The MIT Marine Industry Collegium
Opportunity Brief #21

Nondestructive Evaluation of Fiber Composites

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NONDESTRUCTIVE EVALUATION OF FIBER COMPOSITES

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PREFACE

This Opportunity Brief and the accompanying Workshop (held on May 21, 1980) were presented as a part of the MIT/Marine Industry Collegium program, which is supported by the NOAA Office of Sea Grant, by MIT and by the more than 110 corporations and government agencies who are members of the Collegium. In this instance, the Workshop was presented jointly with the MIT Industrial Liaison Program in its series of Seminars. The underlying studies were carried out under the leadership of Professor James H. Williams, Jr., but the author remains responsible for the assertions and conclusions presented herein.

Through Opportunity Briefs, Workshops, Symposia, and other interactions the Collegium provides a means for technology transfer among academia, industry and government for mutual profit. For more information, contact the Marine Industry Advisory Services, MIT Sea Grant, at 617/253-4434.

John B. Bidwell

July 7, 1980

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1.0 A BUSINESS PERSPECTIVE

Advanced fiber composites comprise a group of modern materials that consist of fibers embedded in a matrix, which is often polymeric. These composites are inherently non-homogeneous and slight variations in the fabrication procedure can produce internal flaws and compromised structural integrity. The development of techniques for detecting such flaws is of increasing concern because of the growing use of fiber composites in a variety of applications. Fiberglass is especially widely used as the hull material of small marine craft.

Typical flaws found in fiberglass are delaminations, voids, inclusions, uncured resin, improper ratio of resin to fiber, cracks, and even omission of the glass fiber itself. Undetected, such flaws can cause hull failures. Consequently, there is a need for inexpensive quantitative, nondestructive evaluation techniques for fiberglass, as well as for other composite materials, to provide a basis for ensuring the integrity and serviceability of products made from them.

The development of such a technique could provide boat manufacturers with a useful means of inspecting hulls. The Coast Guard or other government agency could use the technique as the basis for setting and enforcing safety criteria. Insurance companies and marine surveyors could use it as the basis for insurability. In an advanced form as a consumer level kit, the technique could allow boat-yard operators and boat owners to conduct periodic re-inspection of hulls on a "do-it-yourself" basis. In summary, the economic incentives for use of an inexpensive, quantitative, nondestructive technique for evaluating the integrity of fiberglass composites would be widespread.

With Sea Grant funding, Professor James H. Williams, Jr. of MIT's Department of Mechanical Engineering has developed a non-destructive, thermal testing technique to detect flaws in fiberglass composites. Basically, the technique involves uniform application of heat to one side of a composite structure that has been coated with a cholesteric liquid crystal compound. The temperature rise causes the liquid crystal to change color. Anomalies in the pattern of surface temperature, indicative of various kinds of internal flaws, show up as anomalies in the pattern of color change in the liquid crystal coating. Professor Williams' unique contribution has been to provide a quantitative interpretation of the patterns of surface temperature and hence of the underlying flaws, as evidenced by the patterns of color change.

At the May 21, 1980 workshop of the MIT/Marine Industry Collegium, presented jointly with the MIT Industrial Liaison Program, Professor Williams discussed the thermal testing techniques as well as his work in acoustic and ultrasonic testing of fiber composites. Recent progress with these three techniques at MIT was presented in detail, including how they can be integrated into a "complete Non-Destructive Evaluation program" for fiber reinforced composite materials. Following his presentation, Professor Williams provided laboratory demonstrations of all three of these techniques.

Subsequently, Professor Williams provided a brief written Epilogue summarizing his thoughts about the Workshop which is included herein as section 5.0.

2.0 THERMAL TESTING WITH CHOLESTERIC LIQUID CRYSTALS

2.1 Thermal Testing

In thermal testing, patterns of surface temperature are used to detect nonuniformities below the surfaces of materials, to estimate the characteristics of these anomalies, and to predict resultant behavior or serviceability of the materials. An inexpensive technique for displaying variations in surface temperature is to use cholesteric liquid crystals, which change color in ambient light according to their temperature and to the formulation of the liquid crystal compound. In recent developments at MIT, the results arising from this technique can be quantitatively evaluated, and the characteristics of detected flaws can be quantitatively estimated.

