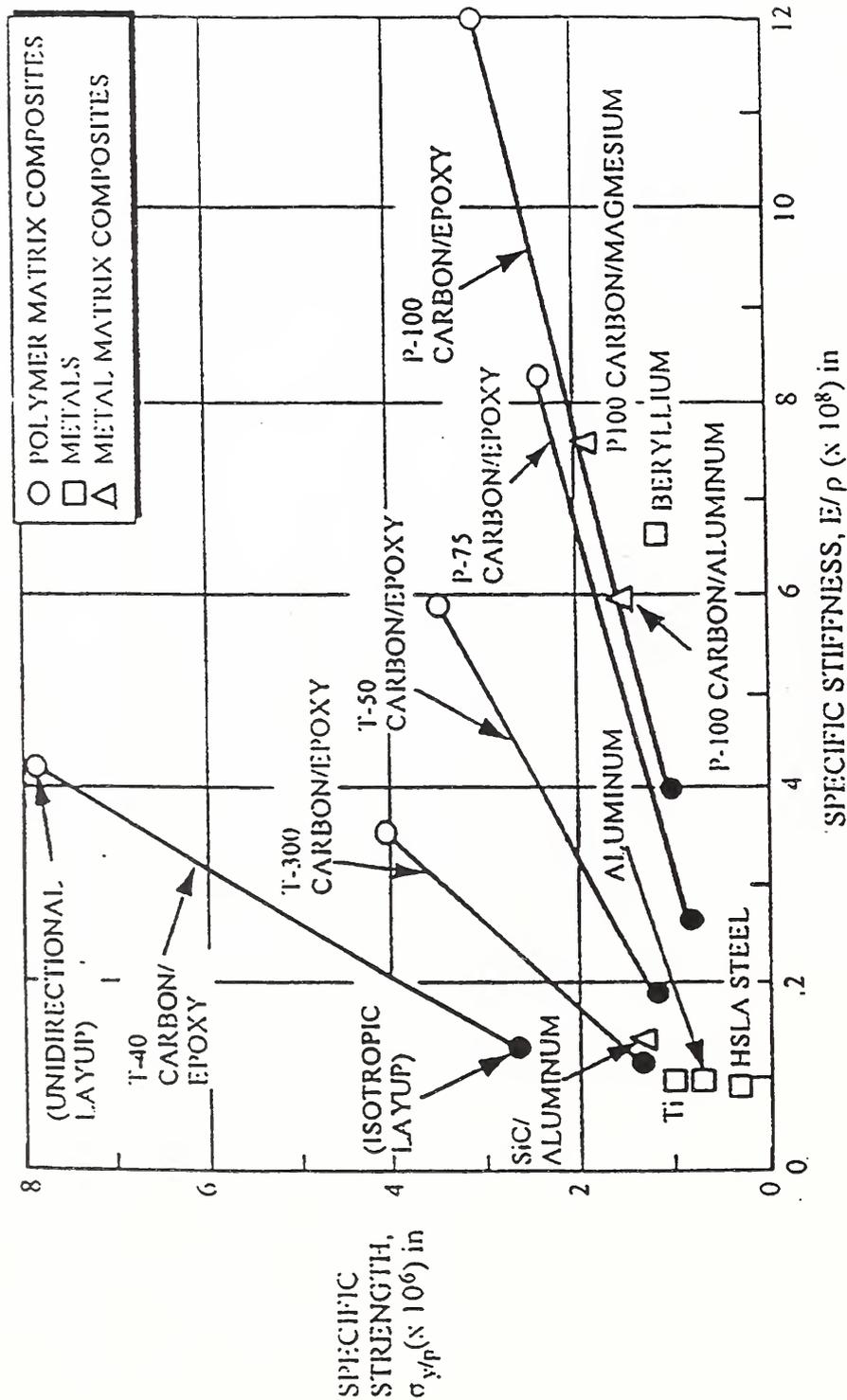
# **OUTLINE**

- **COMPOSITES IN AEROSPACE APPLICATIONS**
  - **EFFECTS OF DEFECTS STUDIES**
- **PAST SENSOR TECHNOLOGY WORK**
- **SENSOR TECHNOLOGY NEEDS**

Figure 12

Figure 13

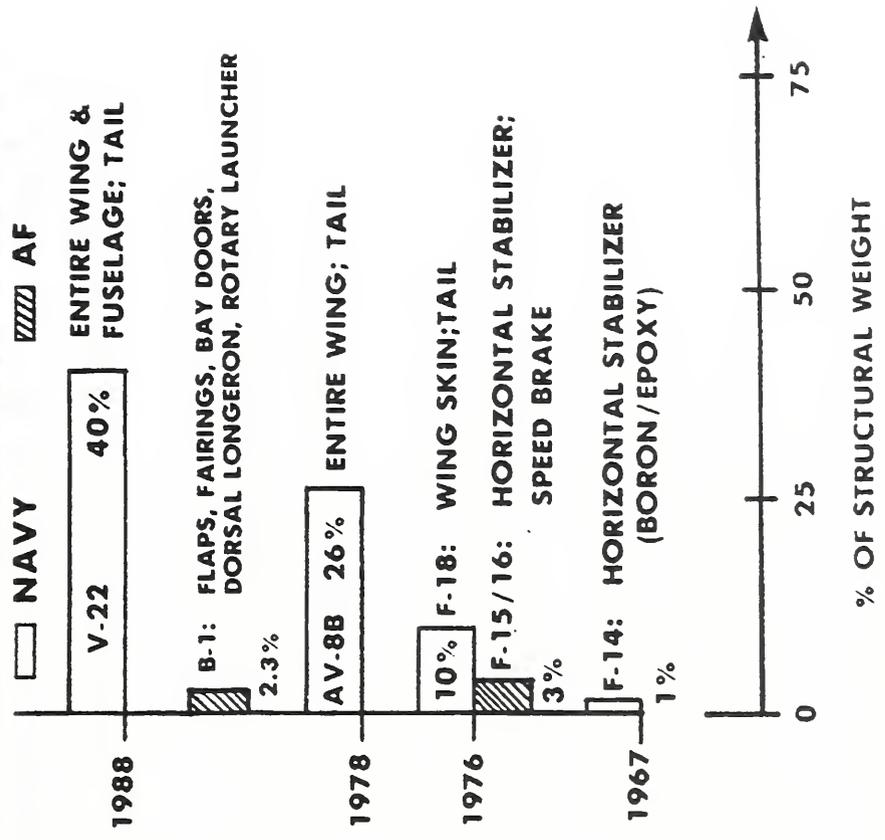
# COMPOSITE PROPERTY COMPARISONS



WE DAVIS "EMERGING TRENDS IN COMPOSITE MATERIALS"  
 APPLICABLE TO SPACECRAFT STRUCTURES  
 DESIGN OF COMPOSITE SPACECRAFT STRUCTURES  
 VIDEO CONFERENCE WORK SHEET  
 11/11/81

Figure 14

**ADVANCED COMPOSITES IN NAVY AIRCRAFT**



**HISTORICAL AGGRESSIVE COMPOSITES UTILIZATION CONTRIBUTES SIGNIFICANTLY TO NAVY MISSION CAPABILITIES:**

# AEROSPACE COMPOSITES TECHNOLOGY

- HIGHLY LOADED, PRIMARY STRUCTURE
- LARGE AND/OR CONTOURED AND/OR INTEGRATED STRUCTURES
- TRIAL AND ERROR PROCESS SPACE DEFINITION REQUIRED (TOOL CONCEPTS, PROCESS PARAMETERS, MATERIALS AND PROCESS SPECS)

Figure 15



# AEROSPACE COMPOSITES TECHNOLOGY

## TECHNOLOGY THRUSTS

- **MATERIALS**
  - HIGH TEMPERATURE (BMI, PT, PI (CONDENSATION))
  - TOUGHNESS (MULTI-PHASE PRE PREG, PHASE SEPARATION ON CURE)
  - LOW COST
  
- **PROCESSES**
  - INTEGRATED
  - LOW COST (FIBER PLACEMENT, RTM, ...)



*NORTHROP GRUMMAN*

Figure 16

## **FABRICATION INDUCED COMPOSITE DEFECTS**

- **POROSITY (MOISTURE-HUMIDITY, SOLVENTS, AIR, DEGREE OF WETTING, TOOLING)**
- **DELAMINATION (FOREIGN FILMS, TOOLING, AIR)**
- **CURE STATE (TOOLING, LOCATION IN AUTOCLAVE, PART COMPLEXITY)**
- **DIMENSIONAL TOLERANCE (TOOLING, DESIGN, MATERIAL VARIANCE, PROCESS CONDITIONS)**



# POROSITY

- ACCEPTABLE LEVELS BASED ON DESIGN, EFFECTS OF DEFECTS STUDIES
- TYPICAL ALLOWABLE LEVEL 1/2 IN. X 1/2 IN. AREA WITH 6 IN. SEPARATION BETWEEN AREAS
- AFFECTS RESIN DOMINANT AND FIBER DOMINANT PROPERTIES
- MINIMIZE BY CONTROL OF HUMIDITY, PRE-PREG TACK, PRE-PREG FILM PROCESS, AUTOCLAVE PRESSURE
- FORMATION CAN BE DIFFICULT TO PREDICT

EXAMPLES: YAV-8B WING  
F-14 OVERWING FAIRING  
F/A-18 STABILIZERS



*NORTHROP GRUMMAN*

Figure 19

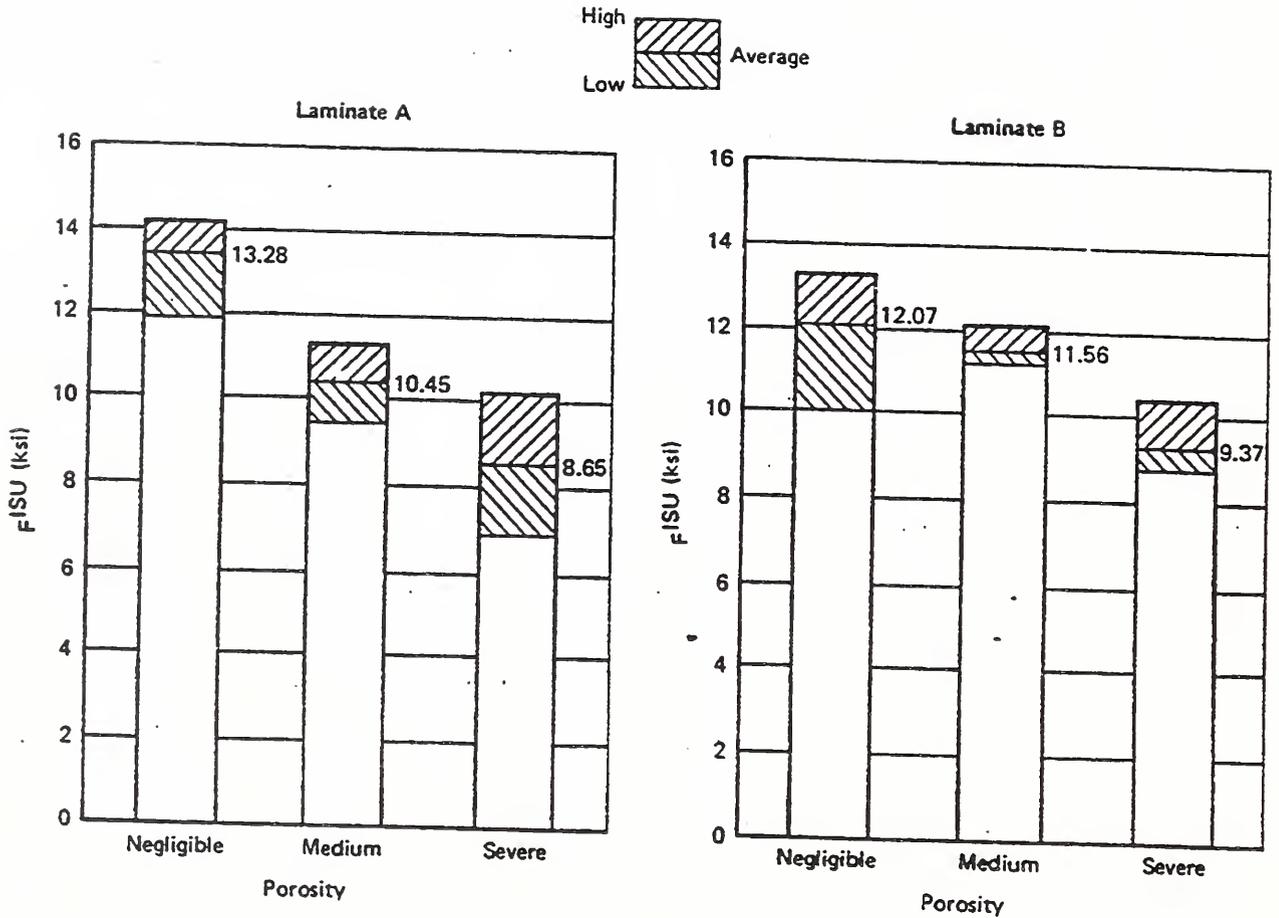
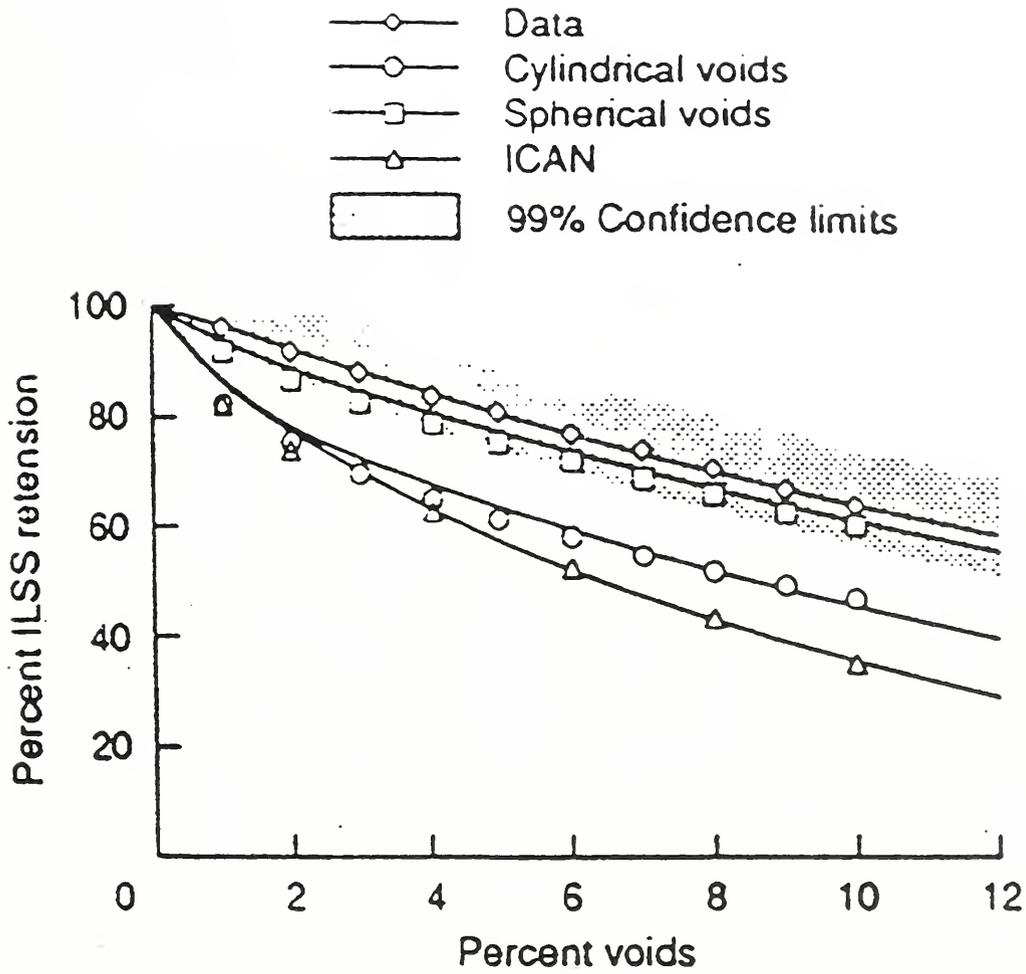


FIGURE 5-20  
EFFECTIVE OF POROSITY ON LAMINATE A AND B STATIC  
INTERLAMINAR SHEAR STRENGTH  
R.T./Dry

RE BOHLMANN, GD RENIERI, JJ KOZAREWICZ  
DEVELOPMENT OF ACCEPTANCE CRITERIA FOR GRAPHITE/EPOXY  
STRUCTURES

MARCH 1982

Figure 20



ILSS as a function of void content for 60 v/o AS/PMR-15 unidirectional com-

KJ BOWLES & S. FRIMpong  
VOID EFFECTS ON THE INTERLAMINAR SHEAR  
STRENGTH OF UNIDIRECTIONAL GRAPHITE FIBER  
REINFORCED COMPOSITES

J. Comp. Mats 26, 10, 1992  
1487-1509

TA418.9 C6J6

Figure 21

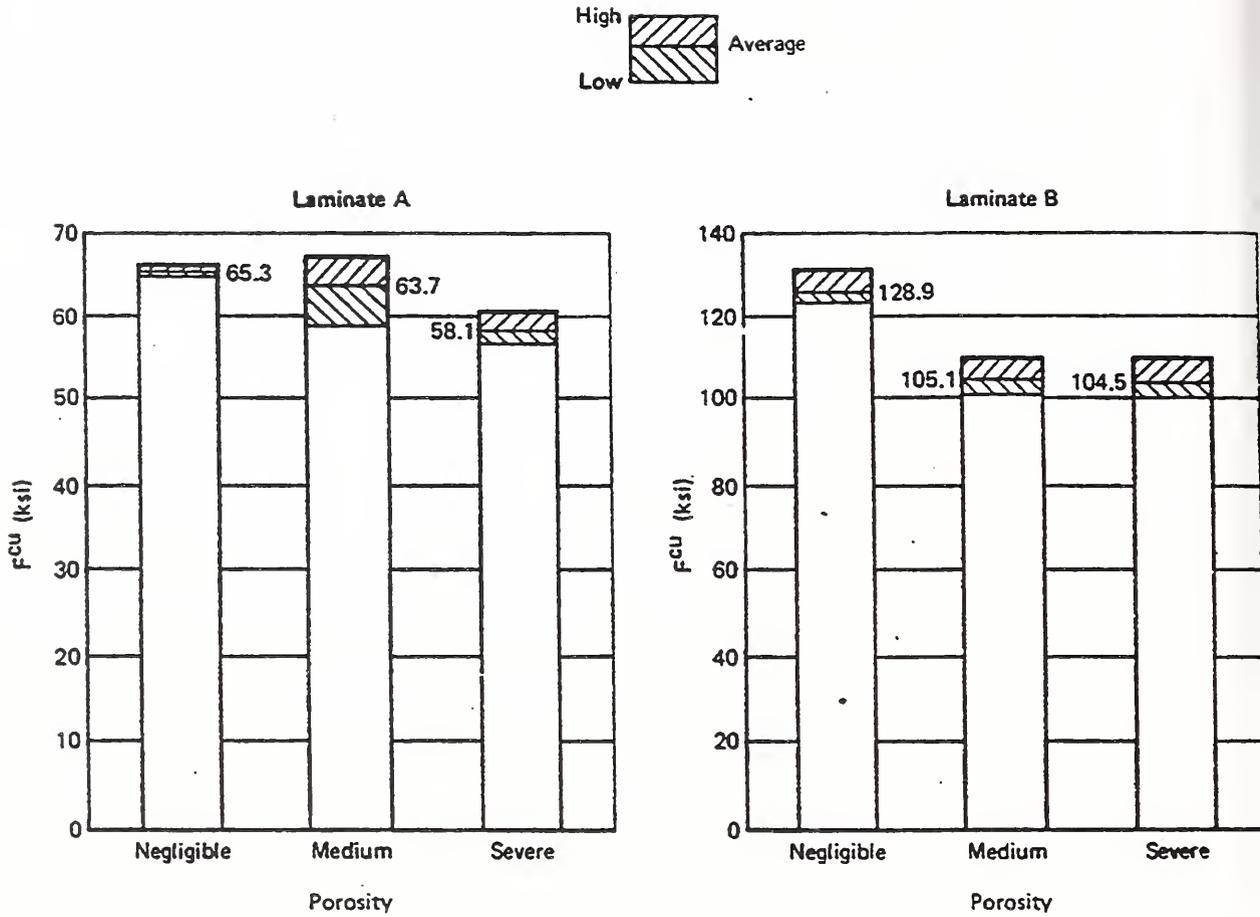


FIGURE 5-18  
EFFECT OF POROSITY ON LAMINATE A AND B STATIC  
COMPRESSIVE STRENGTH  
R.T./Dry

BOYLMAN, RENIER, KOSZARSKI  
DEVELOPMENT OF ACCEPTANCE CRITERIA  
FOR GRAPHITE/EPoxy STRUCTURES

# COMPRESSION FAILURE MODES

## LONGITUDINAL COMPRESSION

- FILAMENT MICROBUCKLING
- MATRIX YIELD FOLLOWED BY FILAMENT MICROBUCKLING
- FIBER / MATRIX DEBOND FOLLOWED BY FILAMENT MICROBUCKLING
- PANEL MICROBUCKLING
- SHEAR FAILURE
- DELAMINATION

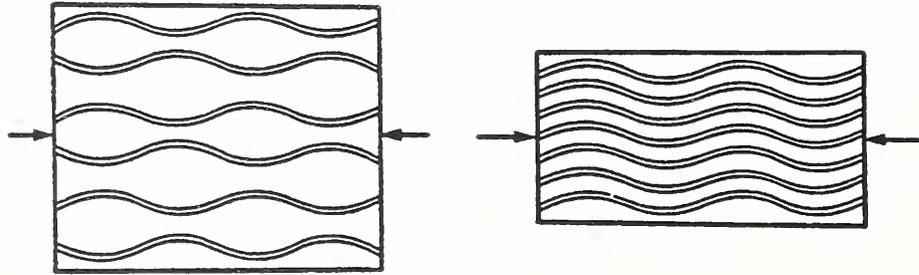
## TRANSVERSE COMPRESSION

- MATRIX COMPRESSIVE FAILURE
- MATRIX SHEAR FAILURE
- FIBER / MATRIX DEBOND
- FIBER CRUSHING

J. W. WINTNEY, C.E. BROWNING  
COMPRESSION OF COMPOSITE MATERIALS  
ADVANCED COMPOSITES SC 50

Figure 22

**MICROBUCKLING**

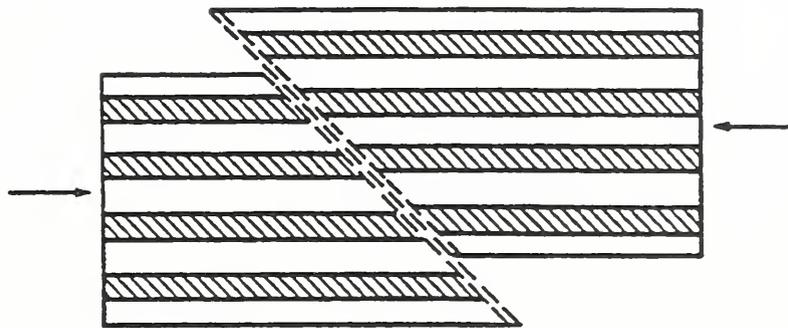


$$X_C = 2V_f \sqrt{\frac{V_f E_m E_{if}}{3(1-V_f)}} \quad (\text{ANTI-SYMMETRIC})$$

$$X_C = \frac{G^m}{(1-V_f)} \quad (\text{SYMMETRIC})$$

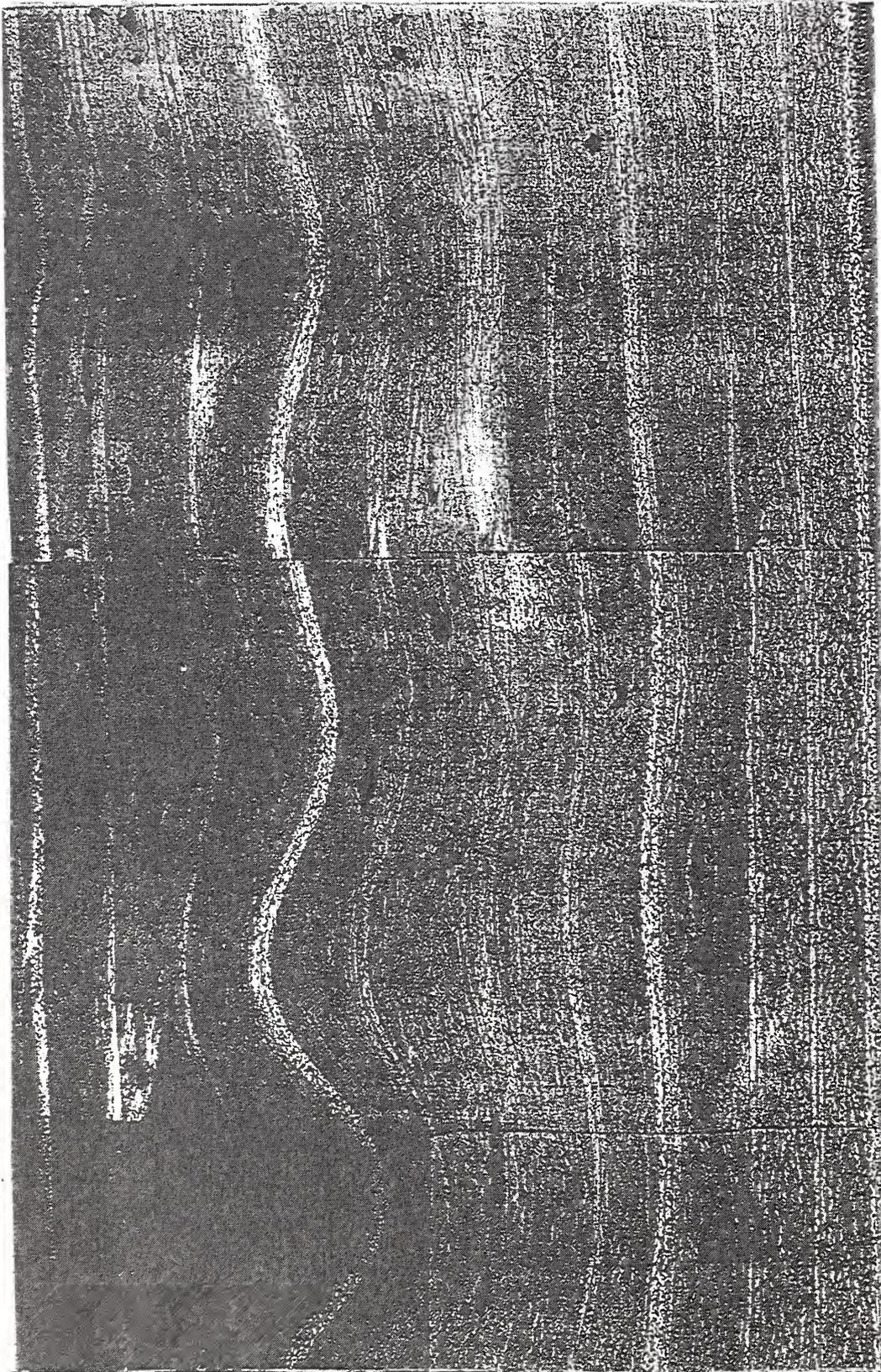
Figure 24

**LONGITUDINAL COMPRESSION**  
**SHEAR FAILURE MODE**



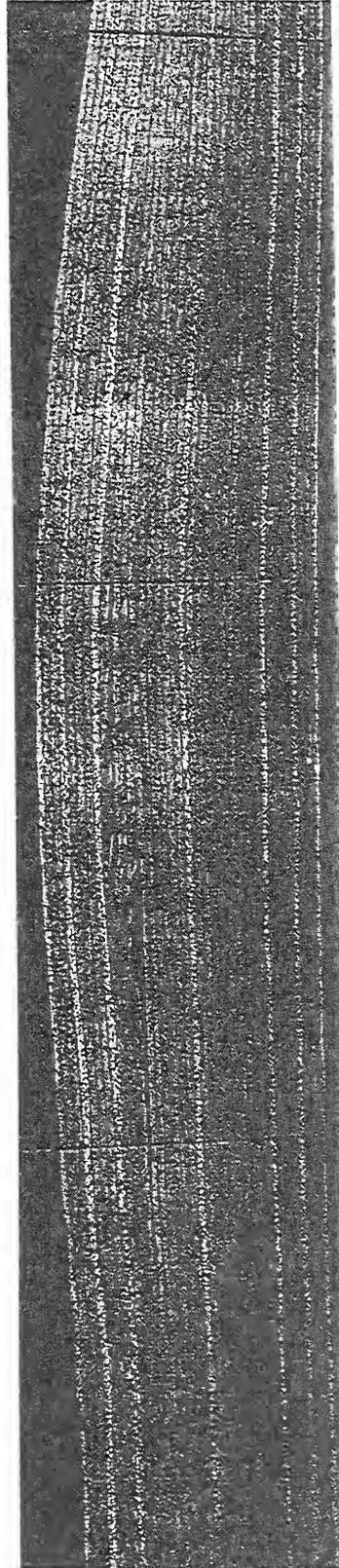
$$X_c = 2X_{gf} \left[ V_f + (1 - V_f) \frac{E_m}{E_{lf}} \right]$$