2.2 Liquid Crystals

Liquid crystals are substances that have some of the properties of both liquids and crystals. One class of liquid crystals is the cholesteric, in which the molecular forces are so delicately balanced that small changes in temperature can produce striking changes in the optical properties of the substances. Some cholesteric liquid crystal compounds change from colorless through a series of bright colors back to colorless as they are heated or cooled through their cholesteric phase.

The specific characteristics of the phenomenon depend upon the formulation of the cholesteric substance. This includes the specific colors displayed and the sequence in which they occur. Likewise, the specific temperature at which each color is displayed and at which each color transition occurs, the color-play range of temperature and the rate of change from one color to another, all are invariant for any given formulation but vary from one compound or combination of compounds to another.

The phenomenon can occur in color-play ranges of temperature from 1 degree Centigrade to 50 degrees Centigrade in width, and it can occur at temperatures as low as -20 degrees Centigrade and as high as 250 degrees Centigrade. In addition, it can occur with temperature resolutions of 0.1 degree Centigrade and with color transition response times of 0.1 second.

By blending different cholesteric liquid crystal compounds in various proportions, one can achieve combinations that display various desired colors and sequences, at desired temperatures and transition points, with specified resolutions and response times. One of the aims of this research program is to find the combinations that are appropriate for purposes of thermal testing of fiberglass composites.

2.3 Detection, Estimation and Prediction

Figure 1 illustrates the concept of thermal testing. Thermal energy, uniformly applied to the surface, propagates uniformly through a structure, if the material is of uniform thickness and composition. A "hot spot" on the surface results from uneven propagation, when the flow of thermal energy encounters a flaw with thermal conductivity less than than that of the surrounding material.

Figure 2 illustrates the concept of flaw detection and recording. It shows how a "hot spot" appears as an anomaly in the pattern of color that is displayed by a coating of cholesteric liquid crystals on the surface of a structure as the surface temperature rises in thermal testing. The resulting pattern can be recorded easily with a camera.

Having detected a flaw in a material, the next step is to use the information gained to estimate the characteristics of the flaw. Research done at MIT and presented in the Workshop, has resulted in what is thought to be

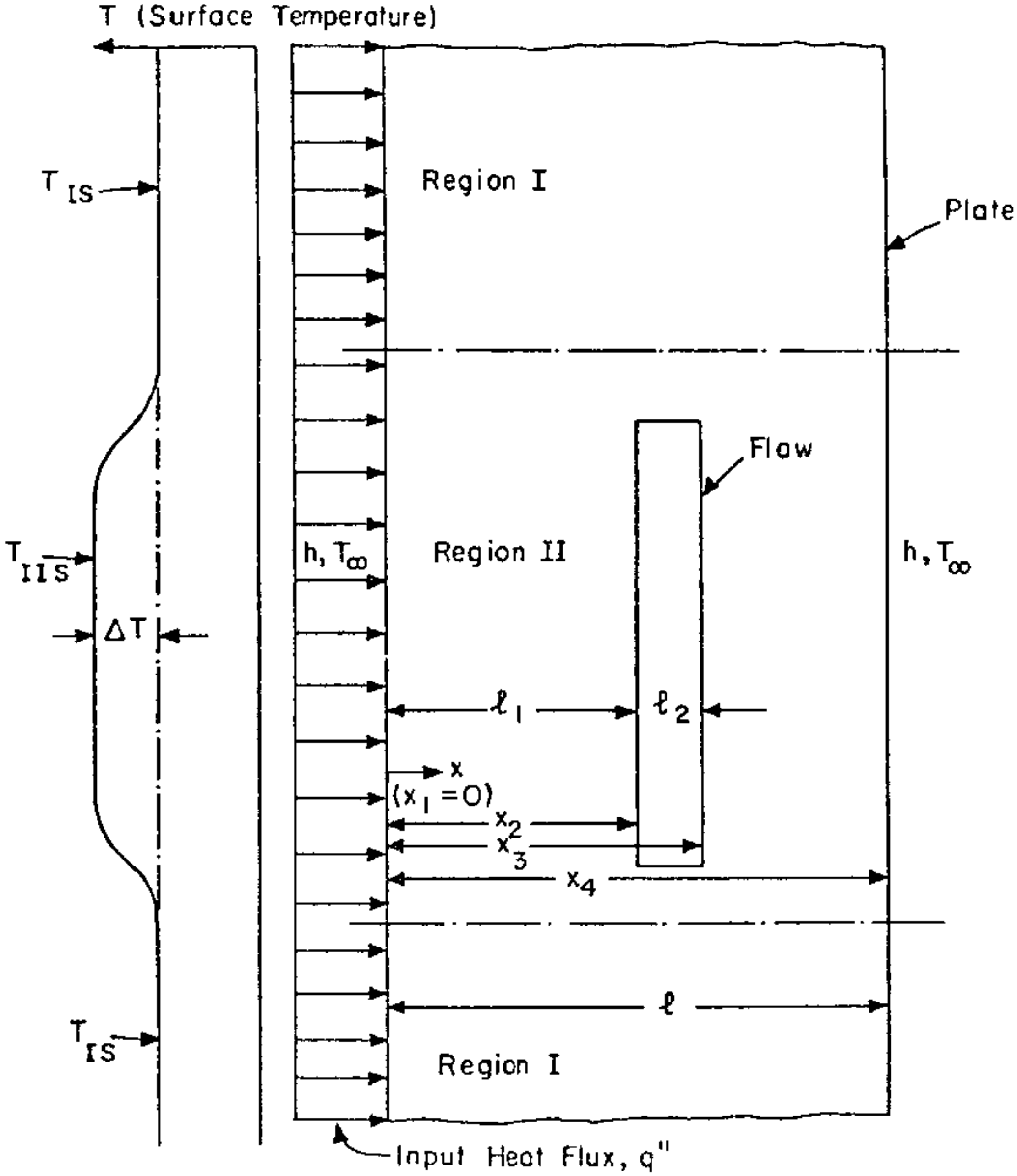

Fig. 1 Cross section of model of plate containing delamination or inclusion flaw, showing geometric and thermal variables.

[from Reference 2]

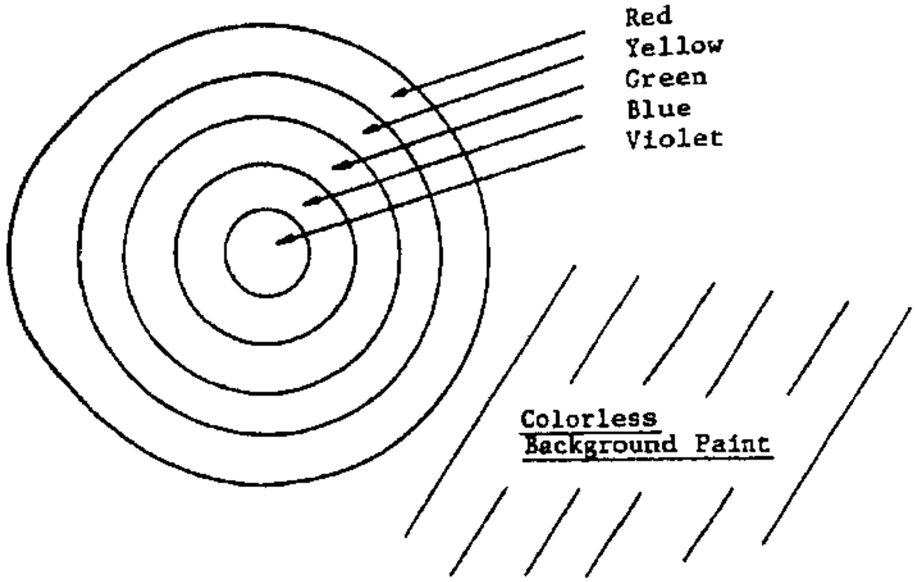

Fig. 2 Colored thermal mapping obtained from liquid crystals for a circular hot spot resulting from a defect as in Fig. 1

[from MIT Sea Grant Program Proposal for 1978-79, Vol II] the only quantitative use of the technique of thermal testing with liquid crystals to estimate characteristics of flaws in laminate materials.

In an analysis using an experimentally validated assumption of one-dimensional conduction of heat, Williams, Mansouri and Lee (Reference 4) have computed, for a wide range of parameters, the profiles of surface temperature that result from inclusion flaws in flat laminates. Further, they have defined quantitatively, a complete classification of the degree of detectability of a well-characterized inclusion flaw, making use of the invariance and repeatability of color change in liquid crystals. The quantitative parameters of liquid crystals, of the environment and the thermal input, and of the laminate and its included flaw, have been determined, which permits optimization of thermal testing using cholesteric liquid crystals.