Figure 25



F-14 Over-Wing Fairing (Porosity)

Figure 26



**F-18 Vertical Stabilizer (Porosity)**

# DELAMINATION

- MATERIAL SPECIFIC EFFECT
  - TYPICAL ALLOWABLE LEVELS 1/2 IN. X 1/2 IN.
  - LARGER IMPACT ON COMPRESSION DOMINATED DESIGNS;  
RELATED TO BUCKLING STABILITY
  - CONTROL BY TOOLING CONCEPT, PROCESS RATES, QC
- EXAMPLE: F-14 OVERWING FAIRING  
SUBMARINE DRY DECK SHELTER



**NORTHROP GRUMMAN**

Figure 28

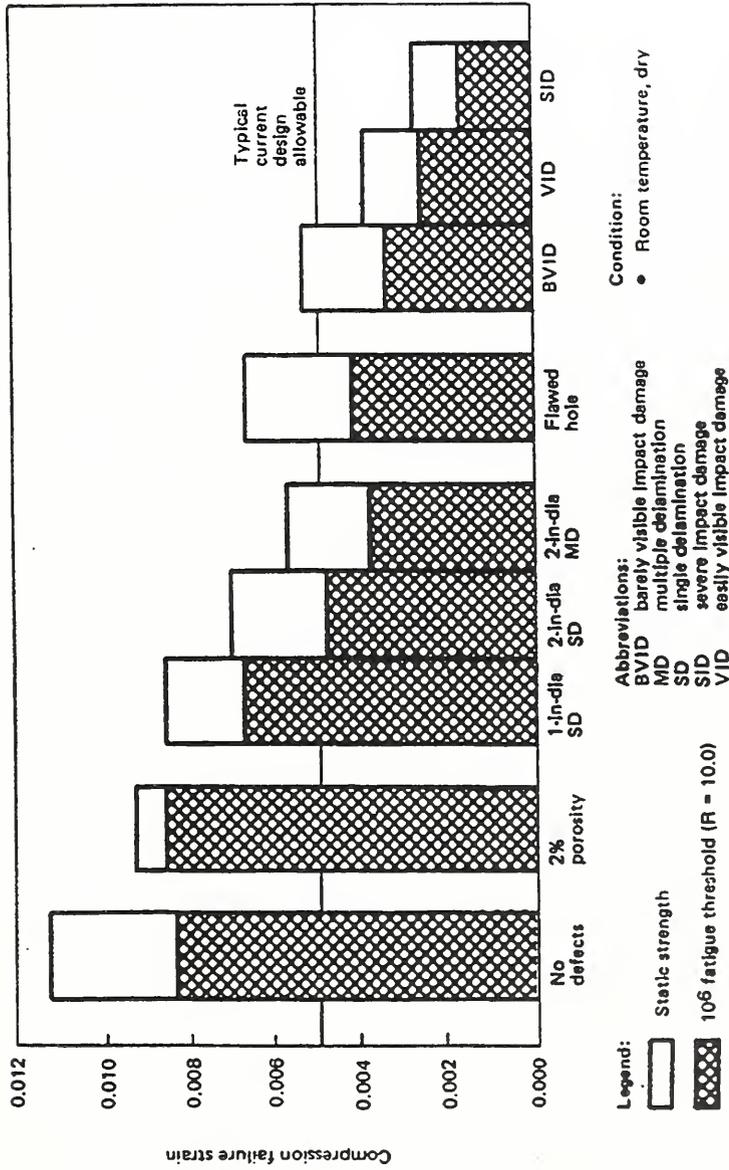


Figure 4-5. Defect/Damage Severity Comparison for Compression Loading

RE: NORTON, R S WHITEHEAD

DAMAGE TOLERANCE OF COMPOSITES  
VOL I DEVELOPMENT OF RESIDUALS  
AND COMPLIANCE DEMONSTRATION

JUNE 1988

# Figure 29

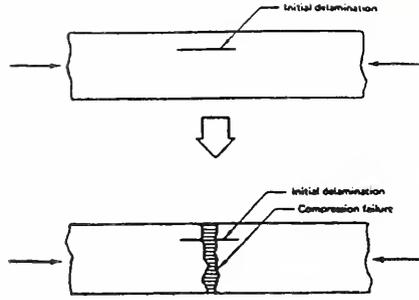


Figure 3-6. Gross Compression Failure (Failure Mode 1)

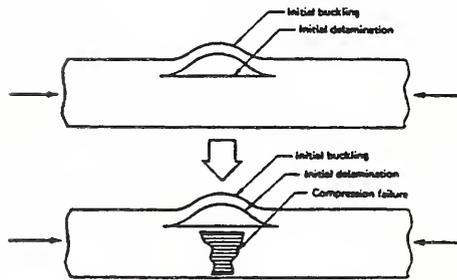


Figure 3-7. Initial Buckling of the Delamination Followed by Compression Failure (Failure Mode 2)



Figure 3-8. Global Buckling (Failure Mode 3)



Figure 3-9. Initial Buckling of the Delamination Followed by Global Buckling (Failure Mode 4)



Figure 3-10. Local Buckling (Failure Mode 5)

Figure 30

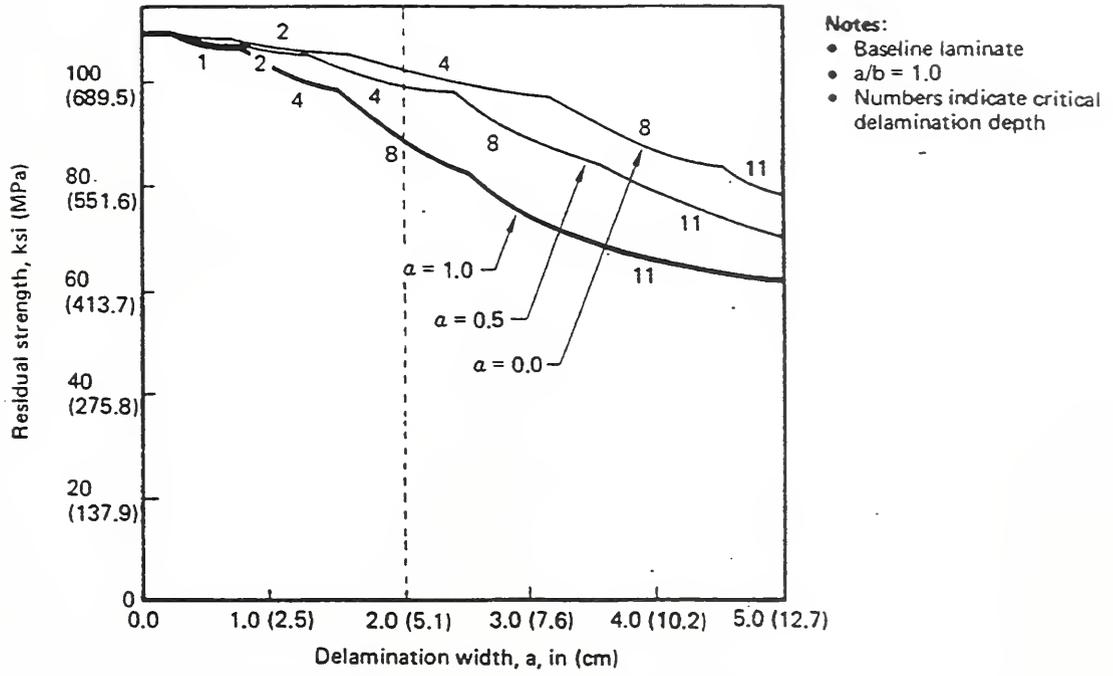
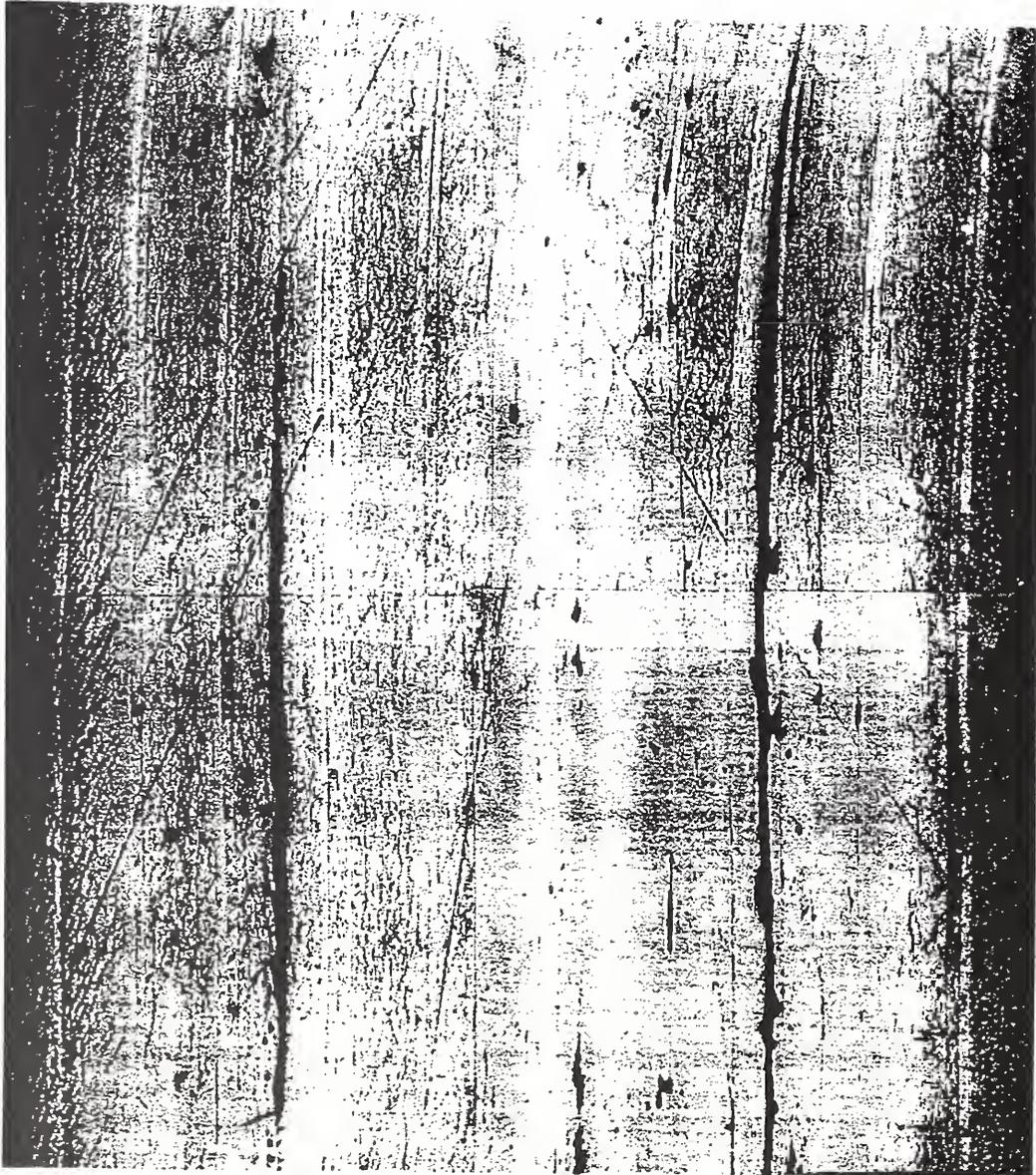


Figure 3-24. Influence of Boundary Fixity on Laminate Static Strength

Figure 31



F-14 Over-Wing Fairing (Delamination)

# CURE STATE

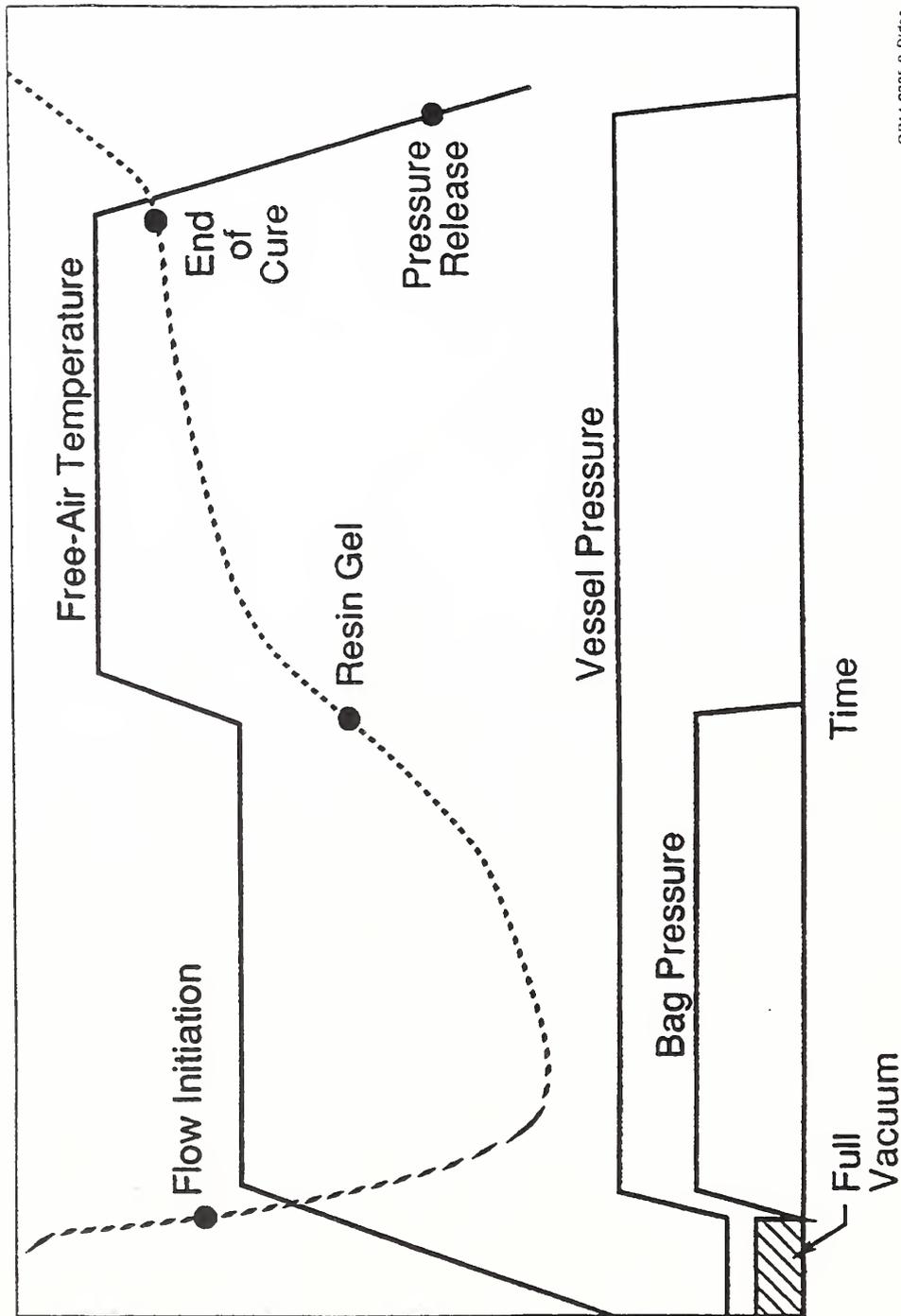
- CHEMICAL STATE CHANGES RELATED TO VISCOSITY, VOLATILE REMOVAL, EXOTHERM, THERMAL AND MECHANICAL PROPERTIES
  - AGE OF STARTING MATERIAL CAN BE A VARIABLE
  - TOOLING AND AUTOCLAVE LOADING CAUSE DEVIATIONS FROM PROCESS SPECIFICATION
  
- UNDERCURE REDUCTION IN HOT, WET PROPERTIES;  
OVERCURE REDUCTION IN TOUGHNESS
  
- CONTROL BY PROCESS DESIGN, SYSTEM THERMAL PROFILES,  
MATERIAL QC



**NORTHROP GRUMMAN**

Figure 33

# Controlling Events Epoxy and Bismaleimide Cures

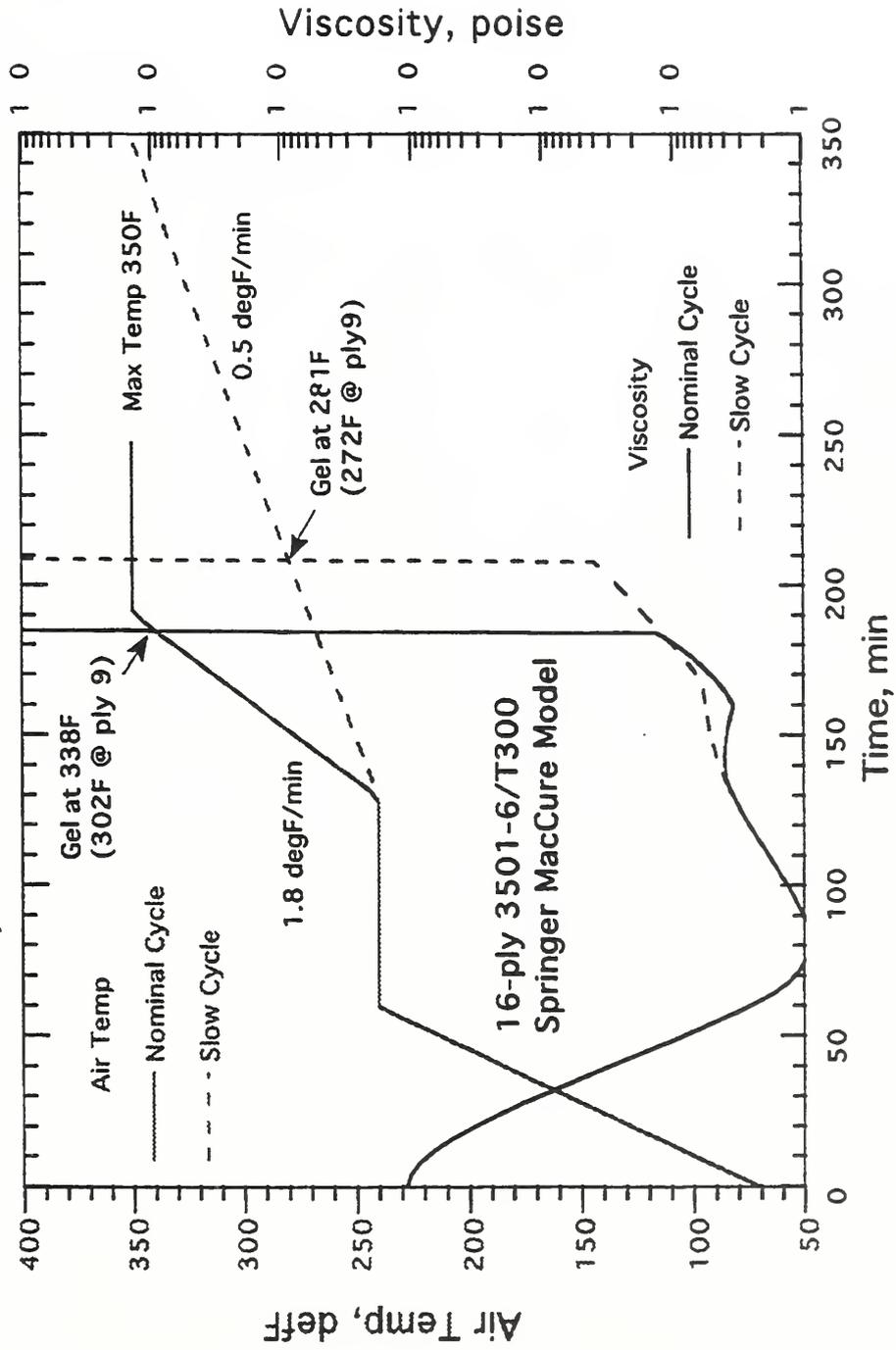


GP14.0335-9.D065

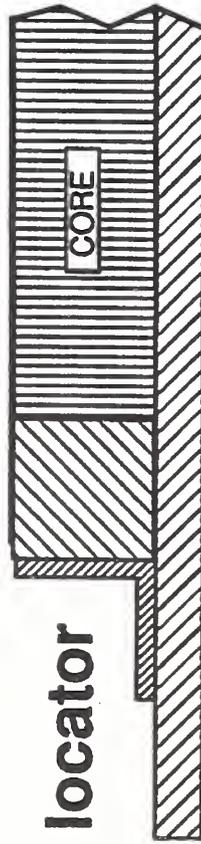
J A THOMAS  
ADVANCED COMPOSITE PROCESSING TECHNOLOGY  
DEVELOPMENT  
INDUSTRY REVIEW 25 JUNE 1992

Figure 34

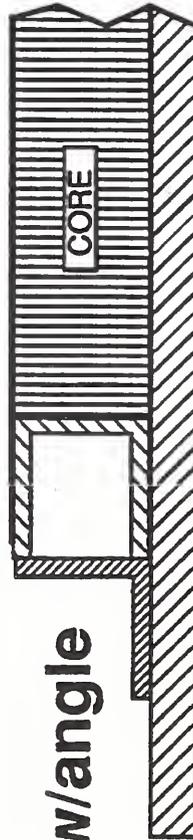
# Slow Heat-up Rate Reduces Matrix Gel Temperature



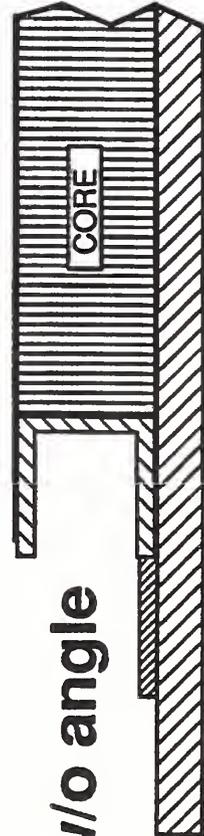
# 2D Thermal Analysis 777 Spar Locator, Configurations



**Solid locator**



**Hollow w/angle**



**Hollow w/o angle**

# Typical 2D Computational Result 777 Spar Locator

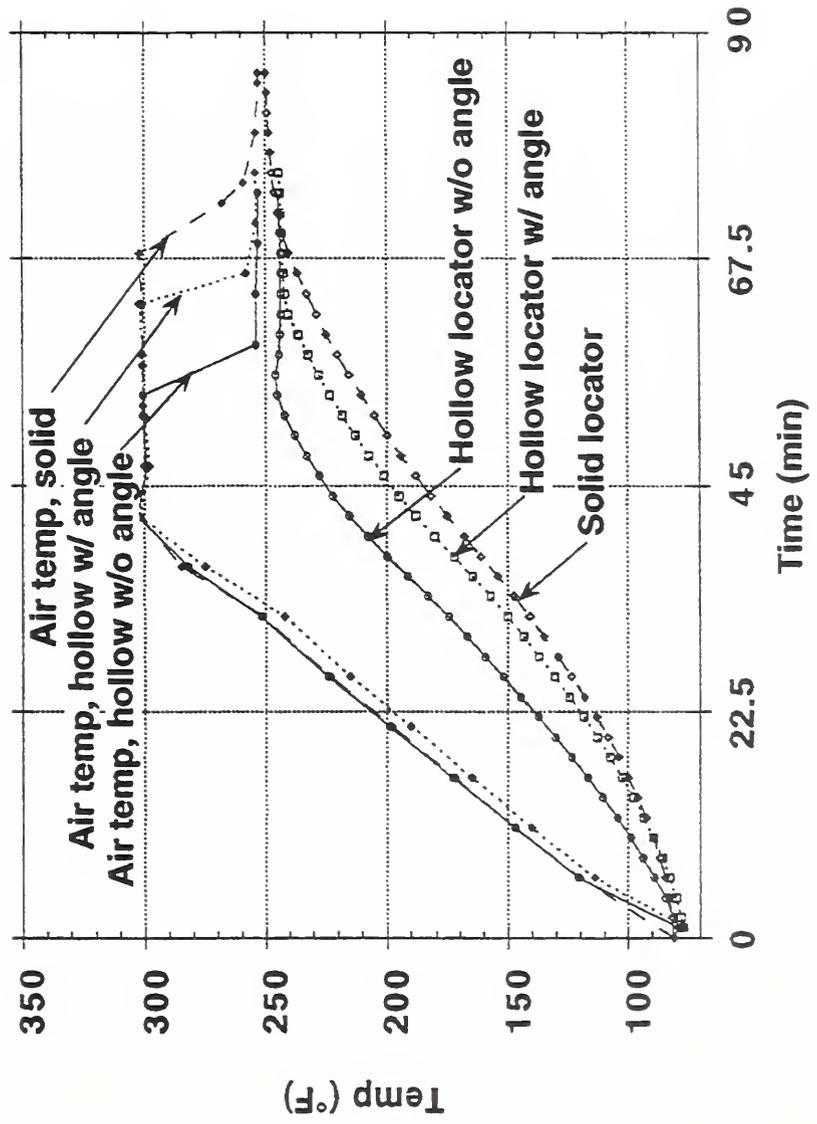


Figure 36

# Typical 2D Experimental Result

## 777 Spar Locator

Figure 37

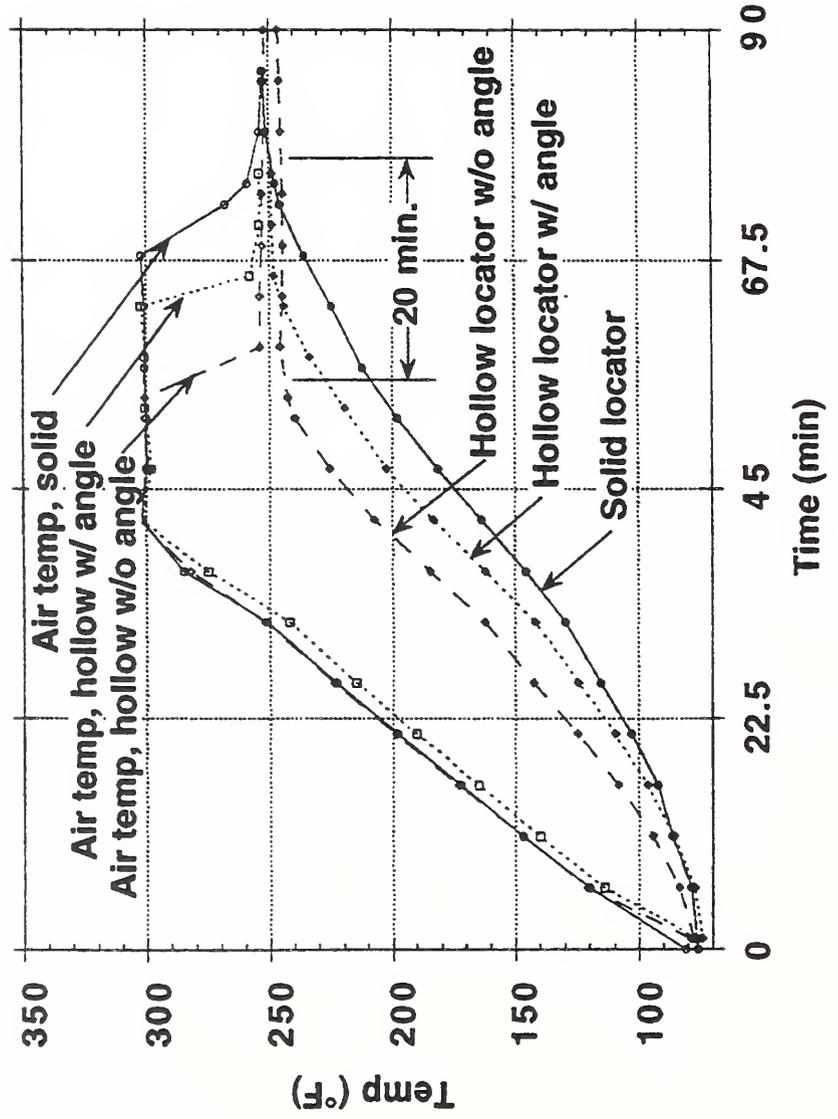


Figure 38

977-3/IM7 LAMINATE Tg DATA

<u>Cure</u>	<u>2 Hrs./180°C</u>	<u>6 Hrs./180°C</u>	<u>2 Hrs./180°C + 2 Hrs./200°C</u>
Dry Flex Tg, °C	NA	215	215
Dry DMA Tg, °C			
G'	164	186	180
G''	180	185, 231	193, 240
Tan Delta	177, 203	186, 239	195, 242
Wet DMA Tg, °C			
G'	141	165	145
G''	154, 177	185, 190	160, 198
Tan Delta	157, 188	186, 202	166, 198

Note:

Wet = 48 hr. water boil



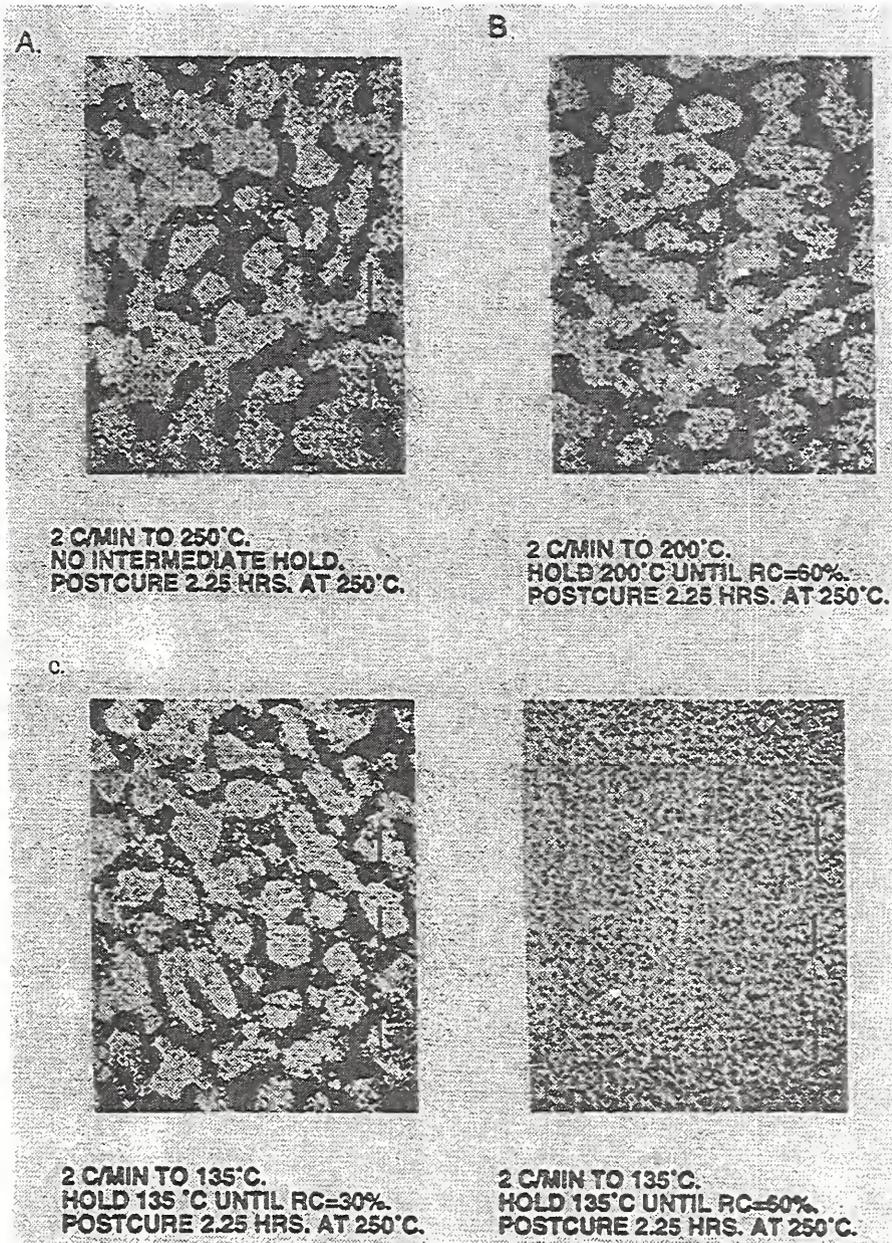
Cure Cycle Evaluation Summary of ICI FIBERITE's Hy-E 5377-3A (977-3/IM7 UDT)  
per F-22 Document CMD/91-033 and E-HH-012-92

	As Cured T <sub>g</sub> <sup>1</sup>	CAI Strength <sup>2</sup> ksi	EDS Onset Strength <sup>3</sup> ksi
Cure Cycle #1 Minimal Heat History Ramp Rate of 5.0-5.5°F/Minute Hold at 340-345°F for 330-340 minutes	Average 239.2 St Dev 2.49 Cov 1.0	Average 34.8 St Dev 0.8 CoV 2.4	Average 37.9 St Dev 0.9 CoV 2.3
Cure Cycle #2 Nominal Heat History Ramp Rate of 2.5-3.5°F/Minute Hold at 353-357°F for 355-365 minutes	Average 240.8 St Dev 1.64 Cov 0.7	Average 29.4 St Dev 1.1 CoV 3.9	Average 36.4 St Dev 1.3 CoV 3.6
Cure Cycle #3 Elevated Heat History Ramp Rate of 0.5-1.5°F/Minute Hold at 365-370°F for 380-390 minutes	Average 239.0 St Dev 1.44 Cov 0.6	Average 26.9 St Dev 0.9 CoV 3.2	Average 36.4 St Dev 1.2 CoV 3.3

- 1) As tested per 5PPTT01 Method 2.6 (TMA Penetration)
- 2) As tested per 5PPTT01 Method 4.30 with the following exceptions from F-22 document E-HH-077-92  
Panel Orientation [+45,90,-45,0]4s; Impact energy of 1500 in.-lb/in., based on nominal per-ply thickness; Impact Tup 0.5 inch diameter and Specimen Dimensions: Figure 3, prior to remachining after impact, and figure 4 (4.0 x 10.0 in.) remachined for compression testing.
- 3) As tested per 5PPTT01 Method 4.27.
- 4) Five specimens were used for each data set.

Figure 39

Figure 40



J. BREWEN ET AL.  
PRODUCTION OF CONTROLLED NETWORKS AND  
MORPHOLOGIES IN TOUGHENED THERMOSETTING RESIN  
USING REAL TIME IN-SITU CURE MONITORING  
41ST SAGAMORE CONFERENCE PROCEEDINGS  
US ARMY RESEARCH LAB  
AUGUST 1994

## **DIMENSIONAL TOLERANCE**

- **STARTING MATERIAL VARIANCES, TOOLING CONCEPTS, PART DESIGN PRODUCE COMPONENTS THAT HAVE POOR CORRELATION TO DESIGN**
  - **RADIUS PROBLEMS - MALE TOOL - THINNING**
  - **FEMALE TOOL - THICKENING**
  - **THICKNESS CONTROL - ASSEMBLY INDUCED DELAMINATIONS**
  - **GEOMETRY CONTROL - SPRING IN/OUT - RESIDUAL STRESS**
- **CONTROL BY TOOL DESIGN, COMPONENT DESIGN, PROCESS**



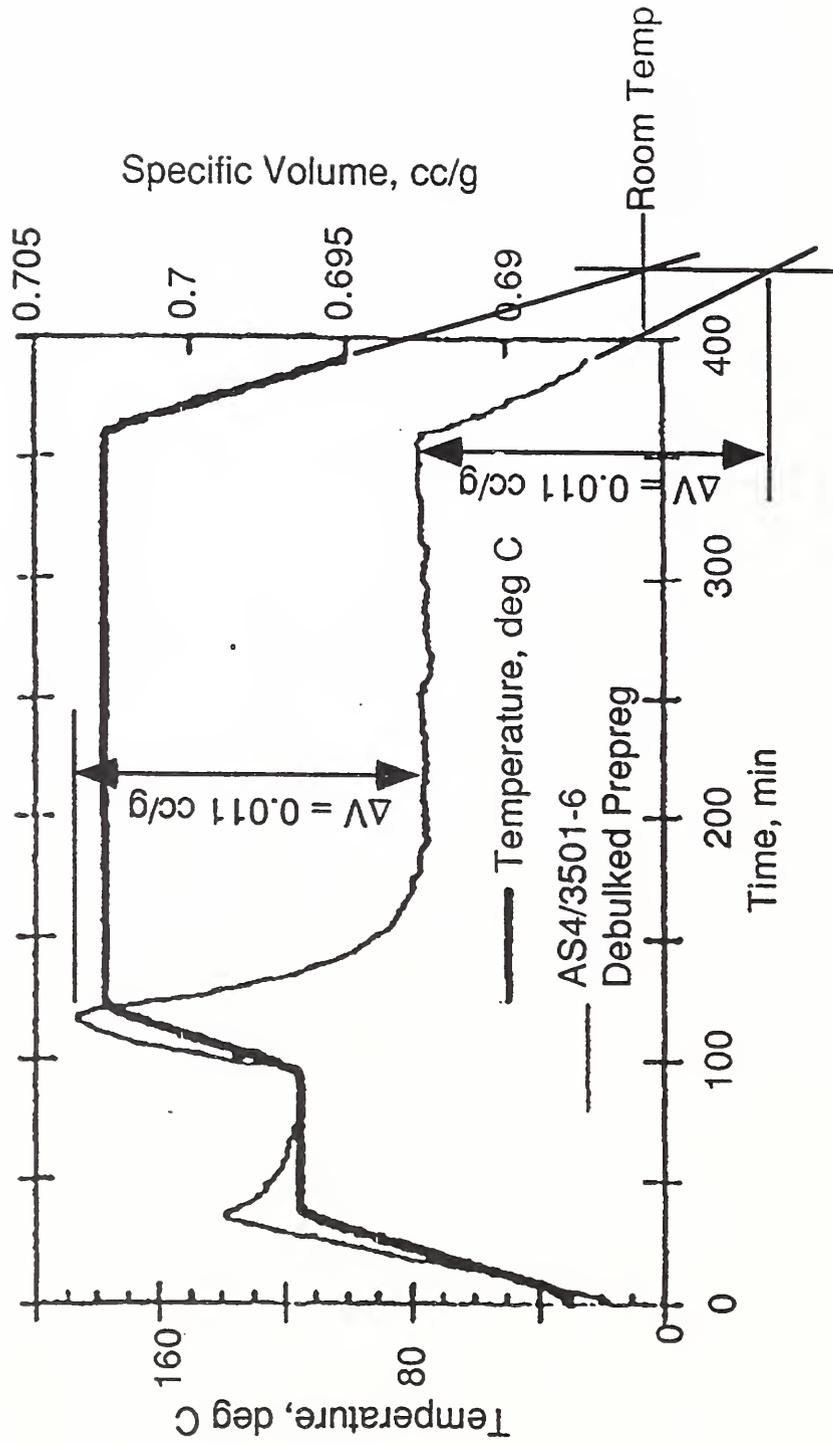
# **Factors Contributing to Spring-In**

- **Anisotropic thermal shrinkage during cool down**
  - composite lay-up
  - number of plies
  - radius of curvature
  - initial angle
- **Process-induced residual stresses**
  - cure cycle dependent
  - chemical shrinkage during cure
  - time history of volume change vs. viscosity change
  - fiber volume fraction gradient through the thickness

Figure 42

Figure 43

### Effect of Cure Cycle on the Volume Changes in Debulked AS4/3501-6 Prepreg



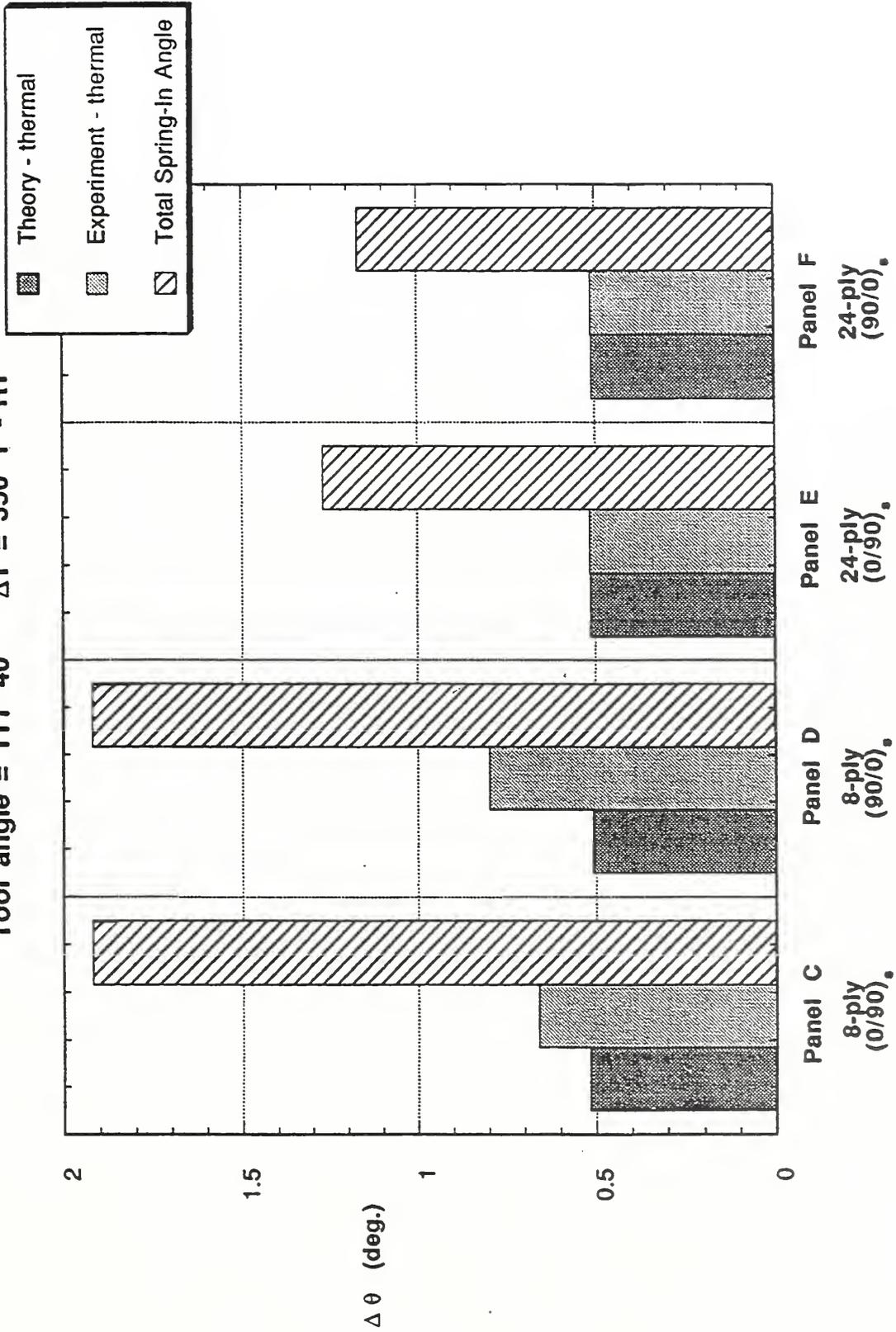
J. D. Russell, 1993, SAMPE Quarterly, 24-2, 28-33

TL958.S 582.5

Figure 44

# Spring-In Angle Comparison

Tool angle =  $111^{\circ} 40'$   $\Delta T = 350^{\circ} F - RT$



# TYPICAL SKIN DELAMINATION

Outboard Wing

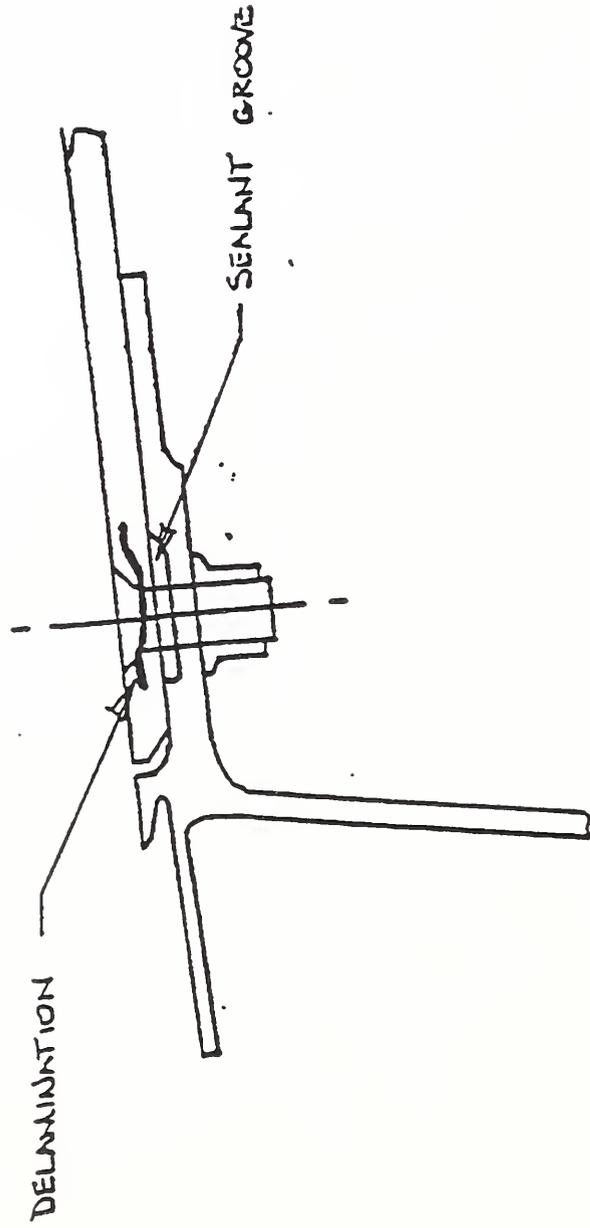


Figure 46

# TYPICAL SPAR DELAMINATION

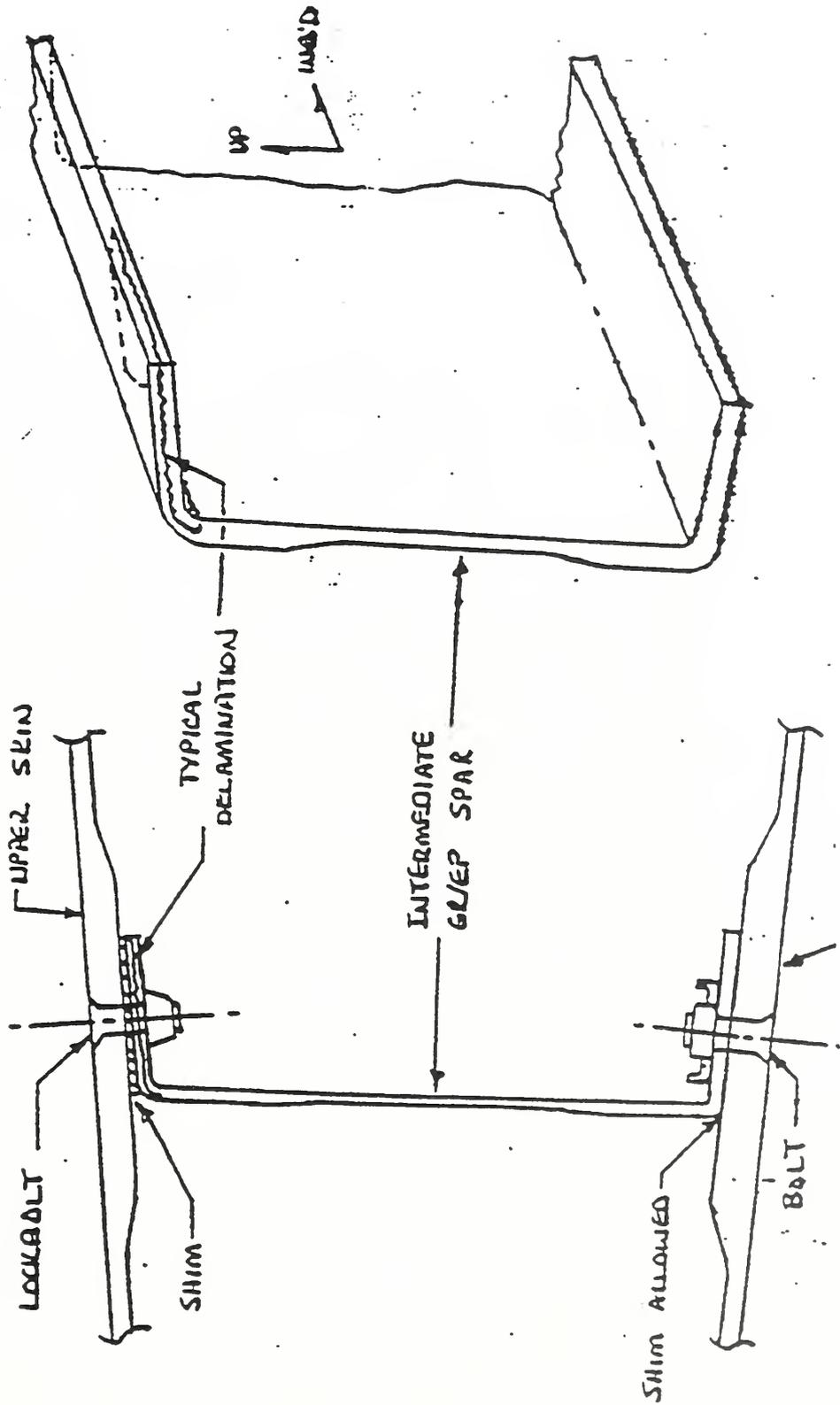
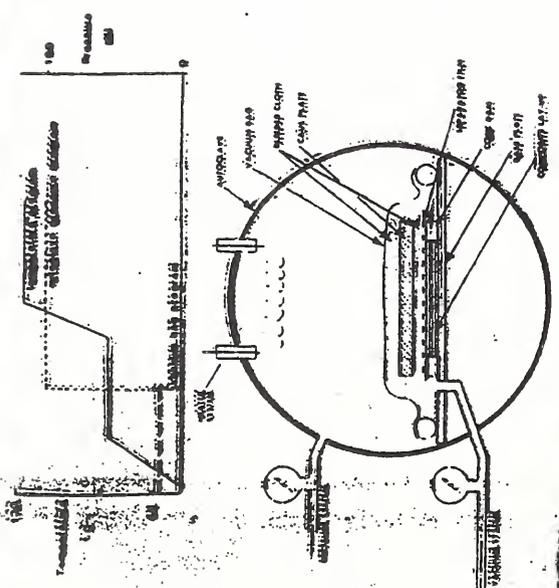


Figure 47



**APPROACH:**

- PROCESSING MODEL DEVELOPED INTO CURE OPTIMIZATION PROGRAM INSTALLED ON PRESS
- A THICKNESS GAUGE FOR USE IN AUTOCLAVE DEVELOPED

**OBJECTIVE:**

- MODEL RHEOLOGY OF RESIN FLOW DURING COMPOSITE PROCESSING
- PREDICT CONDITIONS FOR VOID FORMATION
- CONTROL CURE CYCLE TO PREVENT VOID FORMATION

**TRANSITIONS:**

- AIRFRAME MANUFACTURERS (F/A-18, AV-8B, V-22)
- MODEL USED FOR CURE CYCLE DEVELOPMENT FOR 6.3 PROGRAM ON RESIN CHARACTERIZATION
- MODEL USED FOR ANALYSIS OF CUMULATIVE EFFECTS OF PROCESS VARIABLES IN V-22 PROGRAM

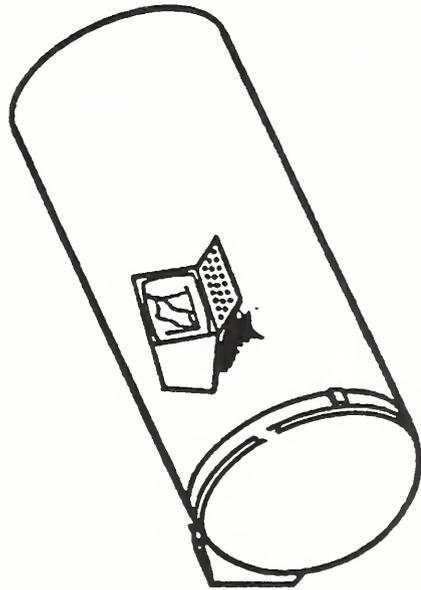
**PERFORMERS:**

- DR. JOHN WILLIAMS, NAVAIRDEVGEN

N1808-0A-88-02079

Figure 48

# Background



## "Advanced Composite Processing Technology Development"

Program Manager: J.E.Kurz, MCAIR  
 Principle Investigator: F.R. Muncaster, MCAIR  
 Program Monitor: J. Russell, WRDC/MLBC

## "Qualitative Process Automation for Autoclave Curing of Composites"

S.R. LeClair, WRDC/MLTC

## "Computer-Aided Curing of Composites"

F. Abrams, WRDC/MLBC

*ATMANS Composite Processing Technology  
 "Advanced Composite Development"  
 Industry Review 25 June 1992*

## Preliminary Sensor Systems

Sensor System	Critical Composite Property	Sensor Expert	Company
Dielectric	Resin State	Dr. David Day	Micromet Instruments
Eddy Current	Thickness	Dr. Masood Zaidi	Douglas Aircraft
Fluorescence Optrode	Resin State	Dr. Ram Levy	McDonnell Douglas Research Labs
Fiber Optics	Voids Resin State	Alan Markus	Douglas Aircraft
Ultrasonics	Thickness Voids	Mark Reighard	McDonnell Aircraft

# Dielectric

<b>Function:</b>	Resin State Sensor
<b>Critical Data:</b>	Flow Initiation Point, Flow State, Gel Point, End of Cure Point
<b>Equipment:</b>	Tool-Mounted Ceramic Sensors (Reusable) Durable Autoclave Cabling Three Prong Thermocouple-Like Connectors Eight Channel Multiplexer and Interface Boxes System II Microdielectrometer
<b>Advantages:</b>	Easy Hook-Up Does a Good Job With Flow Initiation and Flow State Determinations Good Vendor Support and Equipment Availability Eight Channel Capability Very Durable
<b>Disadvantages:</b>	Not Sensitive Enough to Determine End of Cure Point Filter Mark-Off On Part Surface

Figure 50

# Fiber Optics

<b>Function:</b>	Void and Resin State Sensors
<b>Critical Data:</b>	Part Temperature, Resin Pressure
<b>Equipment:</b>	Fiber Optic Temperature Sensor Fiber Optic Pressure Sensor Fiber Optic Extension Cables Fiber Optic Multisensor System (4 Channel)
<b>Advantages:</b>	No Limitation On Cable Length Refractive Index Cure Sensor (In Development) May Be Used In the Same Unit
<b>Disadvantages:</b>	Extension Cables Must Be more Durable

# Real-Time Autoclave Control Benefits

## Near Term: Advanced Manufacturing Technology

- Process Development
- Part/Tool Design

## Long Term: Composite Production

- Improved Part Quality
- Increased Part to Part Consistency (Resin Content, Thickness, Mechanical Properties)
- Reduced Autoclave Time

Figure 52

# SENSOR SYSTEM/CONTROL SYSTEM NEEDS/OPPORTUNITIES

- PRE-PROCESS
  - LAY UP AREA
  - NON-CONTACT INSPECTION TOOL FOR ORIENTATION, LOCATIONS, DEFECTS
  - PRE-PREG INSPECTION TOOL - DEGREE OF CURE, DEGREE OF WETTING, RESIN CONTENT
  - TEXTILE PREFORM QUALITY SENSOR
- PROCESS - SIMPLE, LOW COST, REUSABLE
  - POROSITY LEVEL SENSOR
  - RESIDUAL STRESS SENSOR
  - CURE STATE SENSOR

Figure 53



# A Manufacturing View of Adaptive Process Control

Foster Lamm

United Technologies Research Center

(860) 727-7382

Figure 54



Notes.  
from a manufacturing  
View any new Technology  
MUST impact The financial  
Side of The business. IT  
is not sufficient to be  
"Good Technology"

Figure 55

ALLEN BRADLEY  
COMPANY

ERIE PRESS  
SYSTEMS

UNITED  
TECHNOLOGIES



NATIONAL CENTER FOR  
MANUFACTURING SCIENCES

# ADAPTIVE PROCESS CONTROL FOR COMPRESSION MOLDED COMPOSITES

Program Team put  
Together To have The  
deliverable be a supportable  
Adaptive process Control System