Given the characteristics of a flaw and the computed temperature profile, a given liquid crystal can be classified in quantitative and time-dependent terms as an "optimal", "acceptable", "moderately acceptable", "limiting", or "unacceptable" detector of that flaw. Thus in the search for flaws with prespecified characteristics, one can choose blends of liquid crystals and testing conditions that will optimize the capability of detecting those flaws. Conversely, in the search for flaws with unknown characteristics, one can choose blends of liquid crystals and testing conditions that will have known capabilities for detecting various flaws. In this case the quantitative characteristics of detected flaws can be estimated from the test results obtained.

There are two problems that complicate the interpretation of test results in terms of detection and characterization of flaws. One is that composite materials, at least fiberglass composites, are inherently nonhomogeneous in composition. Thus the color changes that occur during thermal testing with liquid crystals may relate to the pattern of mats and rovings imbedded in the resin matrix and not to flaws that affect structural integrity. Similarly, under some circumstances of construction, nonuniformities in thickness of the composite material may affect color change patterns without implying the existence of flaws. The second complicating problem is that different types of flaws may have different implications for structural integrity. Figure 3 shows typical flaws that may occur in construction of fiberglass hulls for small craft.

With an estimate in hand of the characteristics of a flaw, the final step is to predict its effect on the serviceability of the material or structure. With knowledge of the relationship between test responses and flaws characterized by type and size, it should be possible to make a quantitative assessment of the structural significance of an identified flaw. What is needed is knowledge of the relationship between the type and size of an identified flaw and the residual strength of the material or structure.

To develop this knowledge, work is proceeding at MIT to develop a pressure-loading system with which fiberglass test panels can be subjected to a plate bending mode of stress from surface pressure. With this testing equipment, a number of panels will be tested to failure. The panels will be made of structural grade fiberglass with a range of fractional content of fiber by volume. They will be constructed to contain a range of types and sizes of flaws, including interlaminar voids, orthogonal cracks and in-plane cracks, with lesser emphasis on uncured resin matrix.

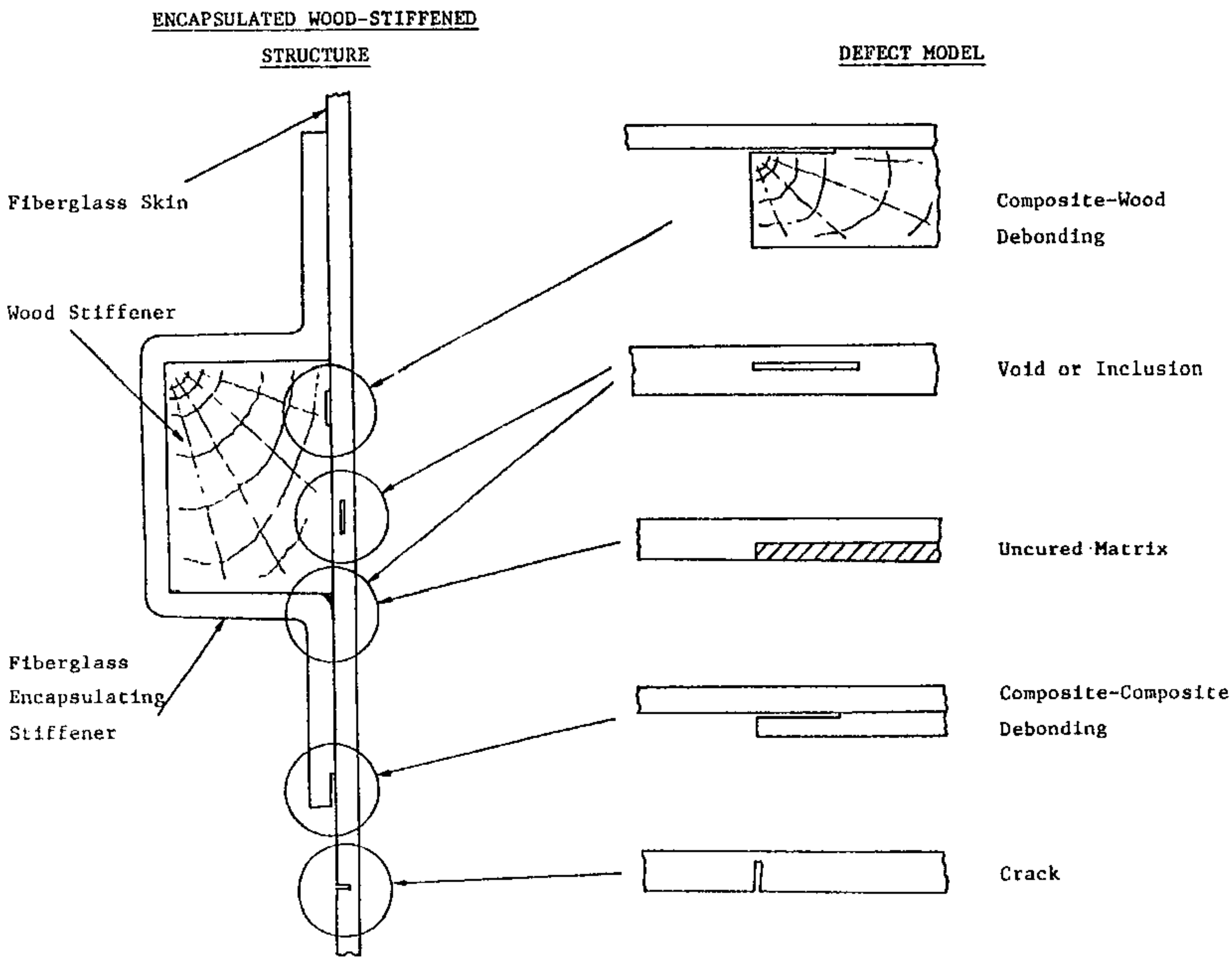

Fig. 3 Defect models for a complex structure evaluated with the liquid crystal NDE technique.
[from MIT Sea Grant Program Proposal for 1980-81, Vol II]

2.4 Results of One-Dimensional Analysis of Heat Conduction

To understand in quantitative terms what happens in thermal testing, the heat transfer and conduction problem inherent in the circumstances of the test must be modeled. This is difficult unless the geometry of those circumstances is simple. Fortunately the geometry of many testing situations can be modeled as a problem in one-dimensional conduction of heat. Figure 1 shows this geometry for a flat plate with an inclusion flaw. Reference 2 establishes the general analytical solution to the problem of one-dimensional transient heat conduction shown in the figure.

The appendix to Reference 4 lists a computer program used to generate graphs based on this analytical solution. These graphs are demonstrated to be useful for planning thermal tests for these circumstances, as well as for interpreting their results. They show the surface temperature over the flawed and unflawed regions of a test panel, and the difference in these temperatures, as a function of time for ranges of values of different variables involved.

This temperature difference between flawed and unflawed regions provides the basis for estimating the extent to which temperature differences must be resolved by a testing system if it is to be useful for the problem modeled. A small difference requires a sensitive detector and vice versa.

The analytical results also show that this temperature difference that provides the basis for detection of a flaw, passes through a maximum at some time after initiating the application of heat flux to the test surface. This point in time of maximum temperature difference is thus the optimal moment for flaw detection. Eventually the surface temperatures over the two regions, and the difference between them, tend to a steady state.

The analysis shows the temperature difference is less sensitive to variations in the depth of flaws than to variations in their thickness. This is especially the case in the steady state. Further, detection is enhanced when convective air currents that increase the heat transfer coefficient are discouraged, and when the difference between the thermal conductivities of the flaw and of the basic material is large. A large heat flux during testing is desirable as well.

The results of the analysis also permit estimation of the thickness of a flaw from the steady-state temperature difference, and estimation of its depth from the early transient values.

With these results, the testing conditions can be established for detection of flaws larger than a given size, and the flaw detection capability can be established of given testing conditions.

2.5 Results of Thermal Testing with Liquid Crystals

To determine quantitatively the effectiveness of cholesteric liquid crystals for detection of inclusion flaws, a classification scheme is proposed in Reference 3. The ability of a given liquid crystal to detect a flaw at a given instant during testing can be placed in one of five classes ranging from "optimal" to "unacceptable."

With "optimal" detection capability, the liquid crystal compound can traverse the entire color-play range of temperatures over the flawed region, before color-transition begins over the unflawed region, while at the moment of maximum temperature difference the surface temperature over the flawed region remains within the color-play range.

With "acceptable" detection capability the conditions for "optimal" capability are satisfied, except that the results are not observed at the moment of maximum temperature difference.