James Smith ← Principal Investigator

Pratt & Whitney

Composites Manufacturing Technology

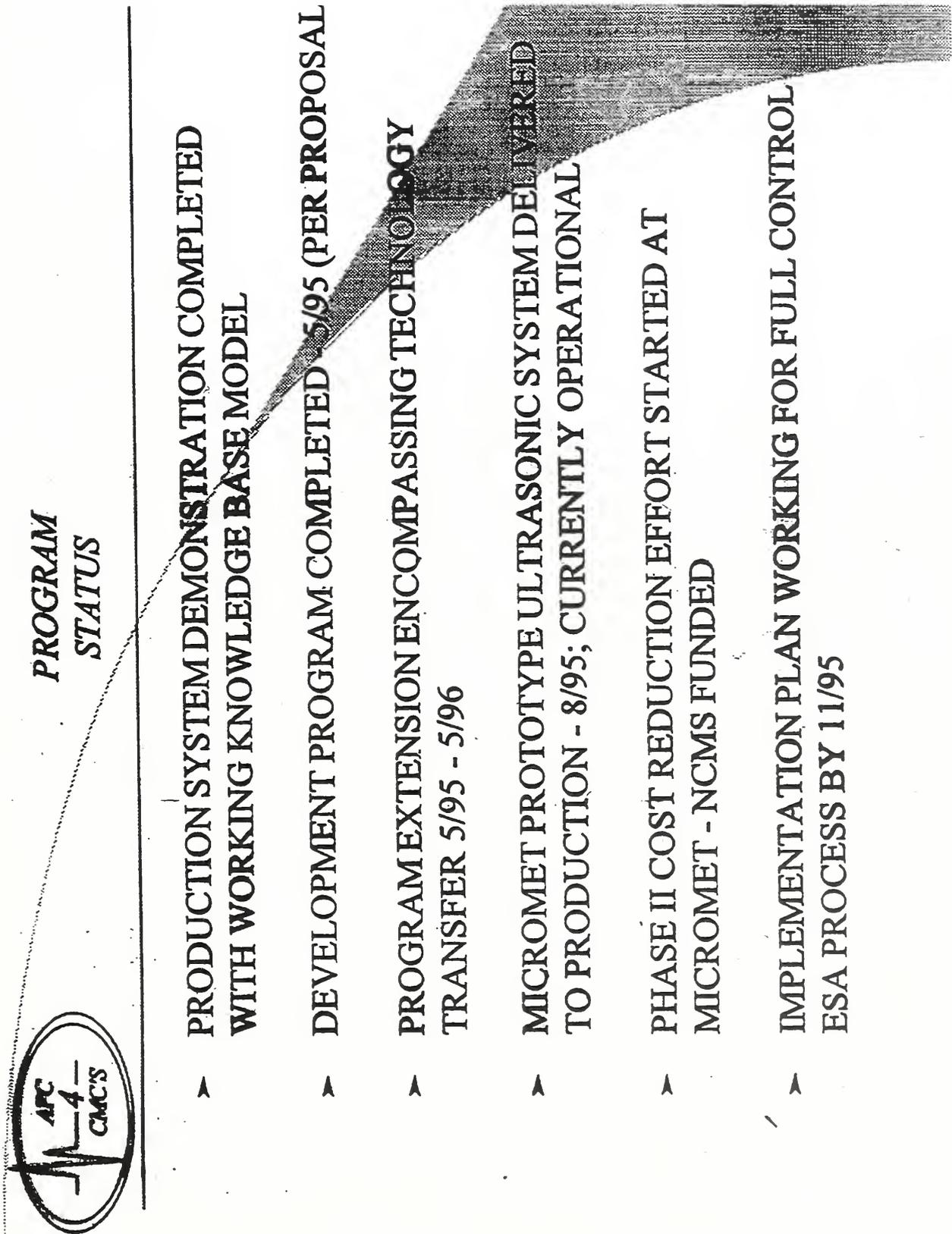


## ADAPTIVE PROCESS CONTROL DEVELOPMENT EXTENSIONS

- ULTRASONIC SENSOR TO PROCESS DEVELOPMENT
  - THEORETICAL CORRELATION OF ULTRASOUND AND VISCOSITY
  - PRACTICAL USES OF ULTRASOUND ON OTHER PROCESSES AND MATERIALS(DETERMINE RULES)
  - DETERMINE REACTION KINETIC RELATION TO ULTRASOUND
  - FURTHER DEVELOPMENT OF PREDICTIVE MAINTENANCE FOR ULTRASONIC SENSOR
  
- RULES BASED APPROACH TO ADAPTIVE PROCESS CONTROL
  - PART DESIGN: *CAUSE* - METALS THINKING; *EFFECT* - PROCESS NOT OPTIMIZED
  - PREFORM: *CAUSE* - AERIAL WEIGHT, RESIN VISCOSITY, LAY-UP, DEBULK, THICKNESS; *EFFECT* - MIGRATION, FIBER VOLUME PERMEABILITY, EXOTHERM, MECHANICALS, QUALITY
  - TOOLING: *CAUSE* - HEAT DISTRIBUTION, CAVITY DESIGN; *EFFECT* - VISCOSITY, FLOW VARIATION
  - EQUIPMENT : *CAUSE* - CALIBRATION; *EFFECT* - BLACK MAGIC PROCESSING

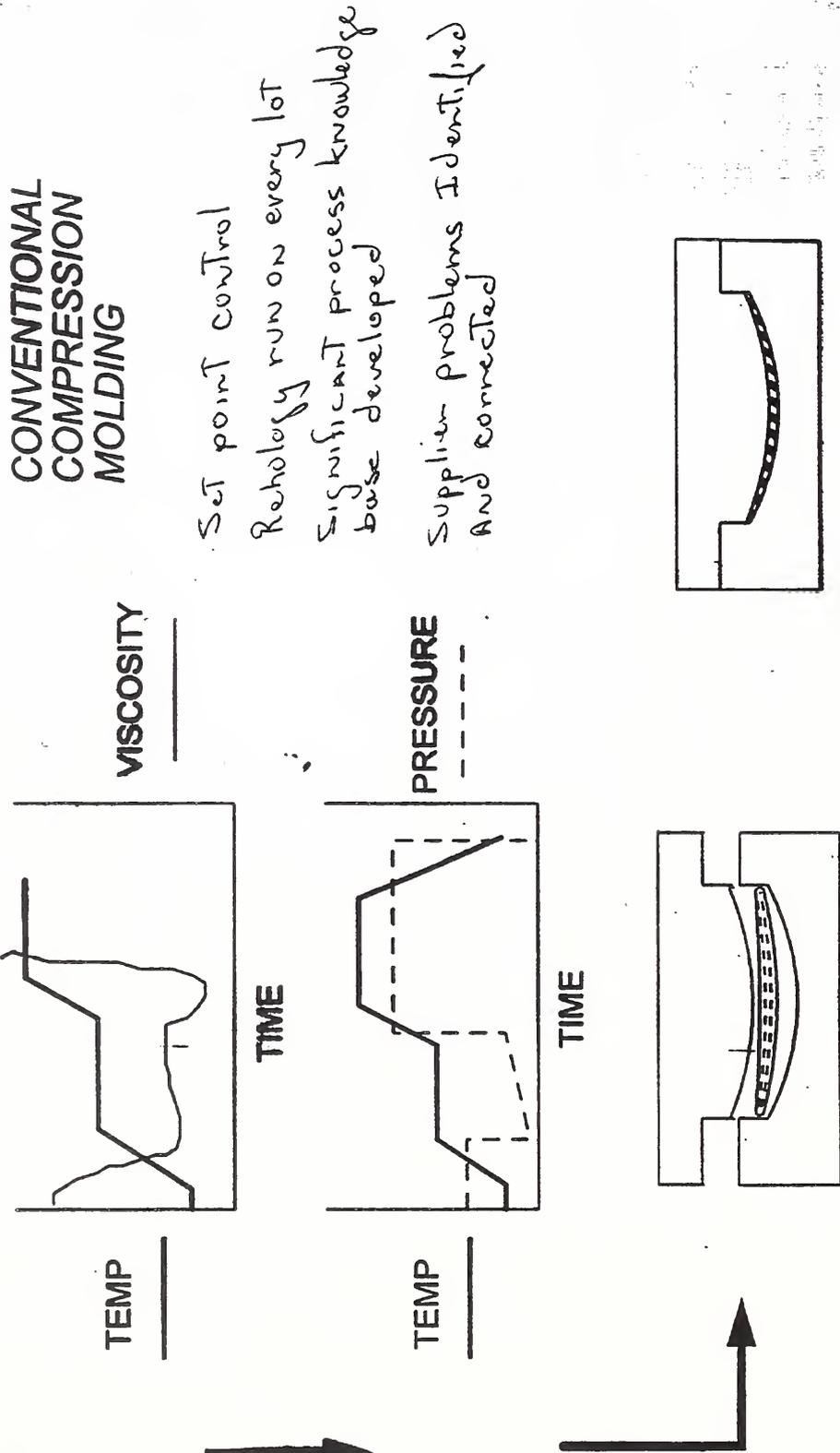
Figure 56

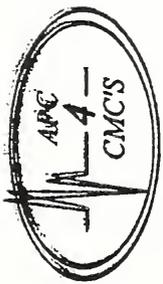
Figure 57





DEFINITION OF NEED

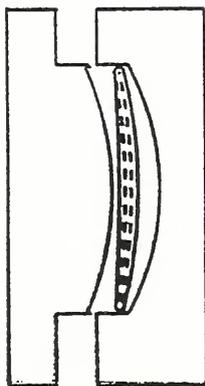




# PROPOSED TECHNICAL SOLUTION

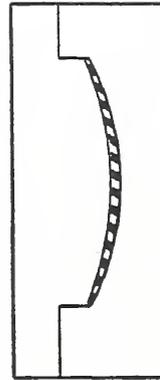
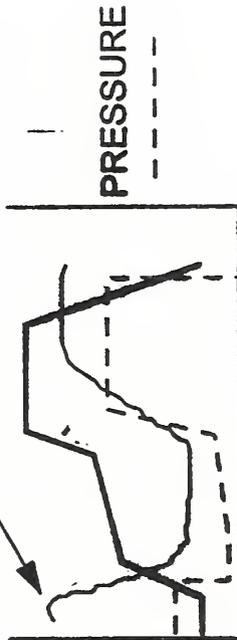
## ADAPTIVE COMPRESSION MOLDING

Control based on current condition of curing resin (viscosity)



TEMP

VISCOSITY (SOUND SPEED)



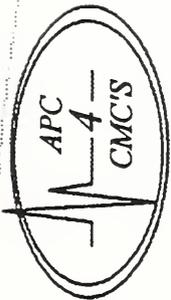


1. TECHNICAL CASE CRITERIA

# NEEDS DEFINITION AND PROPOSED TECHNICAL SOLUTION

- ▶ MATERIAL VARIATION PREVENTS PROCESS ALIGNMENT
  - INDIRECT MANUFACTURING COSTS INFLATE OVERHEAD
  - EXCESS DEVELOPMENT TIME IMPEDES MARKET PENETRATION
- ▶ PROCESS CERTIFICATION UN-ACHIEVABLE THROUGH CURRENT CONTROL
  - CRITICAL VISCOSITY WINDOW CURRENTLY ESTIMATED
  - REOLOGY MEASUREMENTS MAY BE BIAS DETERMINATIONS OF VISCOSITY
  - THERMAL AND VISCOSITY RESPONSE INTERDEPENDENT
  - RESIN THRESHOLD PRESSURE DEPENDENT ON VISCOSITY
- ▶ ADAPTIVE CONTROL FROM SENSOR RESPONSE OF MATERIAL
  - SOUND SPEED IS A MORE DIRECT MEASURE OF VISCOSITY & OTHER NON-LINEAR PROCESS PARAMETERS THAN TIME & TEMP
  - RELIABLE COST EFFECTIVE SENSOR DESIGN
  - USER-FRIENDLY CONTROL SCHEME FOR PROCESS ENGINEER
  - MINIMAL YET PREDICTIVE MAINTENANCE FOR OVERALL SYSTEM

Figure 60



## PROGRAM BENEFITS

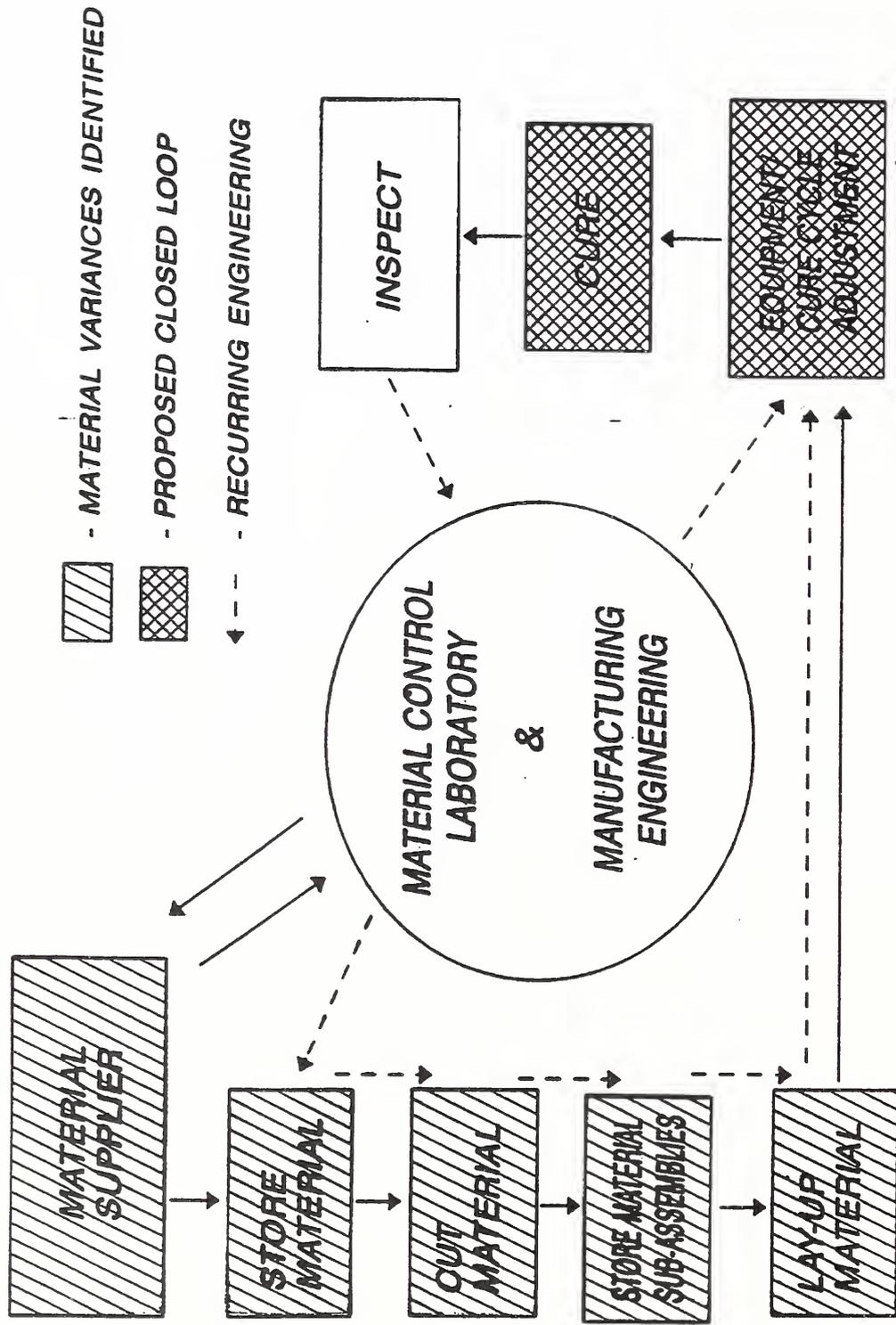
- CONTROL SYSTEM ADDRESSES CURRENT PRODUCTION NEEDS
  - GREATER CONTROL OF LAMINATE CONSOLIDATION
  - PROBLEM IDENTIFICATION THROUGH MEASUREMENT
  
- ADAPTIVE CONTROL
  - LOWER MANUFACTURING COSTS ( [REDACTED] ) THROUGH HIGHER PRODUCT YIELDS
  - LOWER CAPITAL INVESTMENT (\$1000/TON) THROUGH SMARTER CONTROL
  
- EQUIPMENT DEVELOPMENT EXCEEDS PROGRAM GOALS
  - INTEGRAL SENSOR & CONTROL SYSTEM, SIZE REDUCED 95% SINCE START
  - DEPLOYMENT COSTS WITHIN TARGET
  
- PROGRAM LEVERAGE > [REDACTED]

Figure 61



1. TECHNICAL CRITERIA

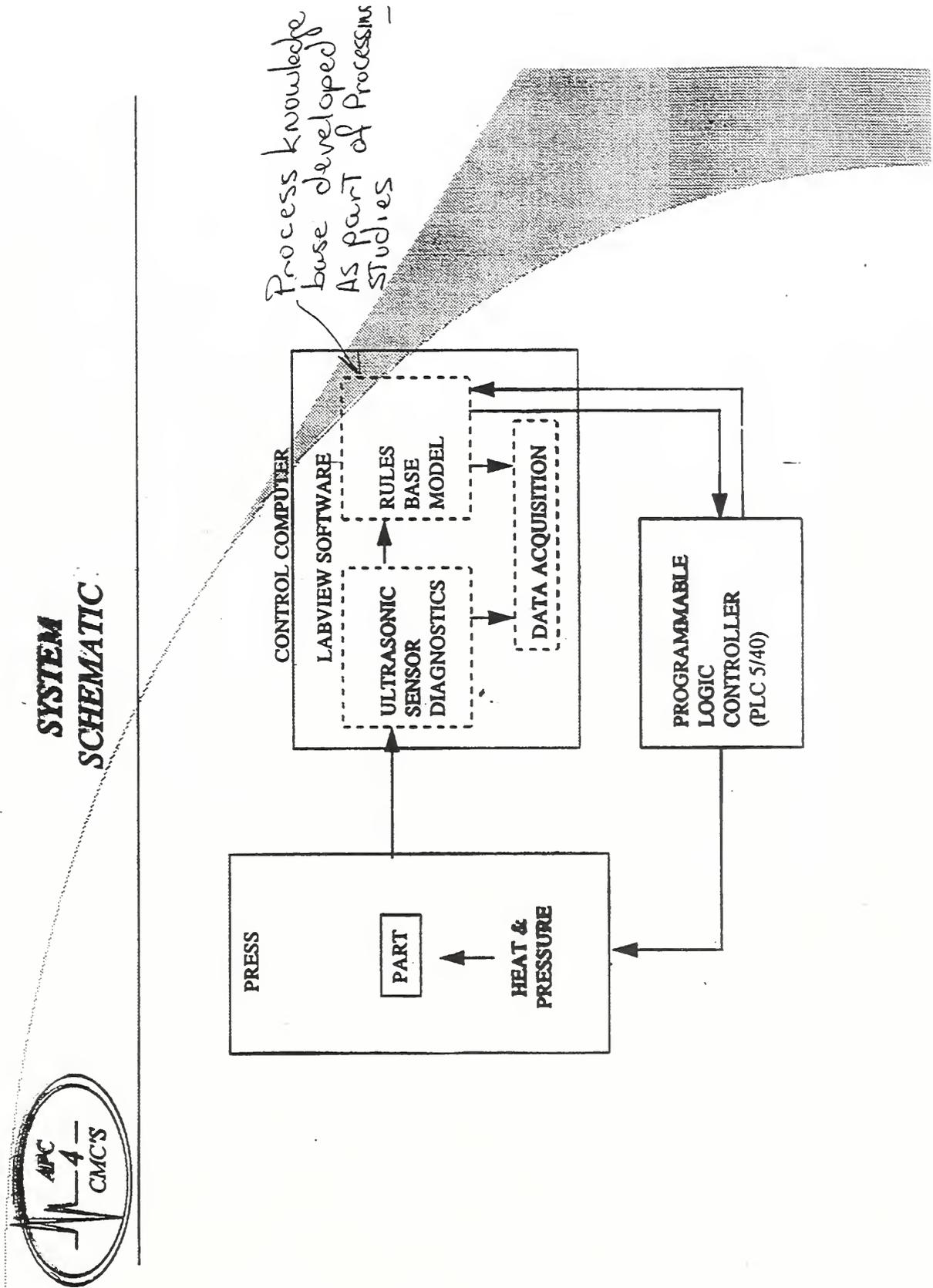
# NEEDS DEFINITION AND PROPOSED TECHNICAL SOLUTION

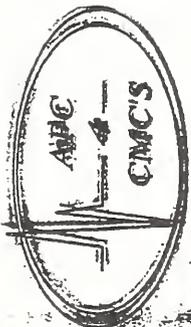


Lower Cost by Eliminating the dotted lines (recurring Engineering)

Figure 62

Figure 63





# SENSOR SCHEMATIC

## PHASE 1-2-3

Ultrasonic Sensor Integrated into FEGV Tooling

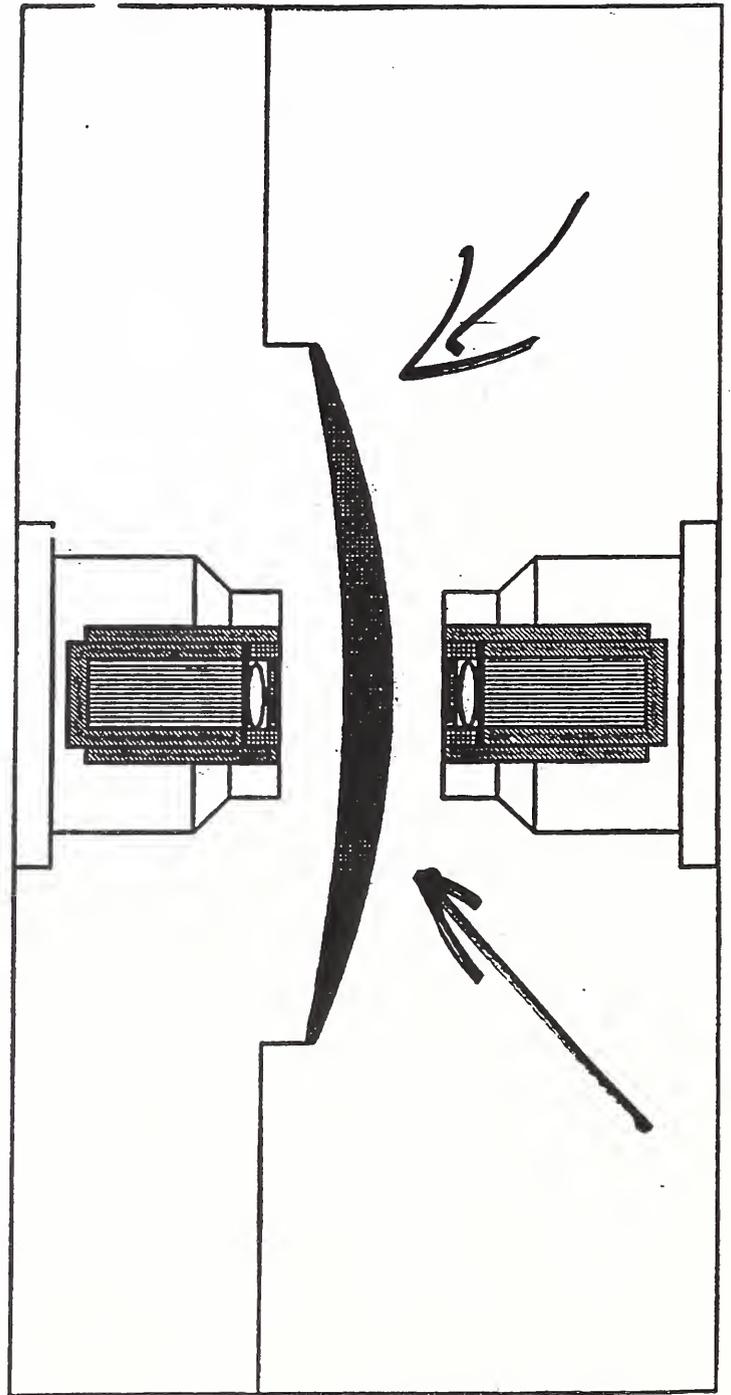
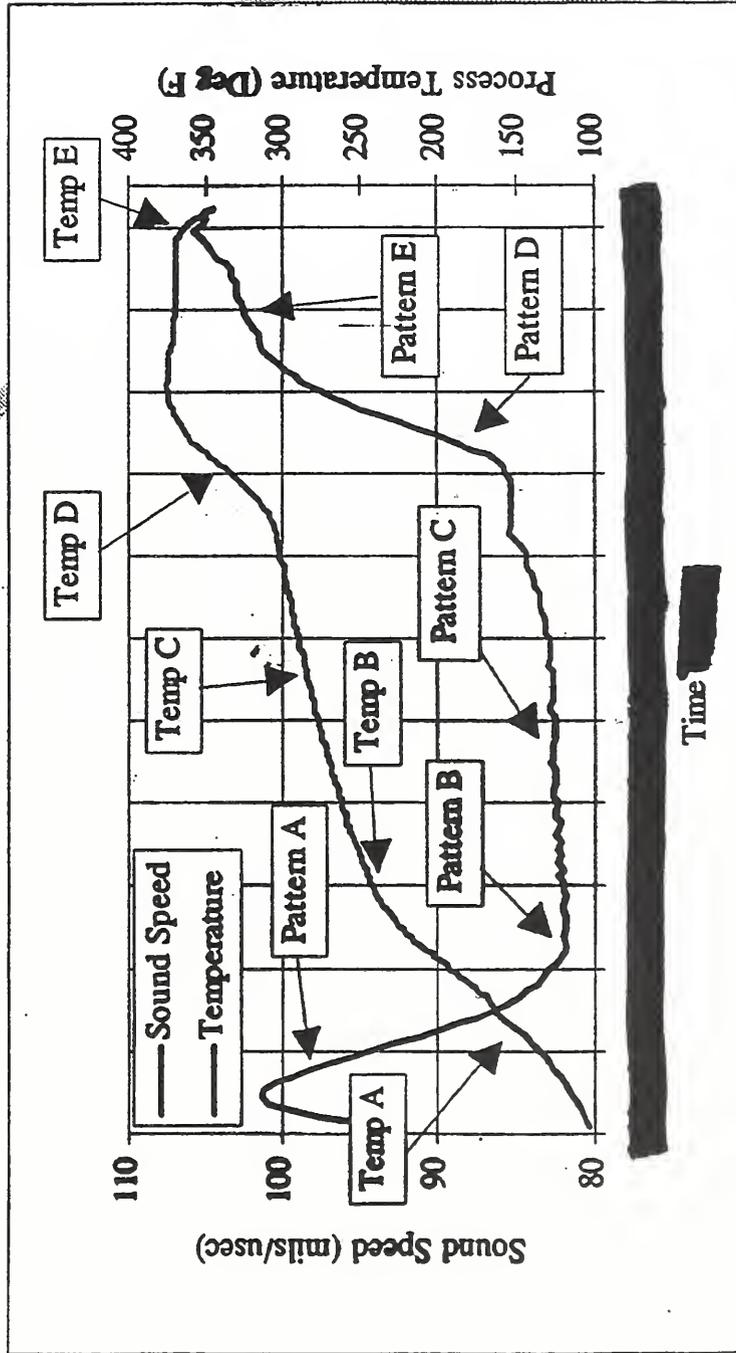


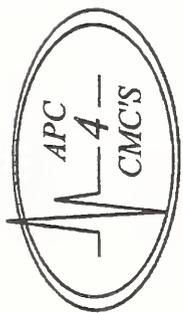
Figure 65

# EMBEDDED PROCESS RULES



## RULE BASED TEMPERATURE CYCLE





# EMBEDDED PROCESS RULES

## RULE BASED PRESSURE CYCLE

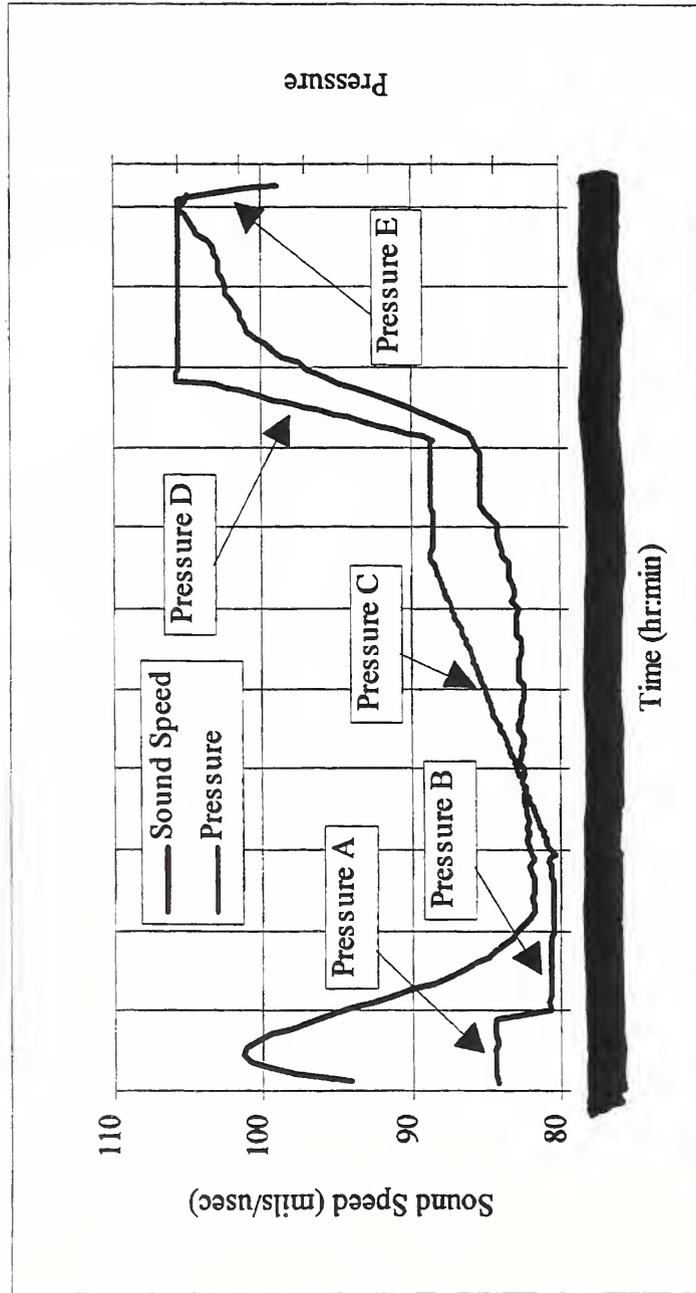


Figure 66

**IN-PROCESS CONTROL OF  
COMPOSITE FABRICATION**

# **Role of NDE**

**B. Boro Djordjevic**

**Robert E. Green, Jr.**

# **CNDE**

**Center for Nondestructive Evaluation**

**The Johns Hopkins University**

# Composite Process Monitoring and Verification

---

## In-Process

### FUNCTION:

- Process Control
- Quality Assurance
- Integrity Validation

### CONTROL PARAMETERS:

- Temperature
- Pressure
- Cure
- Melt and Infiltration
- Material Quality
- Wrap Process Control
- Material Compaction
- Exotherm