Under conditions of "moderately acceptable" detection capability, the magnitude of the temperature difference over flawed and unflawed regions is less than the full color-play range of temperatures of the liquid crystal. However, it still includes transition temperatures for colors that are non-adjacent in the color spectrum, so ability to discriminate color difference is still considerable.

The "limiting" condition for detection is when the temperature difference permits only two colors that are adjacent in the spectrum to appear, one over the flawed region and one over the unflawed.

Finally, if no color difference can appear within the given temperature difference, then there is no detection capability and the effectiveness of the compound for this purpose is labeled "unacceptable."

This classification scheme is useful for cataloging the results of thermal tests. It is useful for planning thermal tests when analytical estimates of surface temperatures are available for guidance.

Experiments were performed to test the one-dimensional heat conduction model and the ability to classify thermographs in accordance with the scheme developed. Figure 4 shows the experimental layout. Panels of fiberglass composite were fabricated for the tests with inclusion flaws containing air and in some instances paper. Commercially available liquid crystals were used. Heat flux was supplied by two 1,000 watt quartz-iodine lamps. Test results were recorded with a camera at various intervals, and the length of time from initiation of heat flux to taking of each photograph was recorded with a stopwatch.

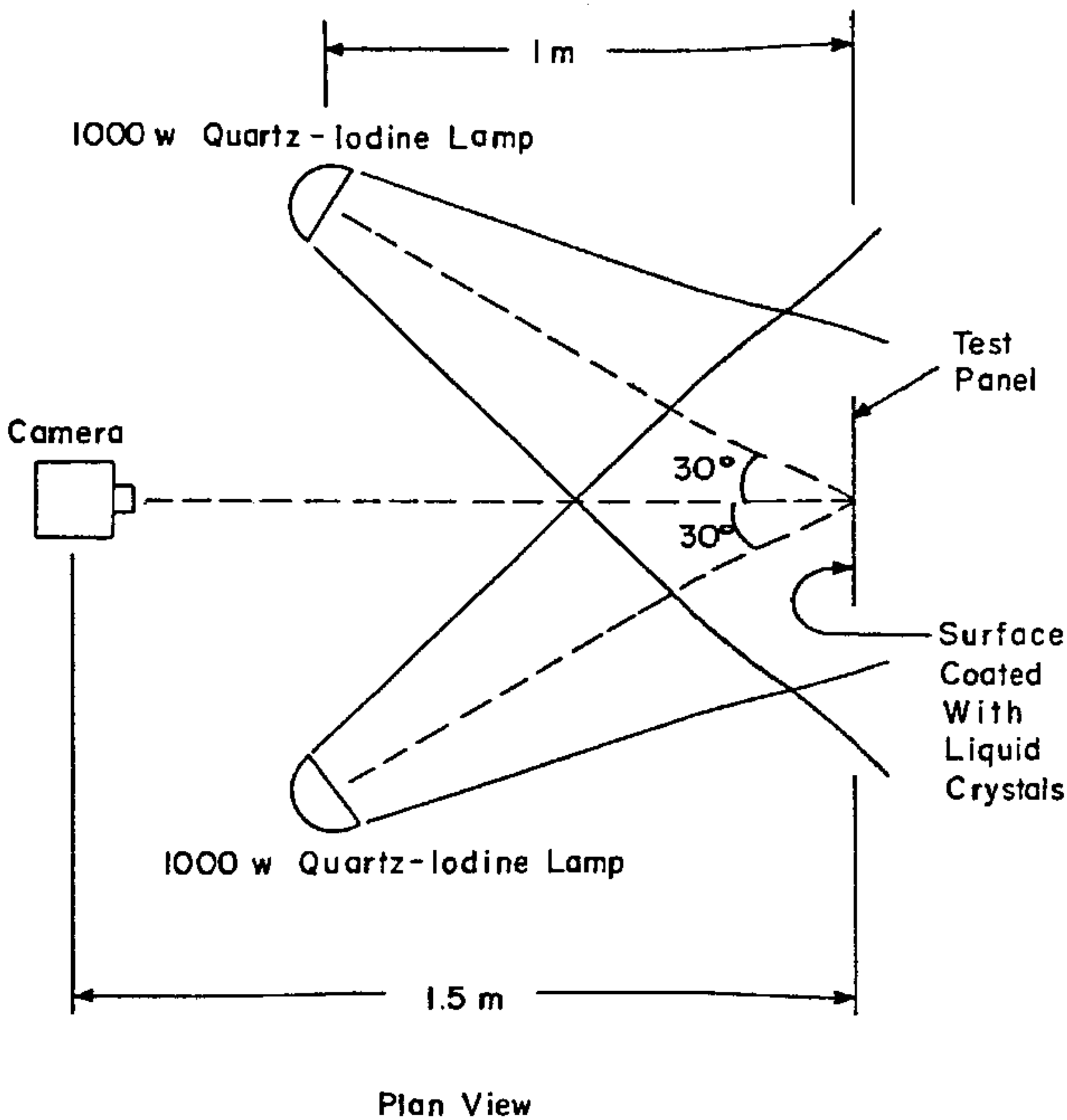

Fig. 4 Schematic of experimental system for thermal nondestructive testing of fiberglass panels using liquid crystals.

[from Reference 3]

The results of the experiments compared favorably with the results of the analytical solution derived from the one-dimensional conduction model. The model is taken to be valid when the effect of heat conduction laterally is important only over a narrow region around the edges of the defect.

By examining the colors displayed, the thermographs that resulted from the experiments were classified successfully in accordance with the scheme developed. Reference 3 includes examples of "acceptable," "moderately acceptable," and "limiting" thermographs from these experiments.

In addition, because the color-transition temperatures of the compound used are known, the surface temperature profiles across the flawed and unflawed areas could be determined from the thermographs.

3.0 ACOUSTIC EMISSION AND ULTRASONIC TESTING

When some materials are subjected to changes in stress, they produce acoustic emissions (AE). In the audible range, the phenomenon has been recognized for centuries as a signal that the material is under stress. The creaking of a wooden floor, of timbers in a ship or of wooden braces supporting a mine tunnel have long been known as real-time warnings of imminent structural failure.

Subaudible AE and ultrasonics can be used in the non-destructive testing of composite materials and metals. Professor Williams has hypothesized that the level of the elastic stresses in a metal could be assessed by introducing slight perturbing forces on the structure and then observing the "triggered" AE.

An analogy for appreciating this concept is that of a plucked violin string. A violin string containing an unknown tension when plucked in a well defined manner produces a sound related not only to the pluck but also contains information about the unknown tension. By observing the resulting musical note (AE) and by knowing the details of the plucking action (the trigger), it is possible to deduce the unknown tension (stress) state.

In AE and ultrasonic testing, sensitive transducers are attached to the material to detect low-level stress waves. The research of Professor Williams' group has lead to quantitative characterizations of these AE signals and to the development of predictive procedures for both metals and composites.

Professor Williams and Dr. Samson S. Lee, a lecturer in the Department of Mechanical Engineering, have found that metals retain a short-lived memory of the forces that deformed them, even though they appear to have returned to their original shape after the forces were removed. The magnitude of the original deformation may be deduced by measuring variations in AE produced when the metals are "interrogated" by subjecting them to a second unloading deformation. This finding may make it possible to determine quickly whether adjacent parts of a structure -- in an aircraft, for example -- need to be replaced when another part has sustained an accidental overload.

The application of ultrasonics to predict static, dynamic and fatigue characteristics of composite materials is a conceptual breakthrough in the NDE of these materials. In conjunction with Alex Vary at NASA's Lewis Research Center, Professor Williams has developed correlations between ultrasonic parameters and several strength measures. With a graduate student, Beth Doll, he has found that the ultrasonic attenuation, or diminution, of the wave amplitude about 1.5 megahertz gives an excellent indicator of the relative fatigue life of a graphite fiber composite. The correlations are significant because they have been obtained from apparently flawless materials before they are subjected to any service loading. Such a test could be an important procedure in establishing accept/reject criteria of manufactured composite components.

In a similar project with another graduate student, Norman Lampert, Professor Williams has subjected graphite fiber composites to tensile fatigue and to controlled multiple impacts by a low-velocity steel weight.

Good correlations between the number of impacts, the residual strength and the ultrasonic attenuation have been developed. These experiments provide an excellent basis for measuring the residual strength of a structure when an aircraft, watercraft or automotive structure made of a composite material has been damaged.