## Pre-Service

- Certification
- Damage Assessment
- Integrity Assessment
- Delaminations and Spalling
- Adhesion
- Fiber Break
- Strain State
- Resin Degradation
- Residual Stress

Figure 67

# DEFECTS IN COMPOSITES

Delaminations	Missing adhesive
Disbond	Misoriented fibers/plys
Porosity	Wavy fibers
Contamination	Mislocated plys/details
Improper cure	Impact damage
Resin rich/poor	Thickness variance
Damaged fiber	Dimensional problems
Voids	Interfaces integrity
Cracks	Structure specific problems

Figure 68

# NDE PRACTICES FOR COMPOSITES

## ADVANCED TECHNIQUES

**Ultrasonic**

**X-ray Computed Tomography**

**Acoustic emission**

**Laser ultrasonic**

**Tap test**

**Holography**

**Resonance**

**Laser-optical**

**X-ray**

**Vibro-thermography**

**Visual**

**Acousto-ultrasonic**

**Optical**

**D-sight**

**Thermal**

**Neutron radiography**

**Microwaves**

Figure 69

<b>THERMOSET:</b>	<b>THERMOPLASTIC:</b>
Cure (Polymerization)	Crystallinity
Temperature	Melt Stability
Exotherm	Melt Temperature
Viscosity	Viscosity
Modulus	Fiber/Resin Ratio
Tg Shifts	Density
Fiber/Resin Ratio	Porosity
Density	Degradation of Resin
Porosity	Delaminations
Fiber Orientation	Consolidation
Delaminations	Fiber Orientation
Overall Material Integrity	Overall Material Integrity
Others	Others

**Parameters and properties that can be nondestructively monitored for process control of organic matrix composites.**

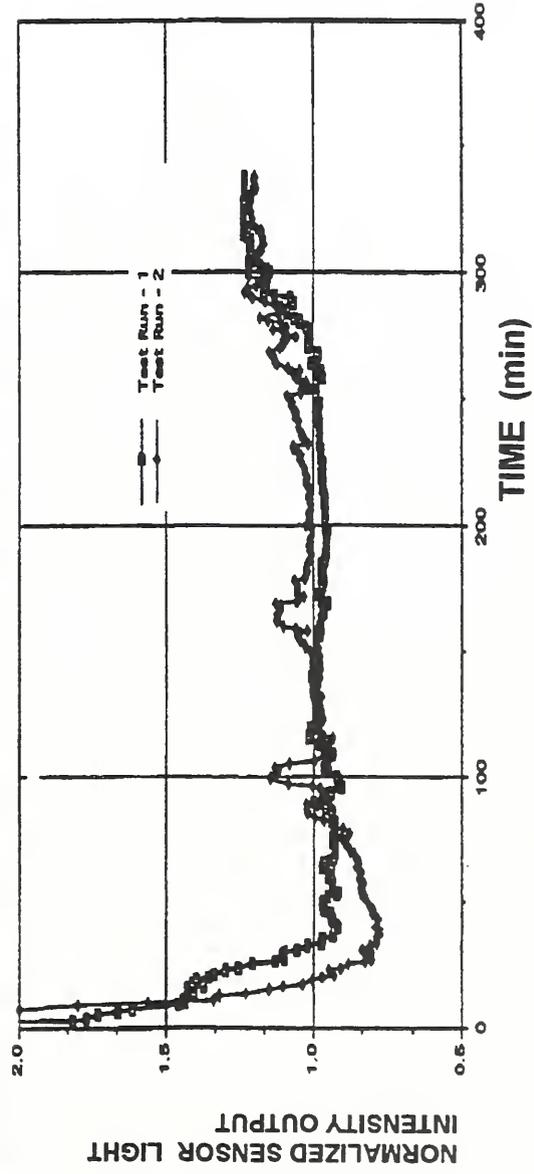
# Nondestructive Evaluation, In-process Enabling Technologies

<u>THERMOSET:</u>	<u>THERMOPLASTIC:</u>
Thermocouple	Acoustic Waveguide
Acoustical Waveguide	Optical Fibers
Optical Fibers	Thermal
Capacitance	Ultrasonics
Conductivity	Magnetostrictive Wire
Dielectric	
Thermal	
FT-IR	
Ultrasonic	
Magnetostrictive Wire	

**Potential devices for process monitoring of organic matrix composite materials.**

Figure 71

# Nondestructive Evaluation Enabling Technologies



## Fiber Optics In-Process Cure Monitoring:

# Nondestructive Evaluation Enabling Technologies

## Optical-Fiber/Glass-Rod

## Cure Monitoring Set-up:

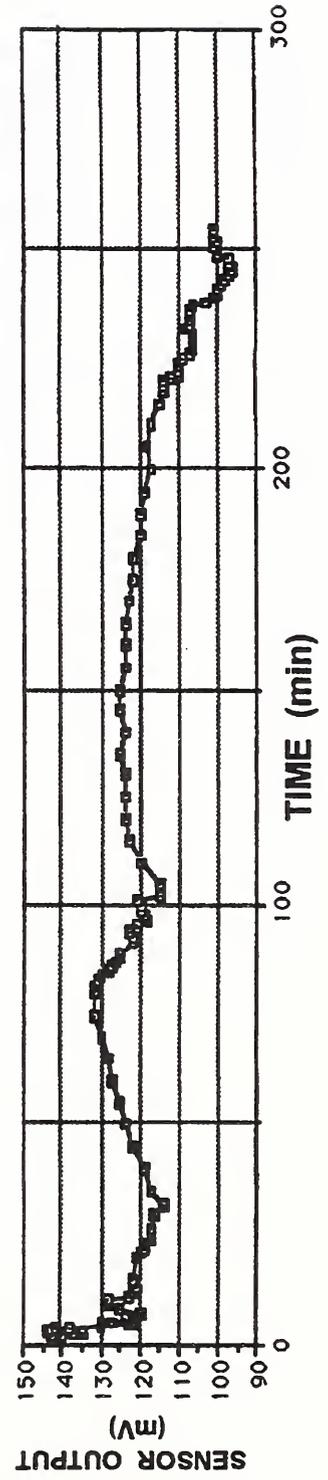
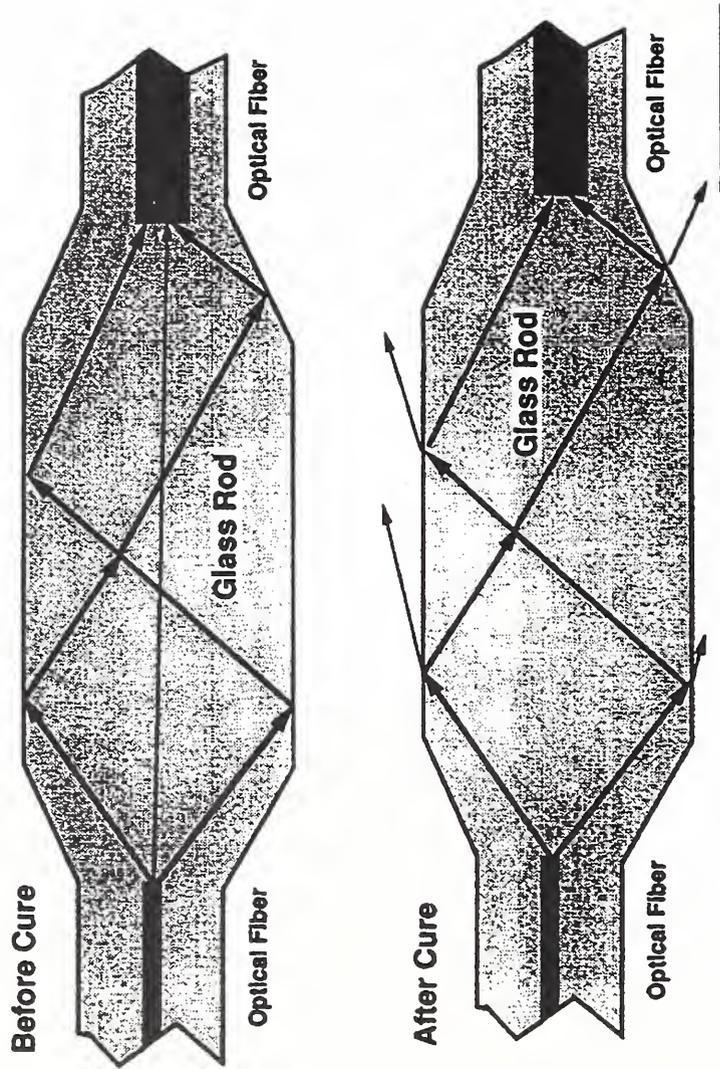


Figure 73

# UT Sensors for In- Process Control

## Embedded Acoustic Sensors

- Flexural Mode

Dispersive for aspect ratios < 1.2

Velocity  $\rightarrow$  0 as aspect ratio  $\rightarrow$  0

$$\begin{aligned}
 a &= 0.05 \text{ cm} \\
 \lambda &= 10 \text{ cm} \\
 a/\lambda &= 0.005
 \end{aligned}$$



- Torsional Mode

Non-dispersive for all aspect ratios



$$\text{Velocity} = \sqrt{\frac{G}{\rho}} = \sqrt{\frac{8.2 \times 10^{10} \text{ Pa}}{8420 \text{ kg/m}^3}} = 3120 \text{ m/s}$$

- Extensional Mode

Dispersive for aspect ratios from 0.1 to 1.2, non-dispersive elsewhere.



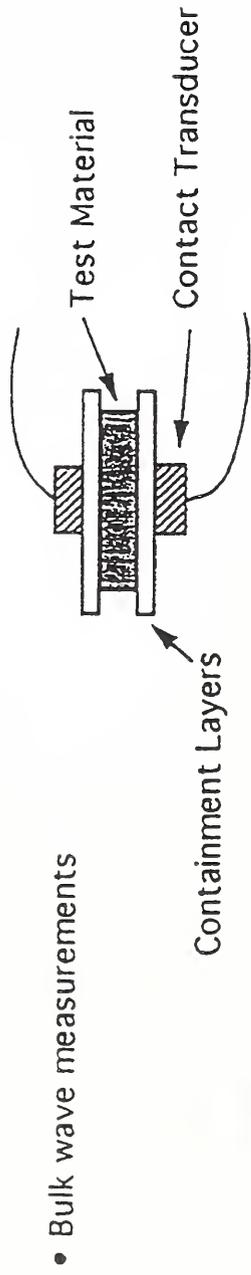
For long wavelengths where  $a/\lambda < 0.1$

$$\text{Velocity} = \sqrt{\frac{E}{\rho}} = \sqrt{\frac{2.14 \times 10^{11} \text{ Pa}}{8420 \text{ kg/m}^3}} = 5041 \text{ m/s}$$

Figure 74

## UT Sensors for In-Process Control

### Embedded Acoustic Sensors



- Interface waves (surface acoustic, Stonleigh, etc)



- Guided Waves

Material geometry (i.e. Lamb and Leaky Lamb waves)



Fiber Waveguide



# UT Sensors for In-Process Control

## Embedded Acoustic Sensors

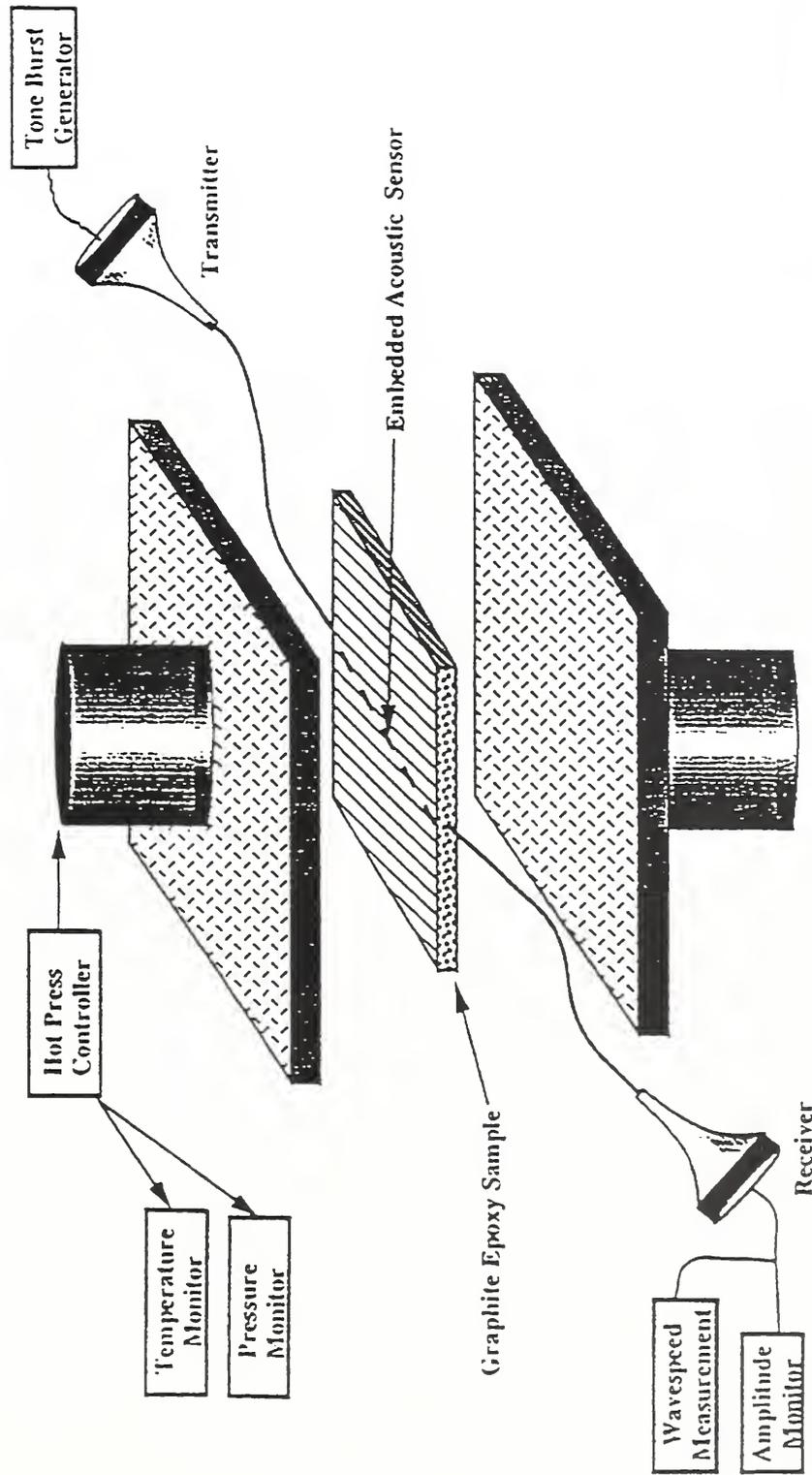


Figure 76

# Nondestructive Evaluation Enabling Technologies

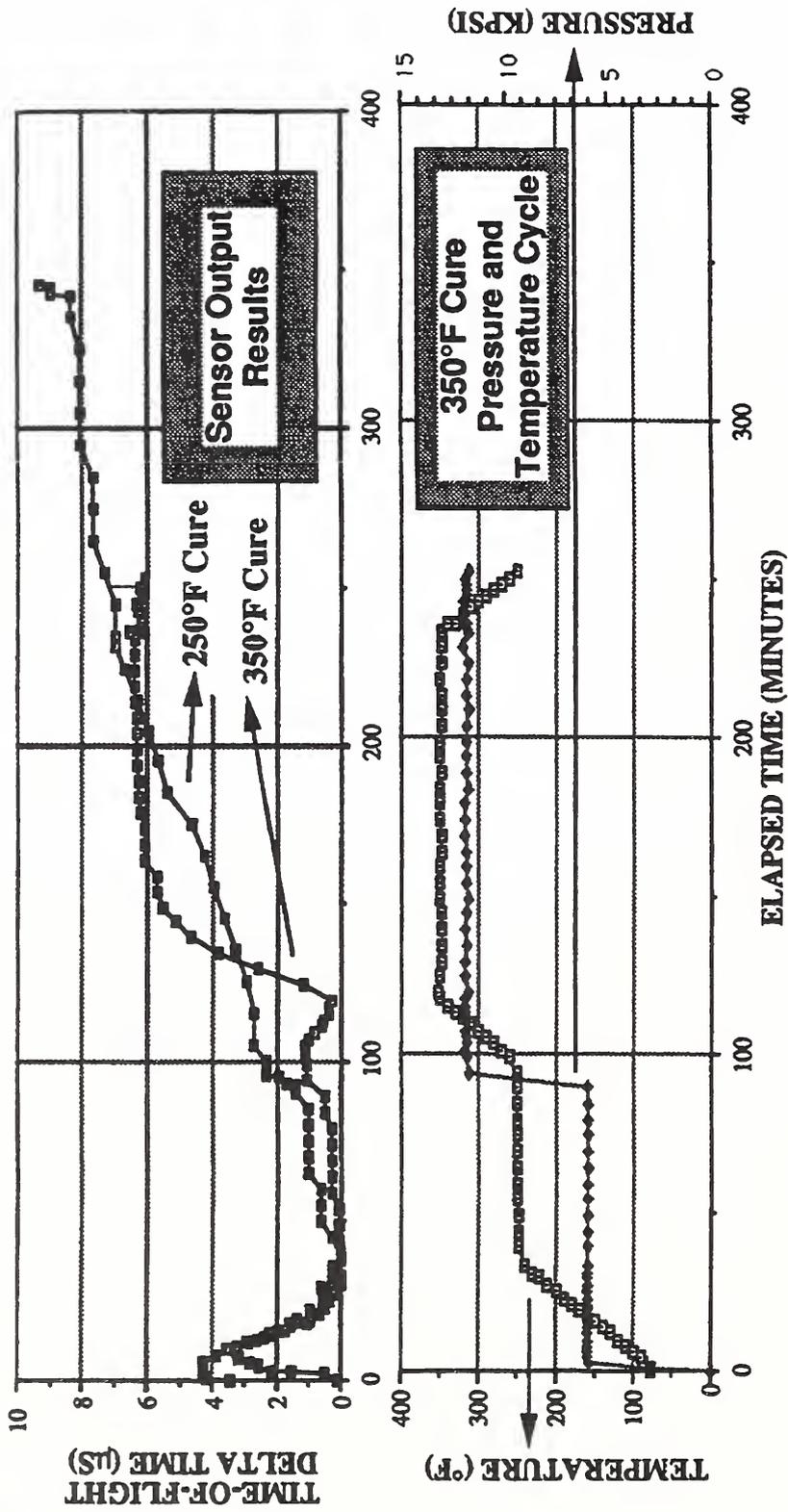


Figure 77

## Ultrasonic Waveguide In-Process Cure Monitoring: Embedded Waveguide Response

# Nondestructive Evaluation Enabling Technologies

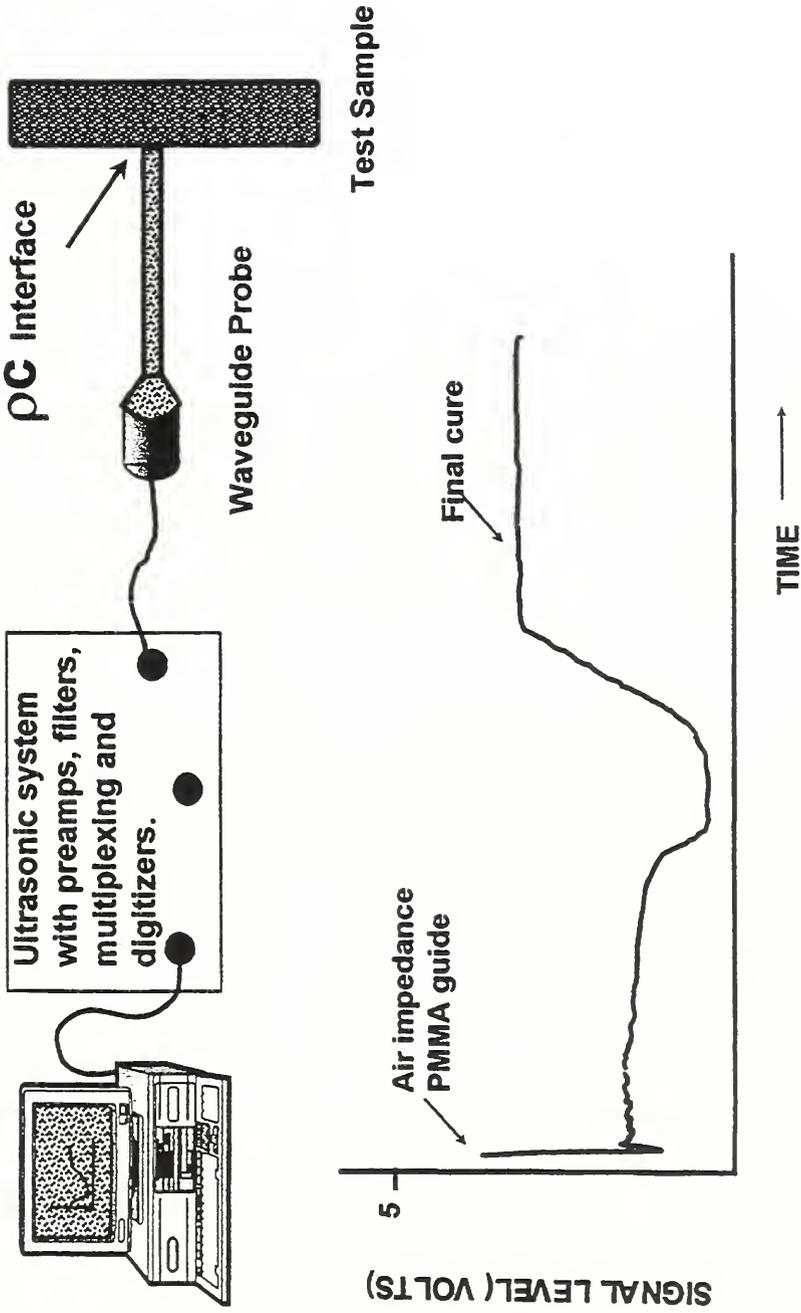
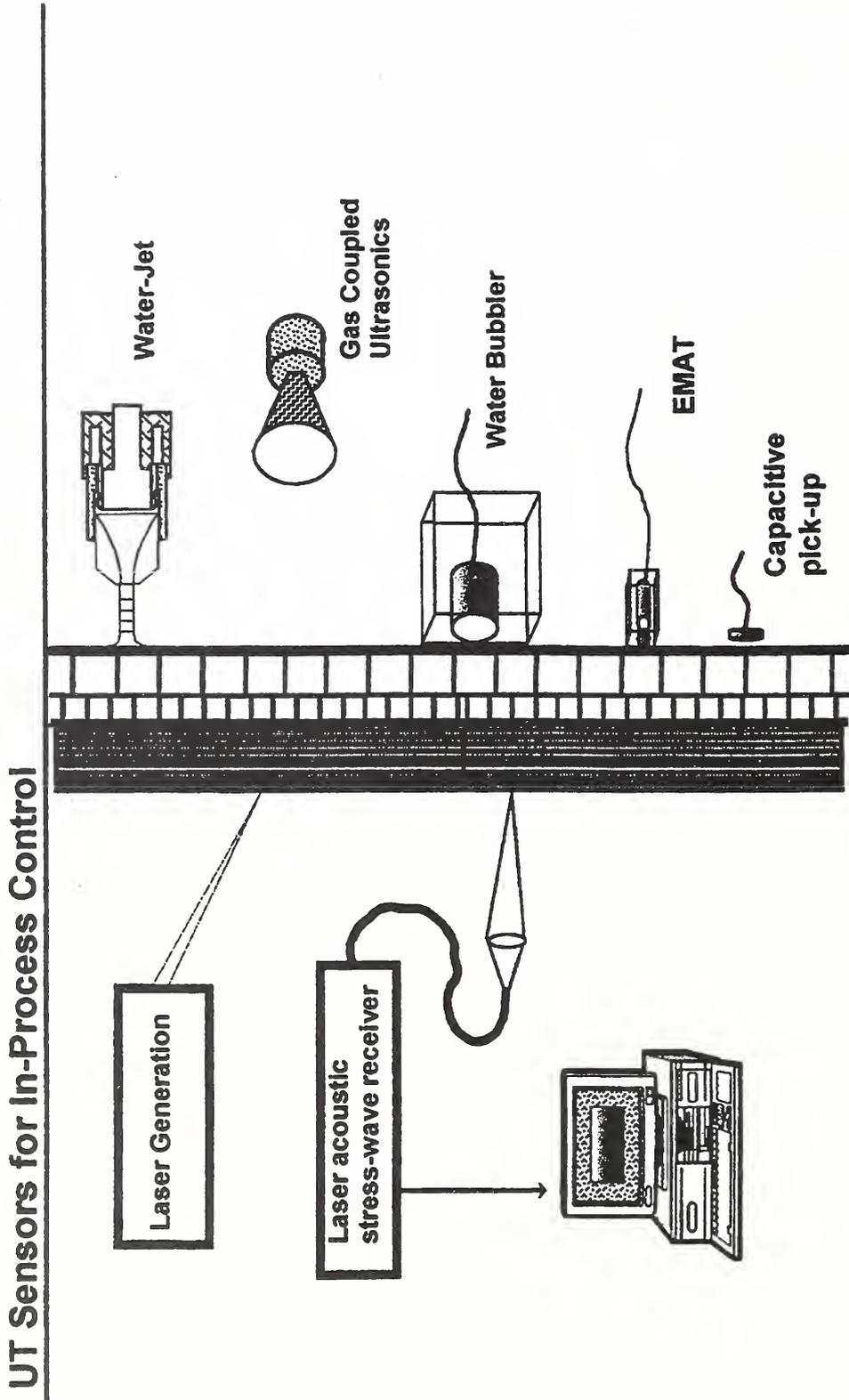


Figure 78

## Ultrasonic Waveguide In-Process Cure Monitoring: PC guide termination response



**Types of scanning ultrasonic probes available for composite manufacturing process control.**

# Non-contact UT for Process Control

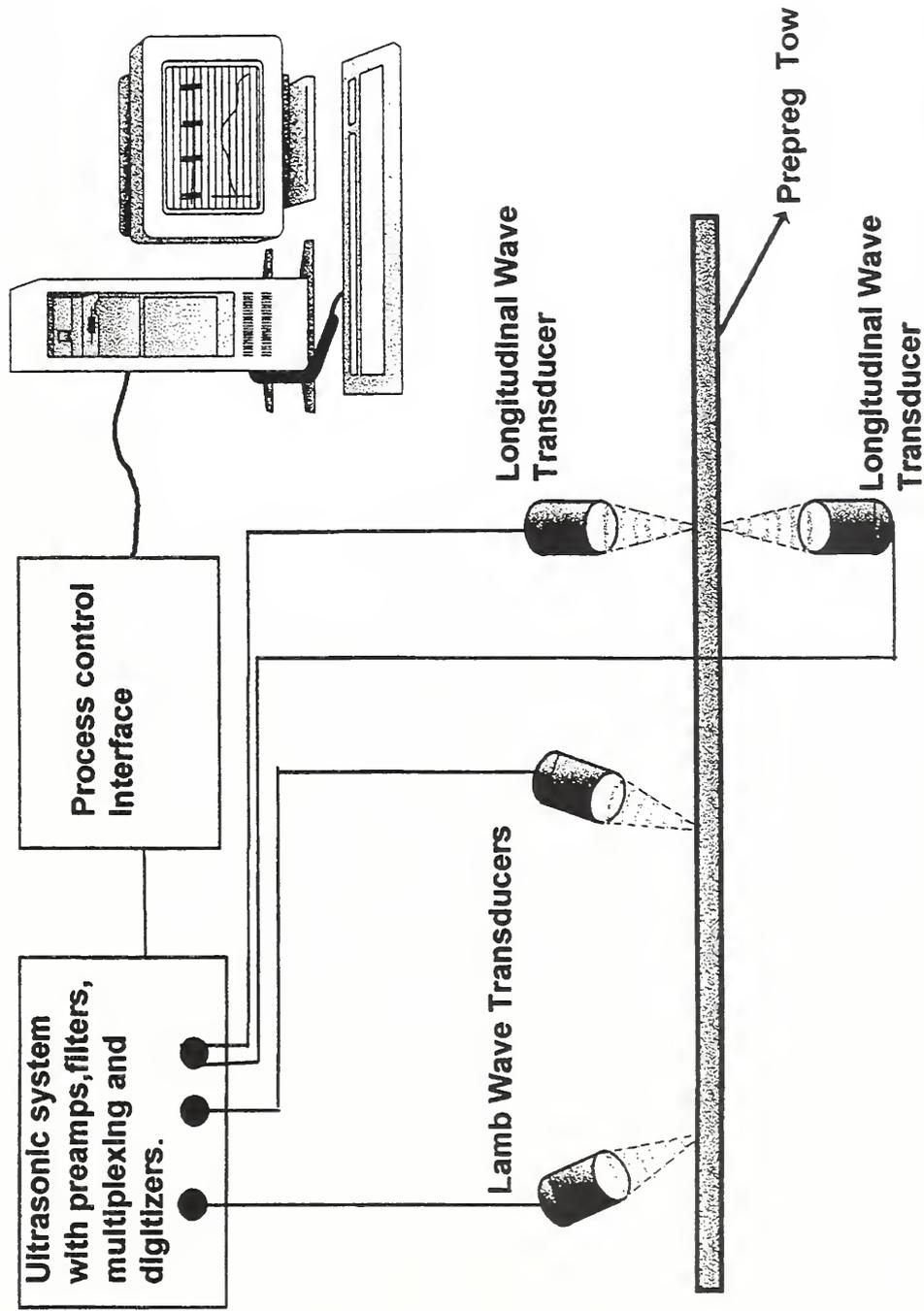
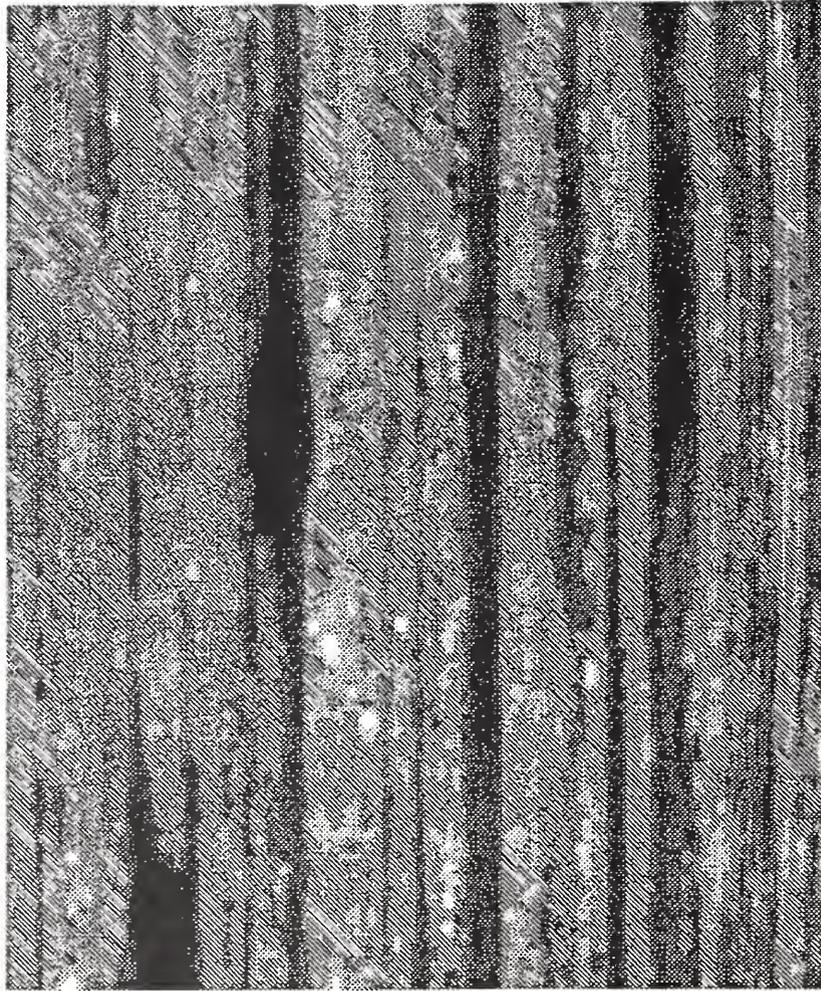


Figure 80

Air-coupled ultrasound system for on-line monitoring of prepreg tows.

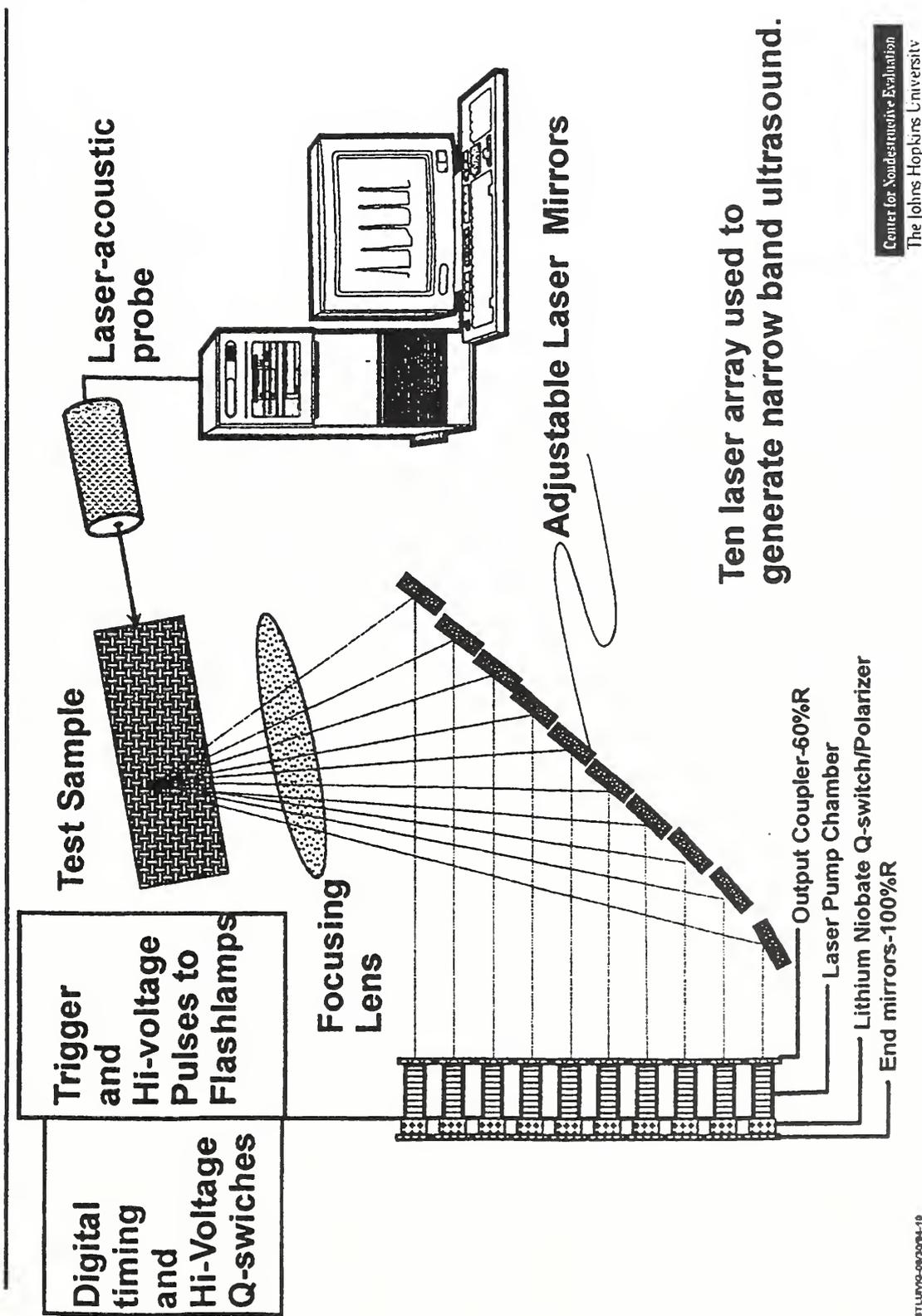


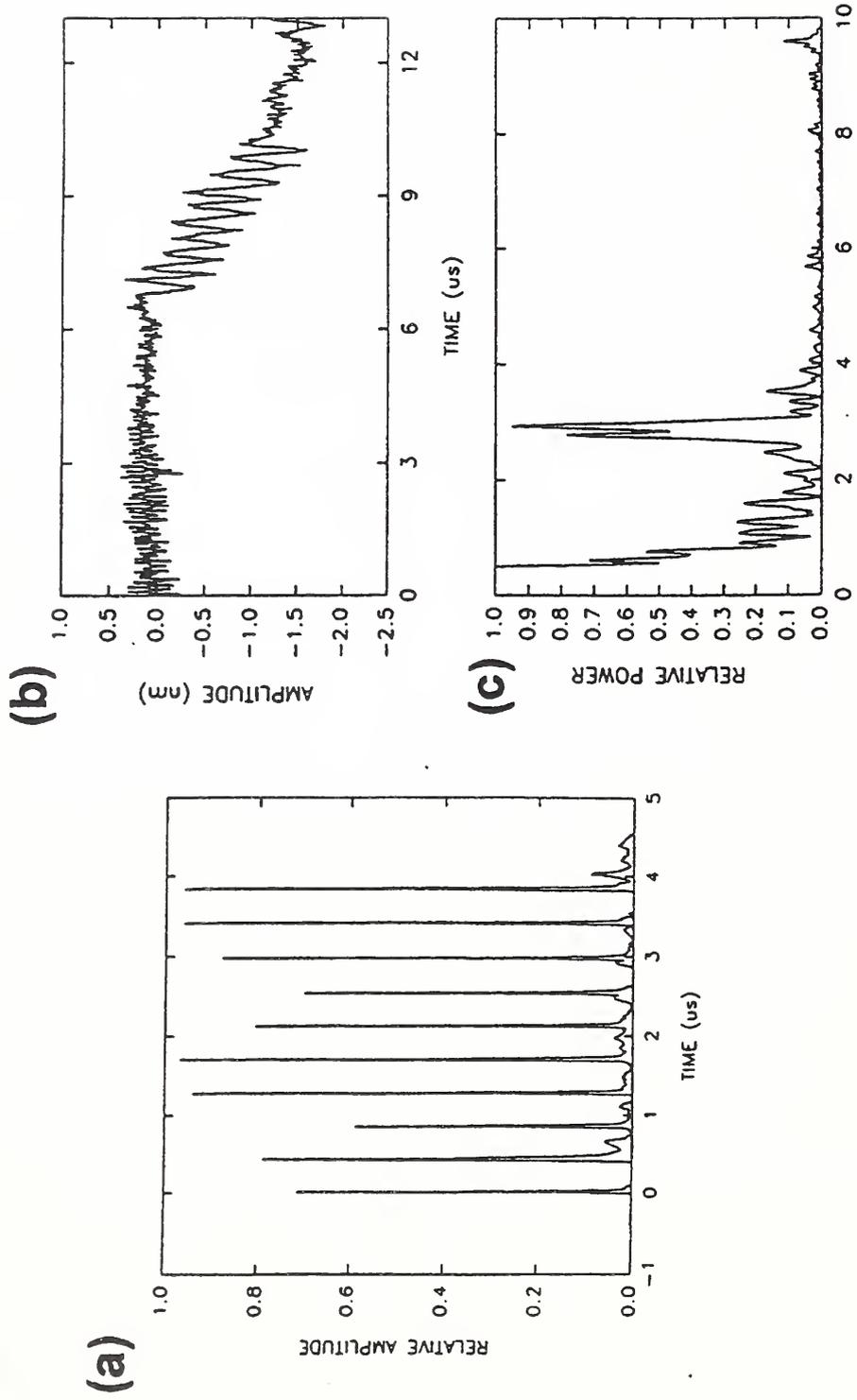
4.5 inch

5.5 inch

Air-coupled 0.5 MHz ultrasonic scan of graphite/epoxy prepreg.  
(AS4/3501-6)

Figure 82





**Laser pulse signal from the ten cavity laser system (a), generating ultrasonic signals (b), that exhibit narrow bandwidth centered at 3 MHz (c).**

# Nondestructive Evaluation - Ultrasonic Scan Technology

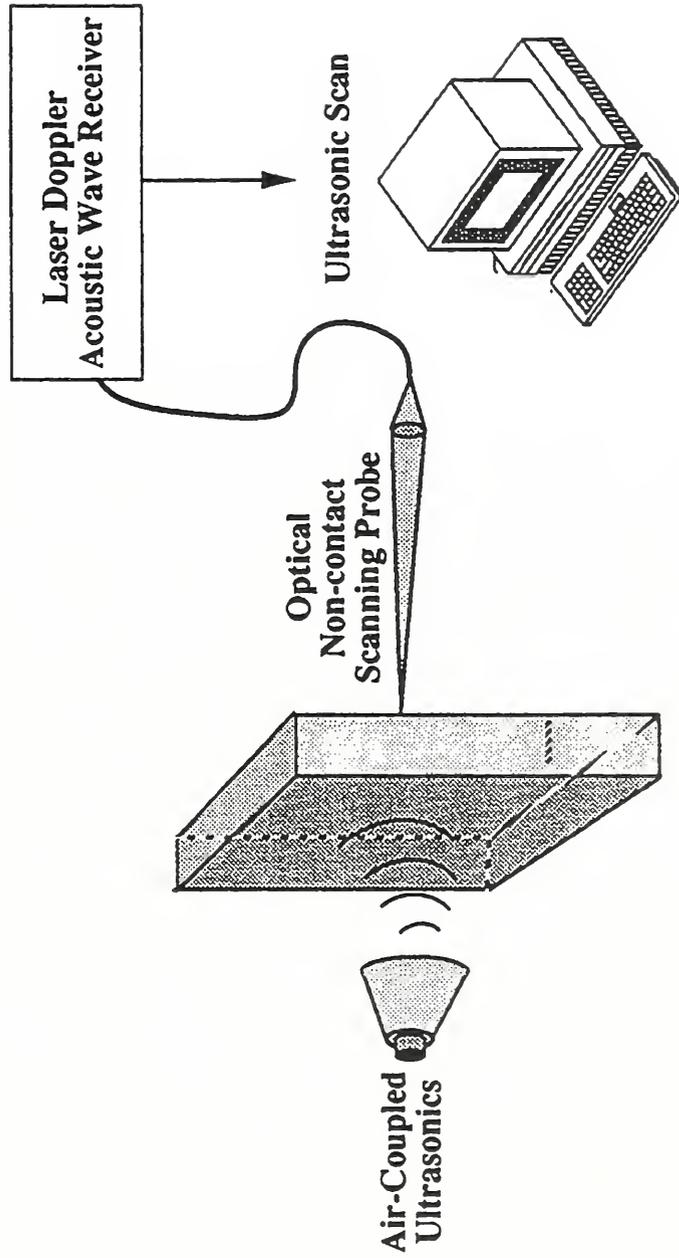
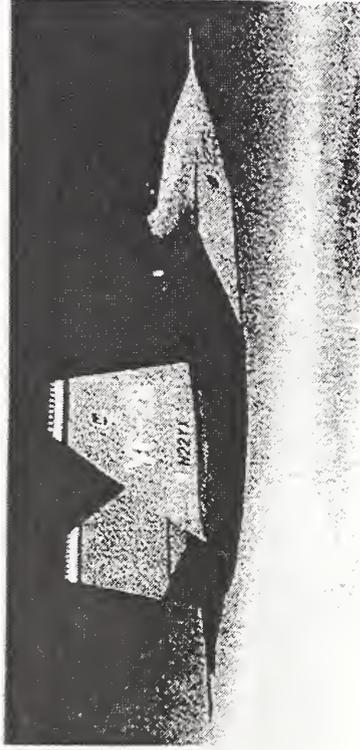


Figure 84

BD0793Austria

## Nondestructive Evaluation, In-process Enabling Technologies



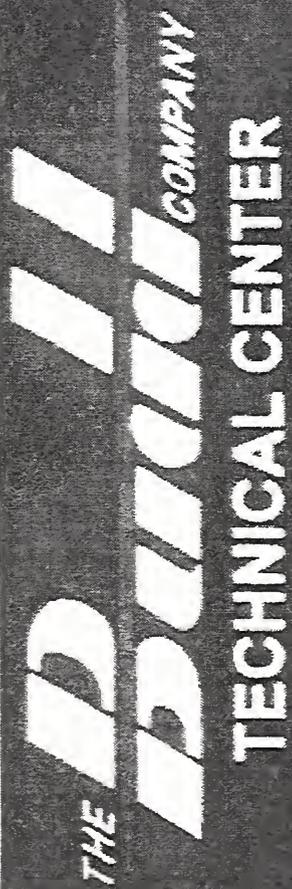
Composites manufacturing requires in-process controls and new sensors to enable viable use of these materials.

Nondestructive tools currently in use are old and focused to final product inspection.

New, non-traditional sensors are required to enable effective process control in manufacturing of composite structures.

Composite materials can meet many new commercial and DoD requirement but potential utilization is limited due to expense and lack of in-process monitoring technology.

Figure 85



# HIGH SPEED PROCESSING OF COMPOSITES

**BRUCE GREVE**

**SR. STAFF ENGINEER**

Figure 86

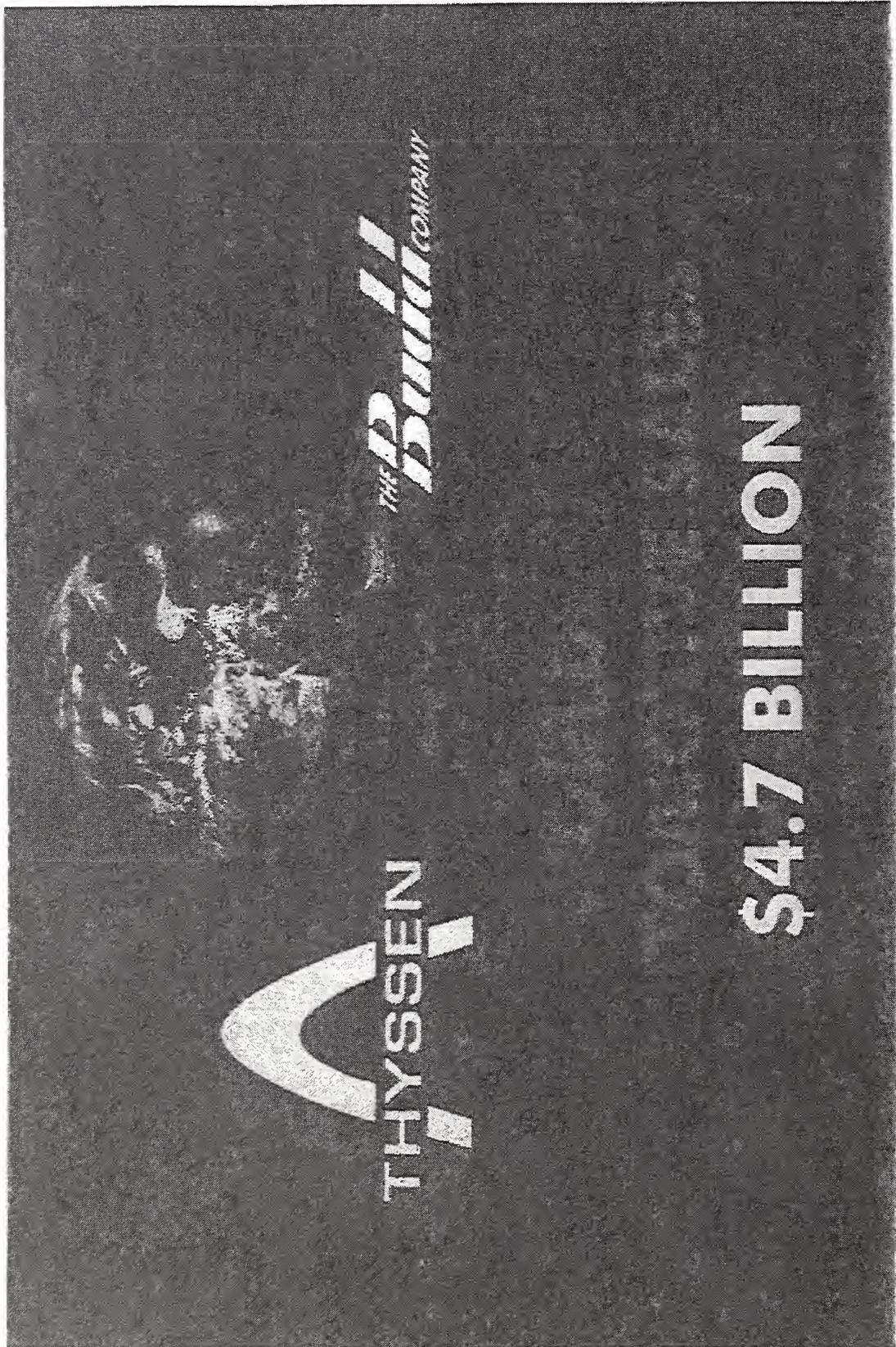


Figure 87

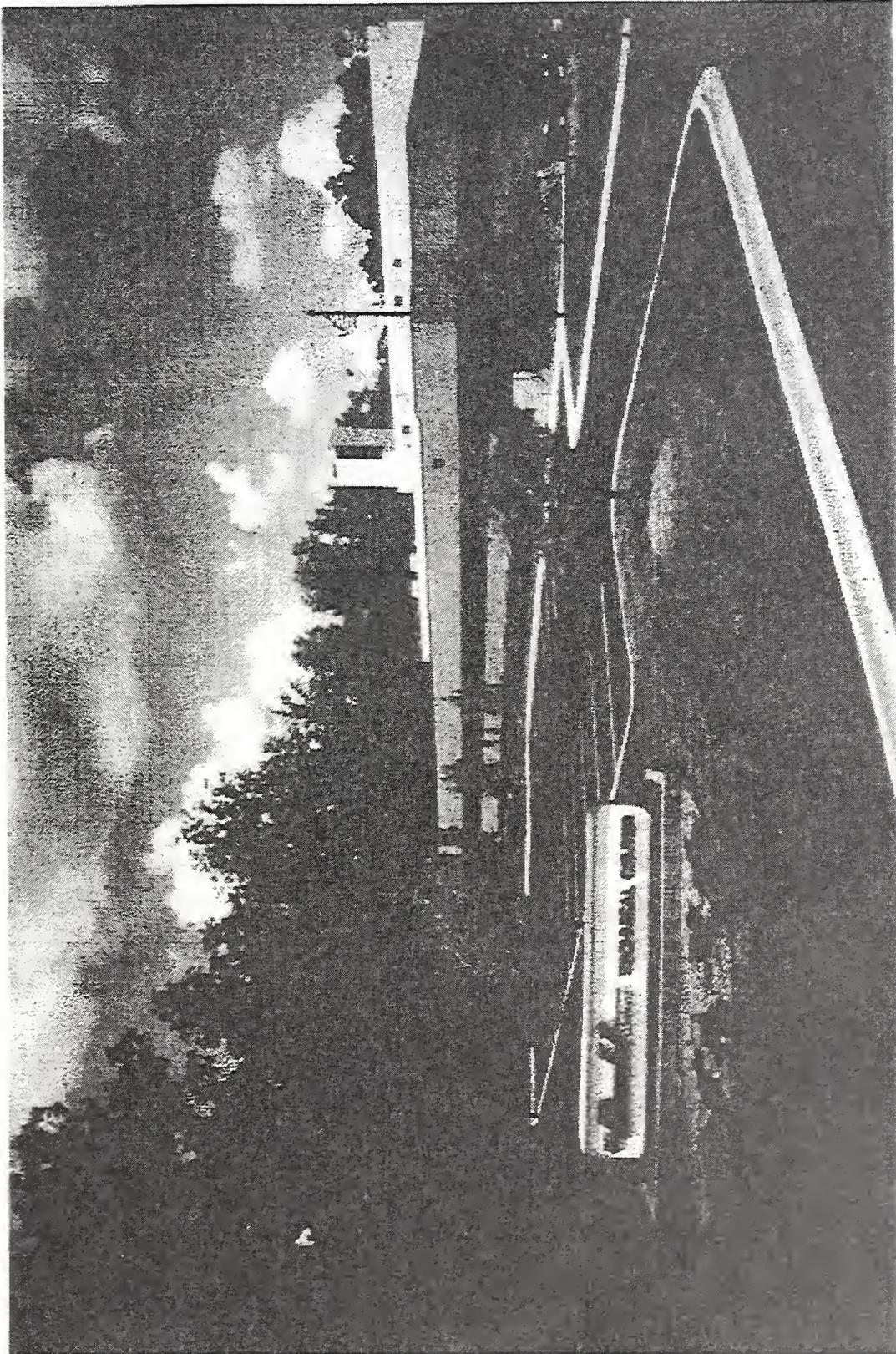


Figure 88

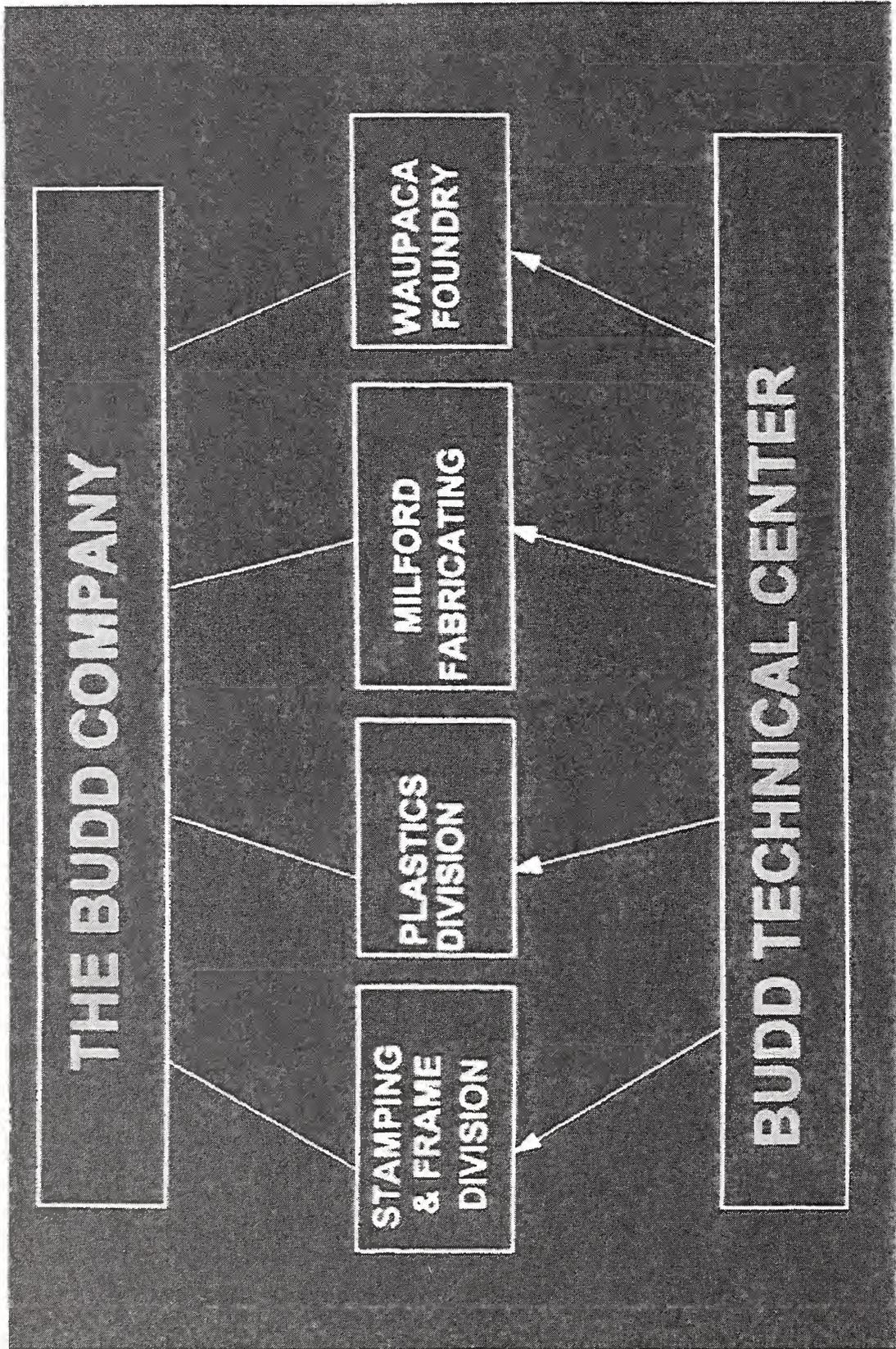
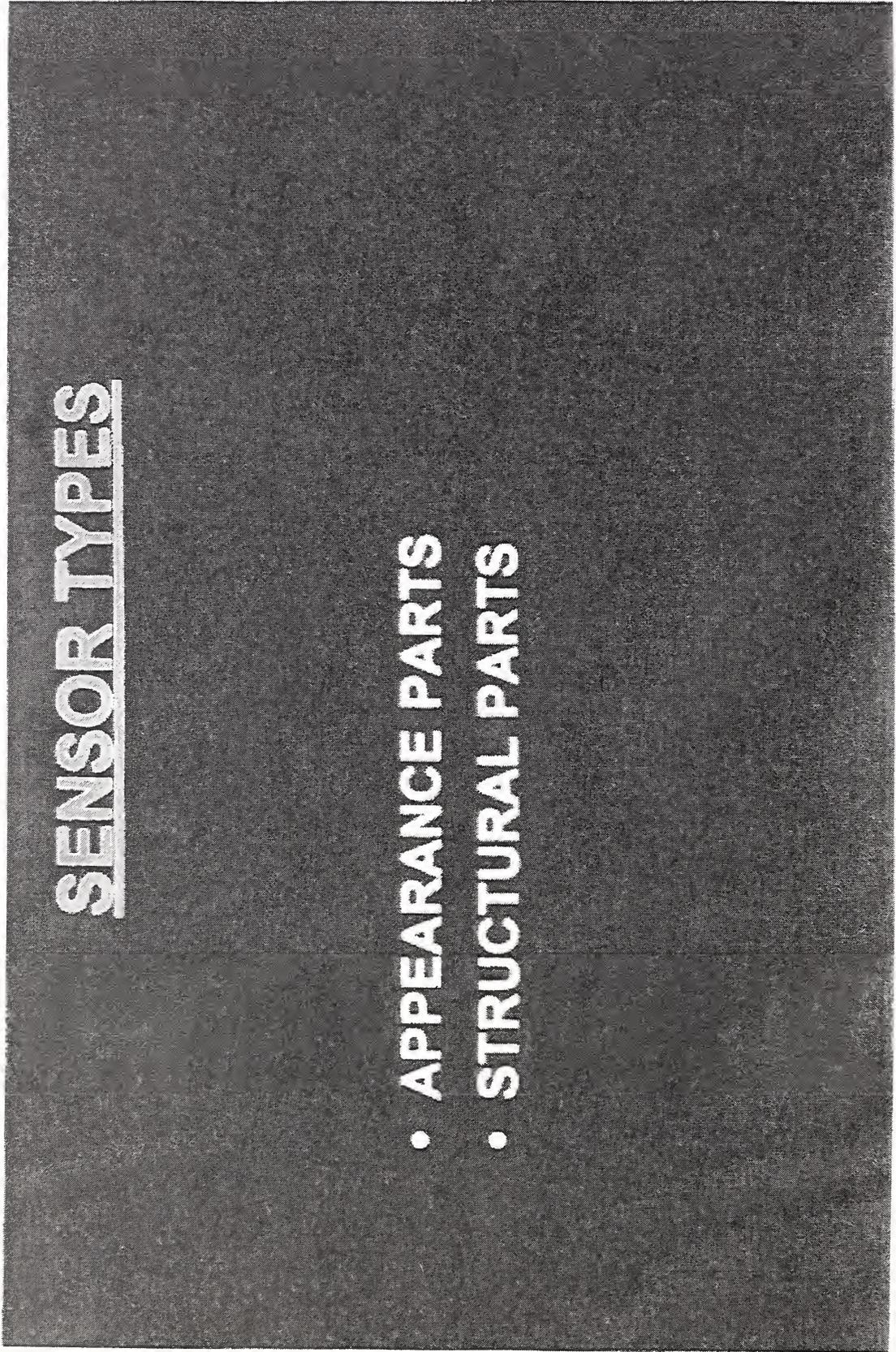