Designing structures and components so that they operate close to the safety limits of their materials provides economic incentives via both the savings in increasingly scarce raw materials and in the energy required to convert these materials to finished products. Automobiles, marine vehicles and aircraft produced in this manner would be lighter and thus more fuel-efficient throughout their operating lives.

Much of Professor Williams' work in acoustic and ultrasonic testing is being supported by the NASA Lewis Research Center and the General Motors Manufacturing Development Staff.

(Note: This section was adapted with permission from an article prepared for the April, 1980 issue of "MIT Reports on Research" by Robert C. Di Iorio of the MIT News Office.)

4.0 REFERENCES FOR THERMAL TESTING

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2. J.H. Williams, Jr., S.H. Mansouri and S.S. Lee, "One-Dimensional Analysis of Thermal Nondestructive Detection of Delamination and Inclusion Flaws," Composite Materials and Nondestructive Evaluation Laboratory, MIT, July 1979.

3. J.H. Williams, Jr., S.H. Mansouri and S.S. Lee, "Thermal Nondestructive Testing of Fiberglass Laminates Using Liquid Crystals," Composite Materials and Nondestructive Evaluation Laboratory, MIT, July 1979.

4. S.H. Mansouri, J.H. Williams, Jr. and S.S. Lee, "Nondestructive Detection of Delaminations and Inclusions of Fiberglass Laminates Using Liquid Crystals," MIT Sea Grant Program (DOC-NOAA), July 1979.

5.0 EPILOGUE by James H. Williams, Jr., Department of Mechanical Engineering

The emphasis of the seminar was to describe some of our work in the development of quantitative nondestructive evaluation (NDE) techniques in thermography, acoustic emission and ultrasonics. An overview of our entire research program, a general discussion of a variety of NDE techniques and their relative sensitivities, and more detailed discussions of themography, acoustic emission* and ultrasonics were presented. The lecture was concluded with a description of a "complete program in NDE" which combines materials, mechanics and NDE to assess structural integrity in both fiber composite and metallic materials and structures. Laboratory demonstrations of thermography, acoustic emission and ultrasonics were provided.

It was noted that although the specific data presented were quantitative and, to some extent, directly transferable to other applications, most of the results represented only a beginning. Yet, the results and techniques used to obtain them should provide meaningful bases for the selection of several NDE techniques and some strong indications of the kinds of quantitative answers that can be anticipated from an NDE program.

While the focus of the program was on fiber reinforced materials, the NDE techniques and concepts are equally effective for metals. In fact, the inspection of welded steel structures is expected to attract a substantial

* Because of potential copyright violations, our paper of the review of acoustic emission of fiber composites was not distributed during the seminar. The reference for this work is J.H. Williams, Jr. and S.S. Lee, "Acoustic Emission Monitoring of Fiber Composite Materials and Structures", JOURNAL OF COMPOSITE MATERIALS, Vol. 12, October 1978, pp. 348-370.

portion of our interests in the near future. Again, it must be emphasized, however, that the selection of the best NDE technique(s) for a specific application requires knowledge of the applicability, the sensitivity, the reliability and the cost of a wide range of NDE equipment and techniques.

The entire lecture could have been devoted to any one or set of the following subjects:

(1) Review of approximately eighty (80) different NDE techniques, their relative sensitivities and their relative direct costs- (However, note that such a review does not address the quantitative aspects of a "complete NDE program".);

(2) Discussion of business opportunities primarily relating to the exploitation (sale) of NDE services - (New entries into the current highly competitive equipment manufacturing markets are not likely to be as profitable.);

(3) Discussion of the costs of the NDE/No NDE product life-cycle decision tree;

(4) Presentation of linear and nonlinear, elastic and inelastic, static and dynamic, fracture mechanics of composites and metals;

(5) Emphasis on the mechanics-materials-NDE interrelationships in the "complete NDE program" to assess structural integrity, and one or two specific examples in which the knowledge of mechanics, materials and NDE were combined to provide an accept/reject decision; or

(6) Review of the state of the art in the NDE-fracture mechanics analyses of welded structures.

BUT, this was a seminar about our efforts to develop the quantitative aspects of the NDE of fiber composites.

We appreciate your comments and recommendations and hope that you will take the initiative to follow up some of them. While I am heartened by the possiblity of our involvement with a number of your companies in joint efforts relating to specific problems, my sole regret is that I did not meet more of you and learn more about your specific concerns, current problems and potential interests. However, the future remains. Keep in touch.