Figure 89

**SBC APPLICATIONS**

- 300 Different SBC Components
- On 100 Domestic and Import Passenger Car and Truck Lines
- Used by 28 Vehicle Manufacturers

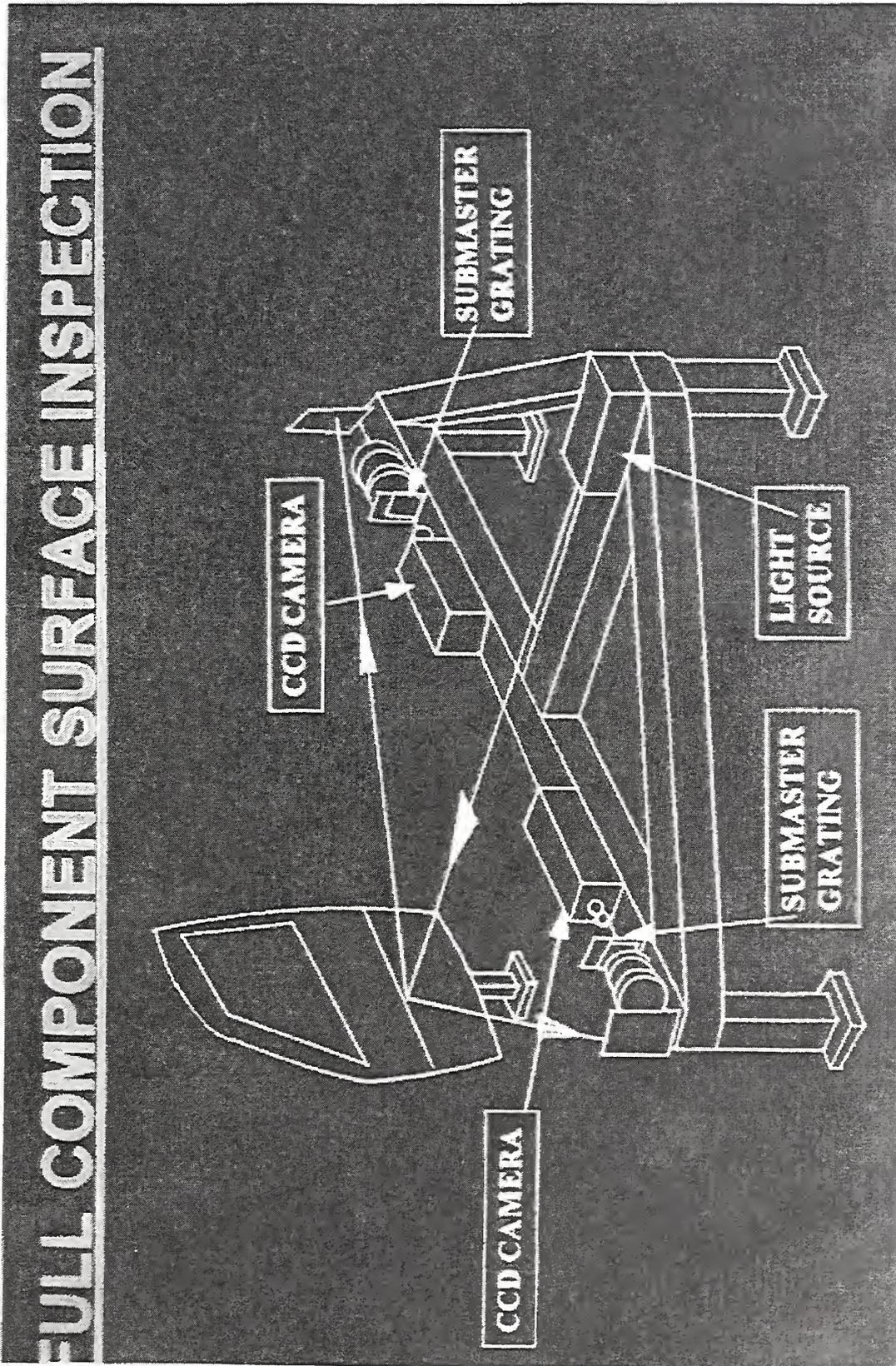
The collage features six distinct images: a dark sedan, a white sedan, a dark sedan, a dark sedan, a dark sedan, and a dark sedan. The text is overlaid on the images in a stylized, bold font.



**SENSOR TYPES**

- APPEARANCE PARTS
- STRUCTURAL PARTS

Figure 91



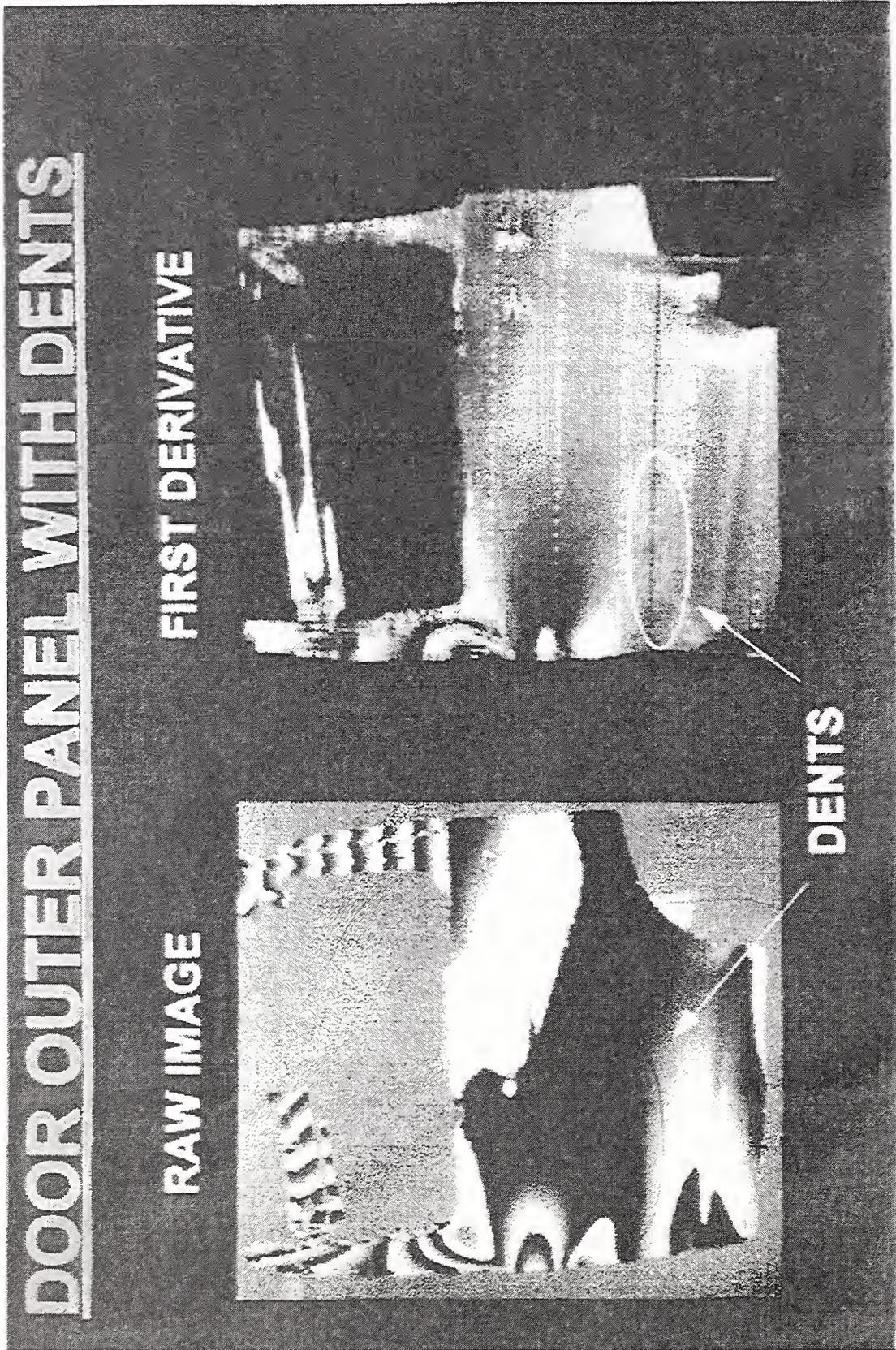


Figure 93

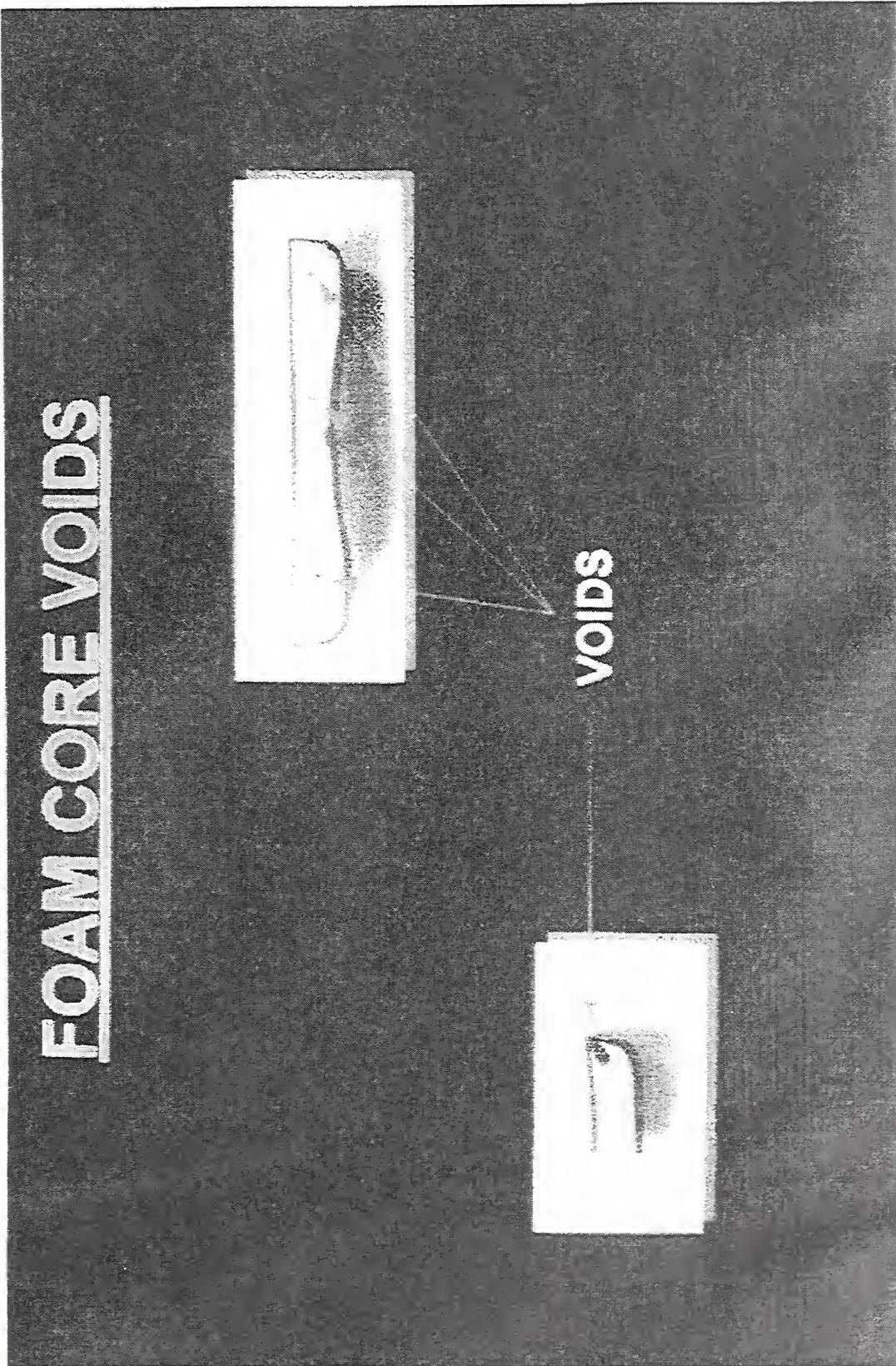


Figure 94

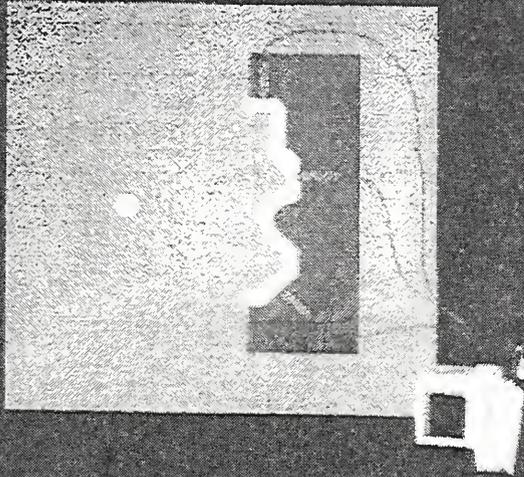


Figure 95

**PREFORM INSPECTION**

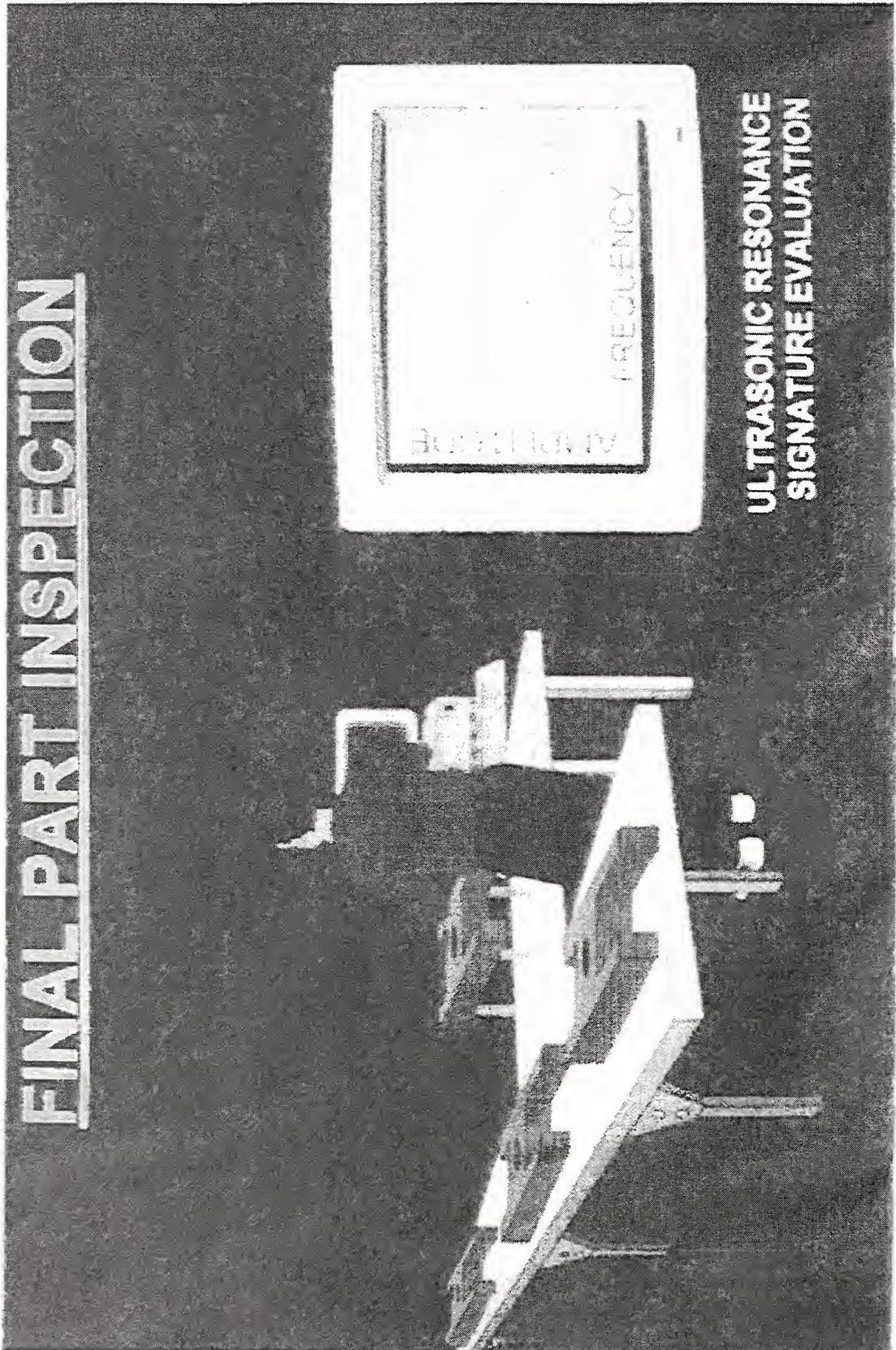


**OFF-LINE QUALITATIVE**



**ON-LINE QUANTITATIVE**

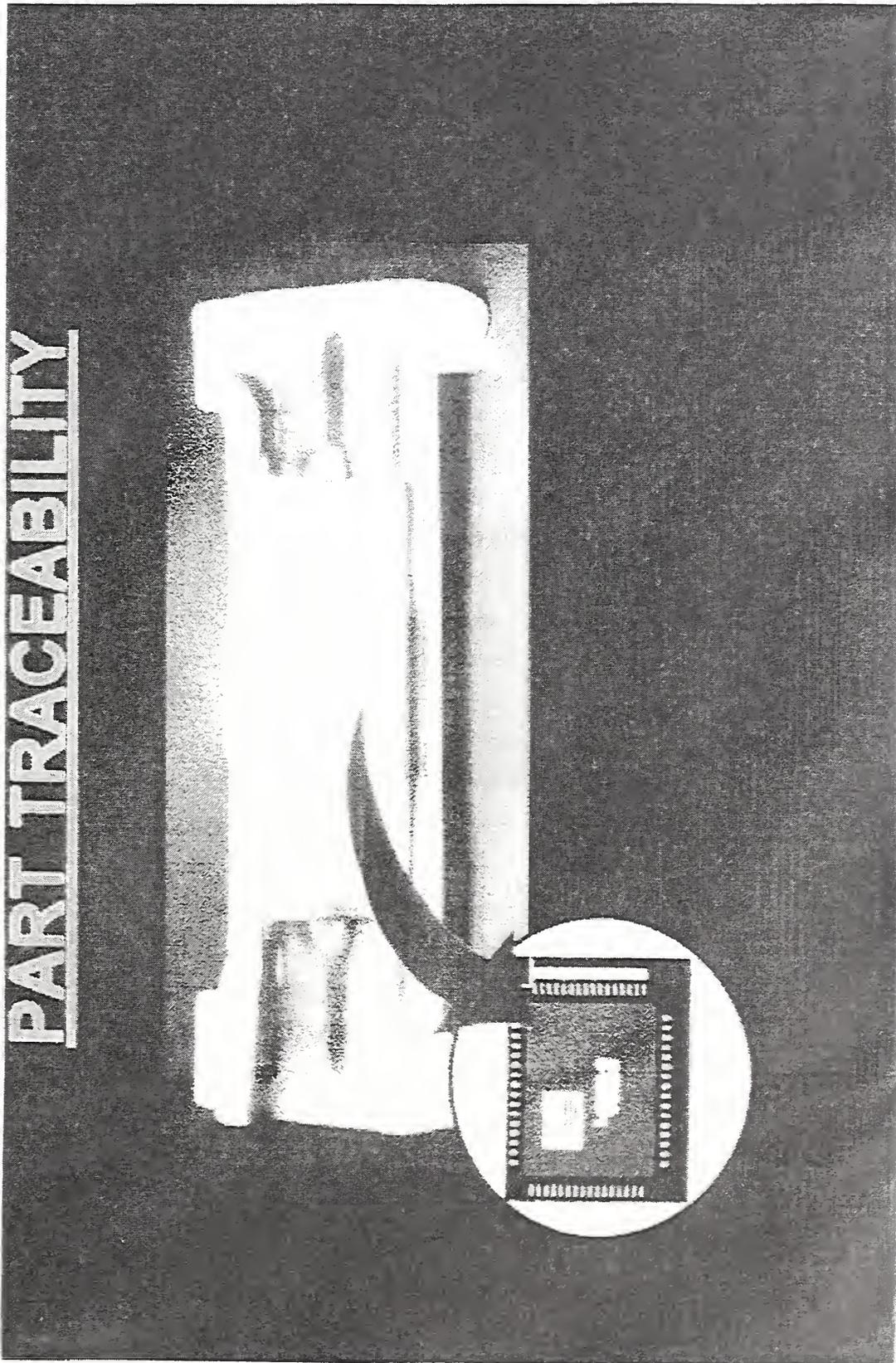
Figure 96



**FINAL PART INSPECTION**

**ULTRASONIC RESONANCE  
SIGNATURE EVALUATION**

Figure 97



# WHERE DO THE PROCESSES FIT?

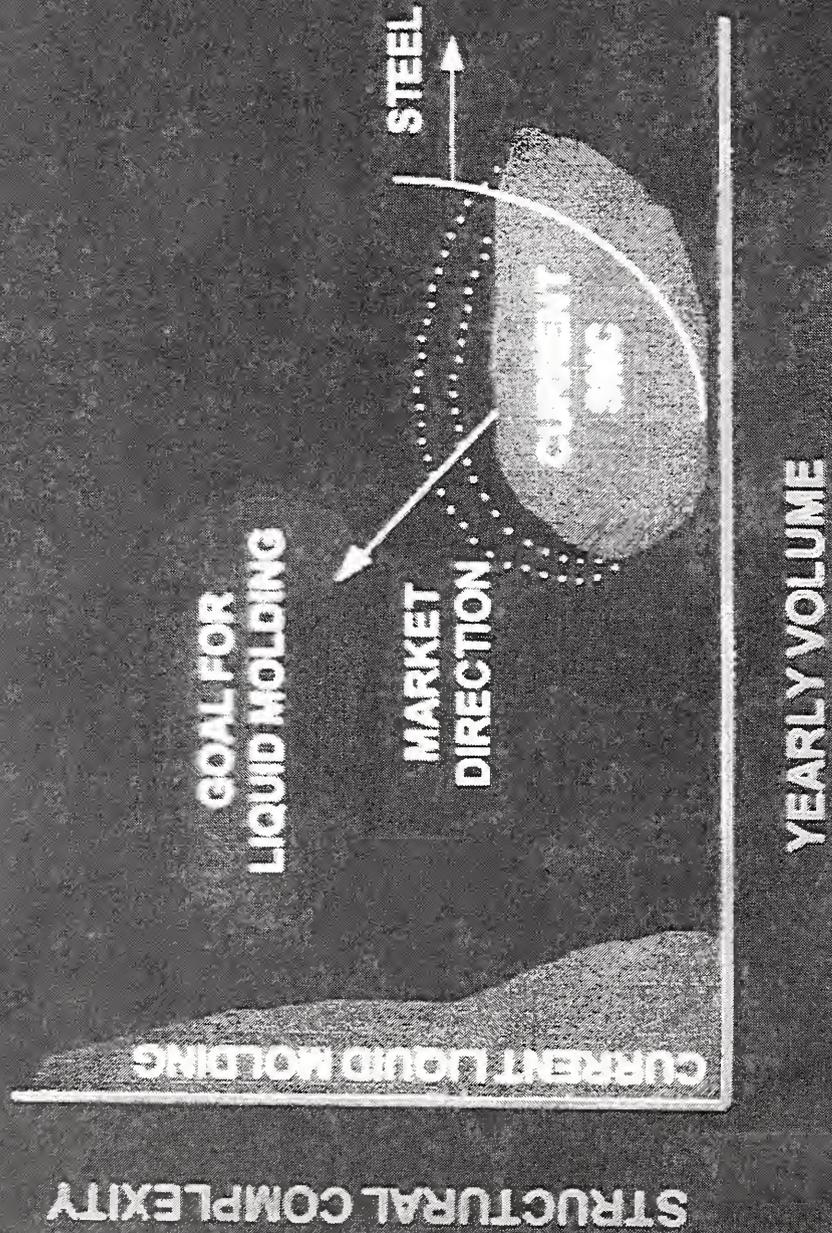


Figure 99

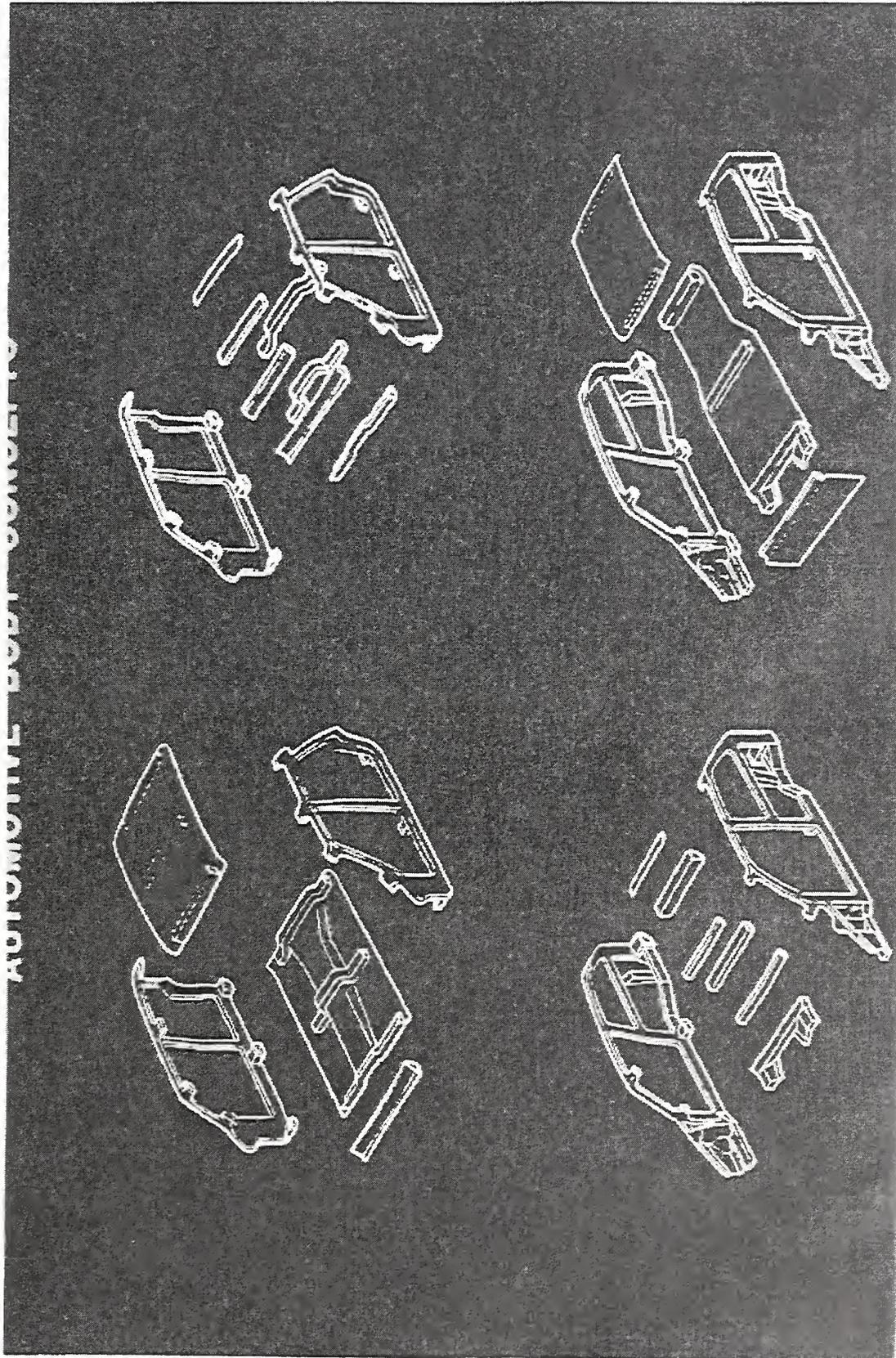


Figure 100

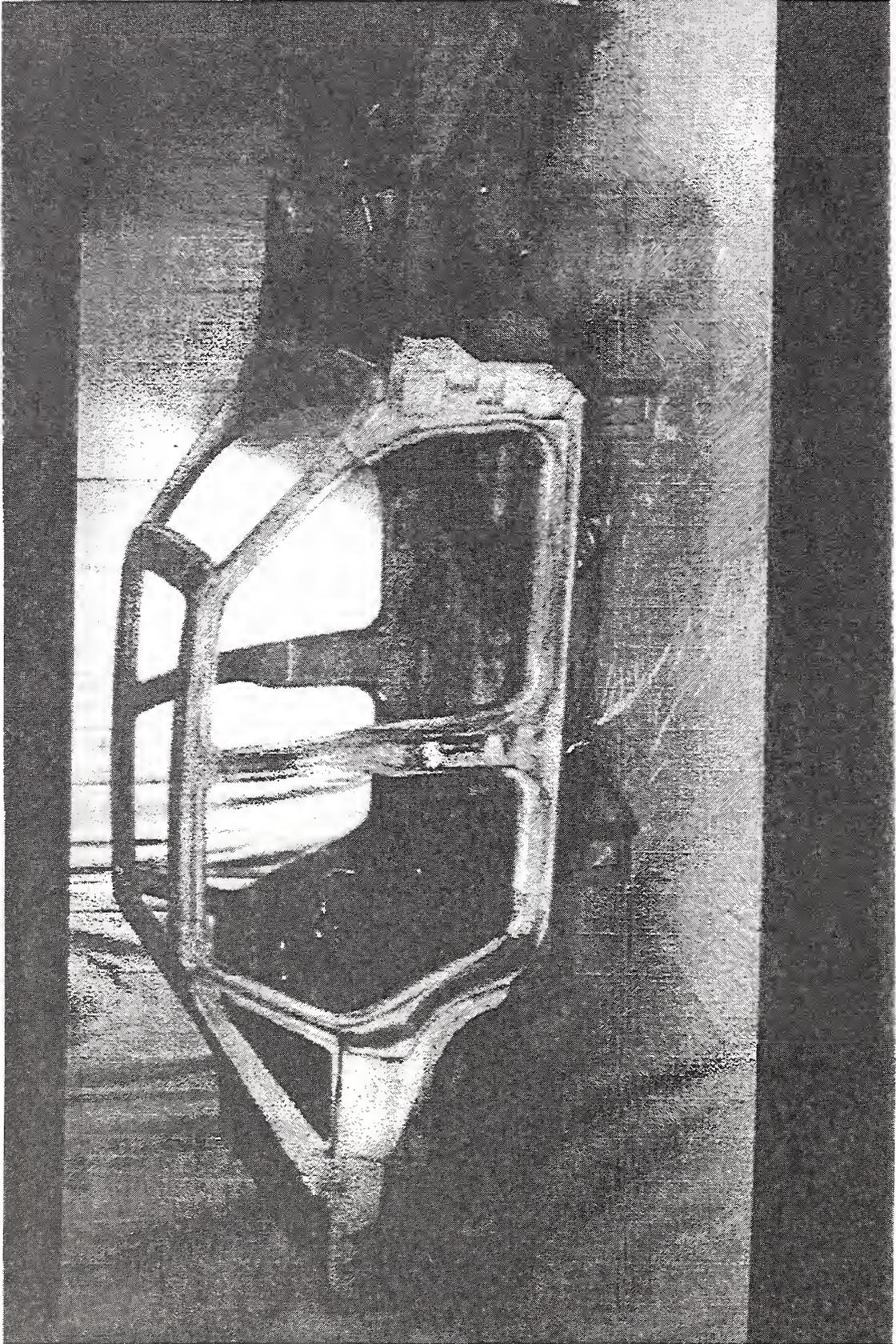


Figure 101

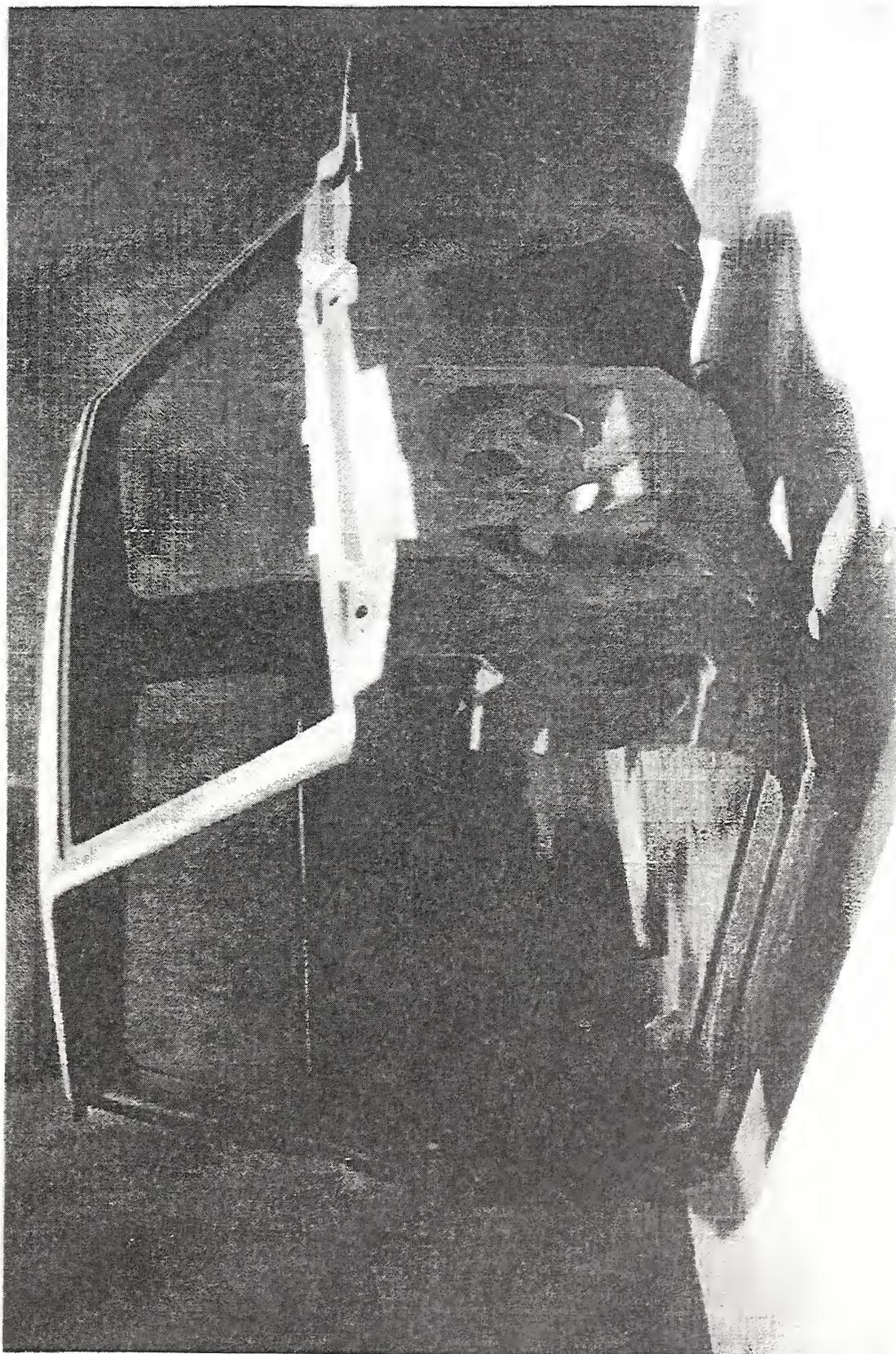


Figure 102

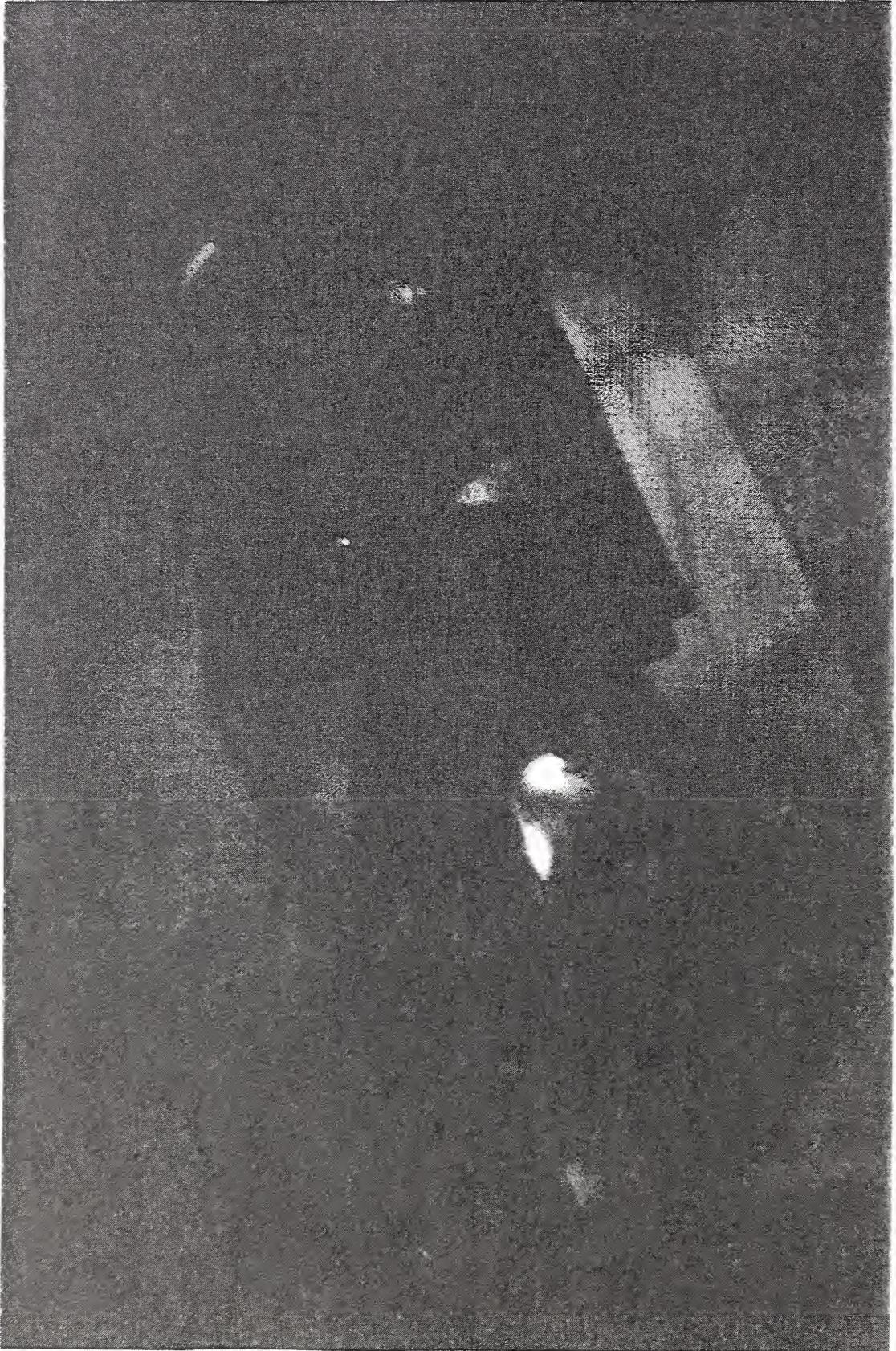


Figure 103

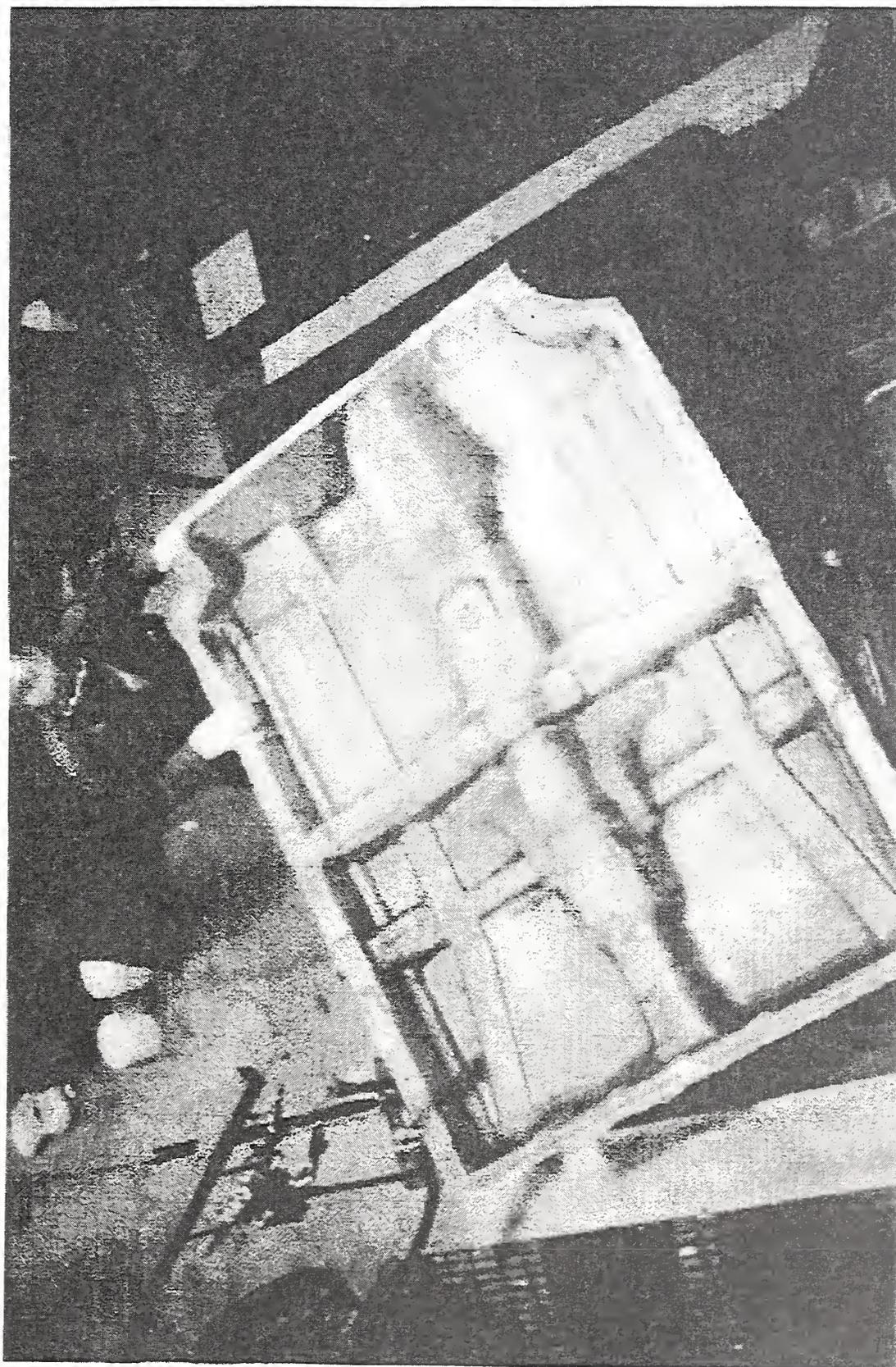


Figure 104

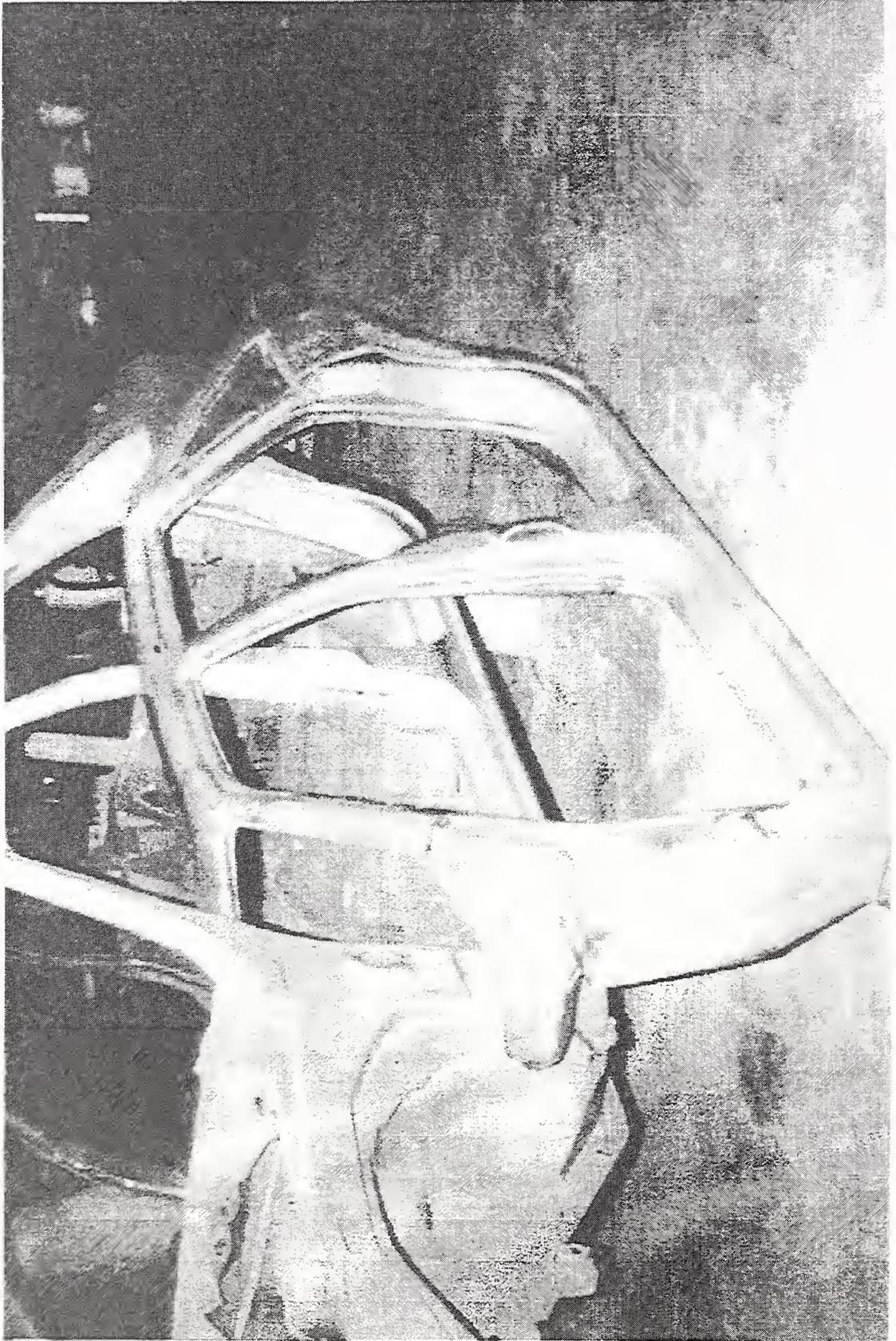


Figure 105

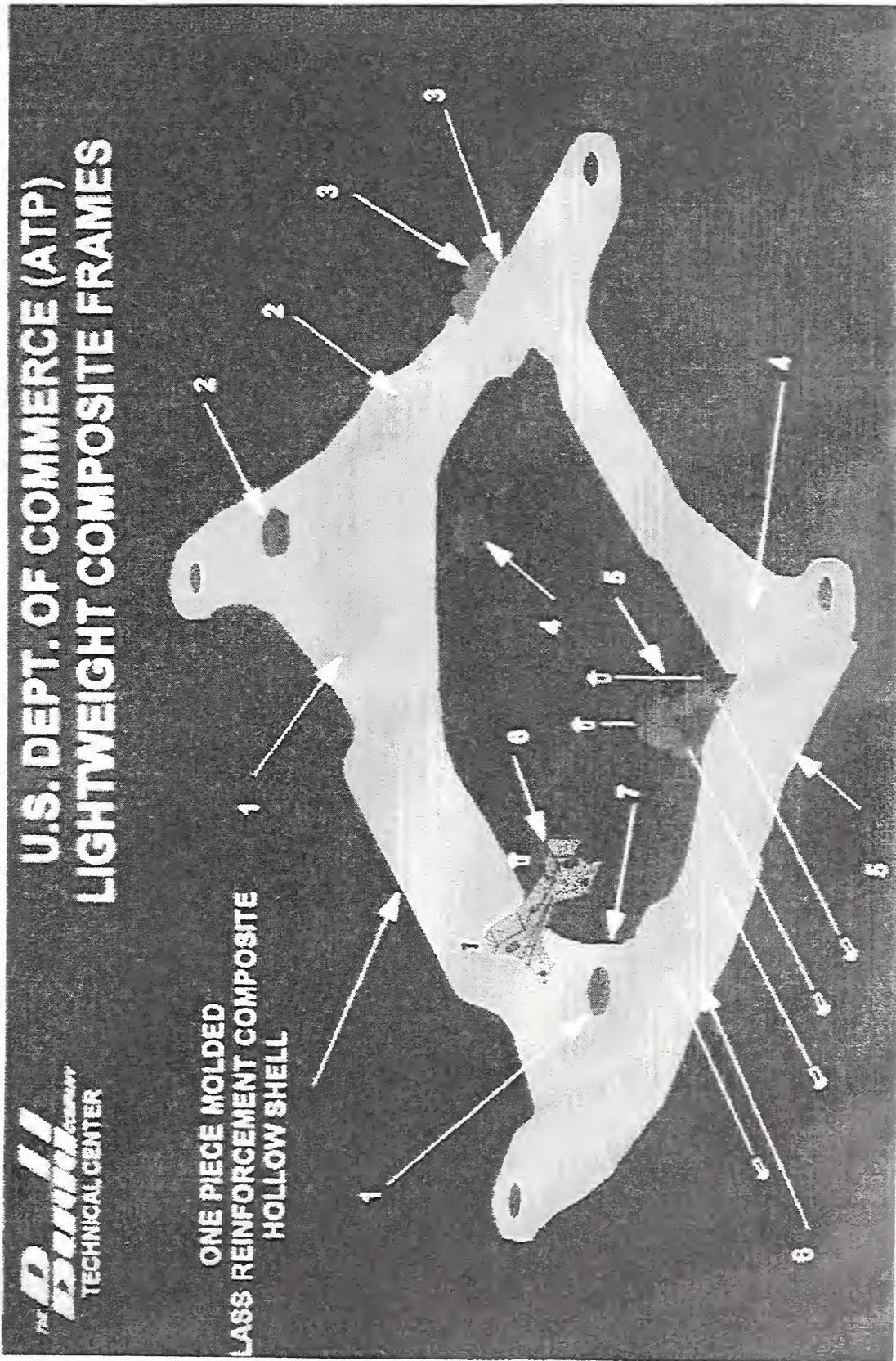
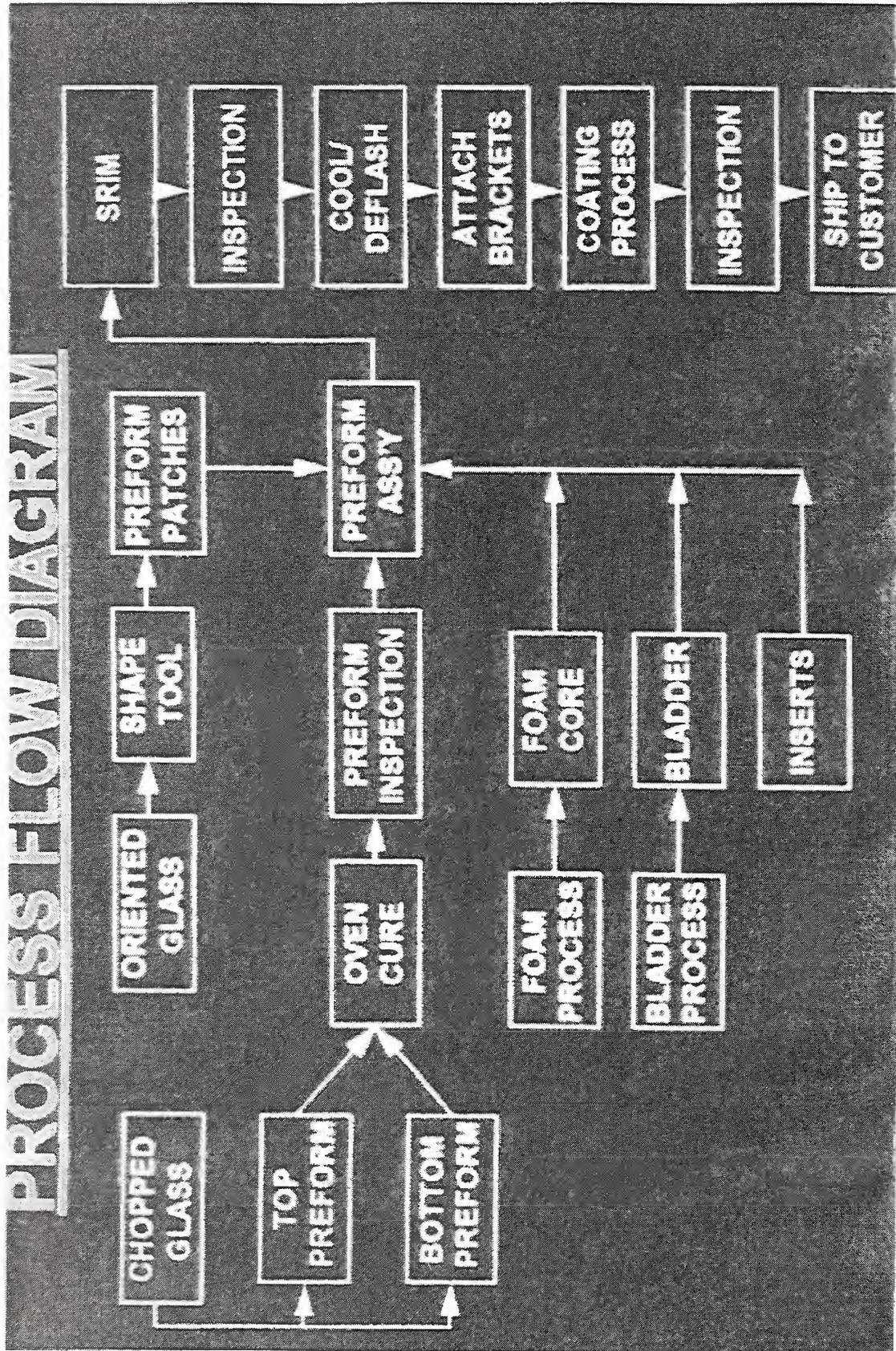


Figure 106



## **ROLE OF SENSORS IN MANUFACTURING**

- **IMPROVE PROCESS PERFORMANCE**
- **EXPAND AUTOMATION**
- **ASSURE COMPONENT QUALITY**
- **MINIMIZE VALUE ADDED SCRAP**
- **REDUCE COSTS**
- **PROVIDE DOCUMENTATION**

Figure 108



Figure 109

# SENSOR TYPES

- EQUIPMENT MONITORING
- COMPONENT MONITORING

## EQUIPMENT MONITORING

- **Pressure**
- **Vacuum**
- **Temperature**
- **Component Ratio**
- **Cure Time**

Figure 111

## **COMPONENT MONITORING**

- **Weight**
- **Void Content**
- **Glass Content**
- **Degree of Cure**
- **Dimensional Accuracy**
- **Assembly Accuracy**

# EQUIPMENT MONITORING

- Pressure
- Vacuum
- Temperature
- Component Ratio
- Cure Time

Figure 111

## **COMPONENT MONITORING**

- **Weight**
- **Void Content**
- **Glass Content**
- **Degree of Cure**
- **Dimensional Accuracy**
- **Assembly Accuracy**





