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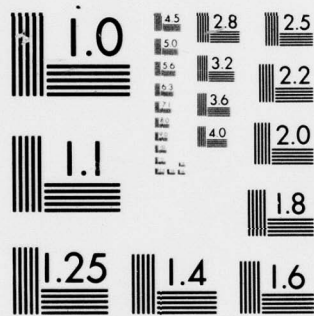
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ENVIRONMENTAL EFFECTS ON THE MECHANICAL PROPERTIES OF GLASS FIBER/EPOXY RESIN COMPOSITES, Effect of Static Immersion in Water on the Tensile Strength of Crossply Laminates,

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BYUNG LIP/LEE, ROBERT W./LEWIS, ROBERT E./SACHER,  
ARMY MATERIALS AND MECHANICS RESEARCH CENTER  
WATERTOWN, MASSACHUSETTS 02172

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## INTRODUCTION

The use of glass fiber-reinforced resin composites in primary structures for Army applications has been on the increase in recent years. They are being, or will be, employed in load-bearing areas in several rotor blade systems - the CH-47 modification, the Advanced Attack Helicopter, the Blackhawk, and the AH-1Q Cobra Improved Main Rotor Blade - as well as being used in critical areas in the VIPER system. Despite this projected usage, the questions on the stability of the material properties upon exposure to outdoor environments remain largely unanswered (1,2). The outdoor weathering conditions are extremely complex and include such factors as temperature, electromagnetic radiation (for example, sunlight), and moisture. These are considered to be the major elements responsible for the degradation of materials in the atmosphere (2,3).

This study is directed at determining how and why the mechanical properties of fiber-reinforced resin composites are affected by exposure to each of the above-mentioned elements with different duration, intensity, and frequency. In this phase of the study, the effect of moisture at ambient temperature without radiation is being investigated. Since precipitation in the form of rain or dew is the most common source of moisture in the outdoor environment, immersion in distilled water was chosen as the first exposure condition to be studied. Among a wide variety of mechanical properties, the uniaxial tensile strength of the laminate under constant-rate deformation was chosen mainly because of availability and simplicity of the testing technique.

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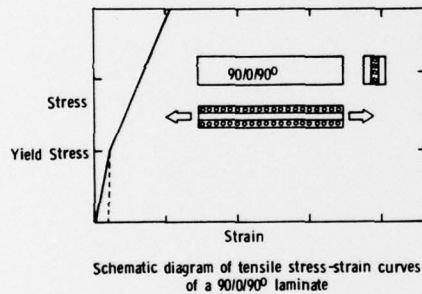
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The techniques and results developed in this work will be eventually translated to the actual systems under consideration for helicopter rotor blades.

A typical composite laminate may be built up with the fibers of each lamina oriented in a different direction. When a laminate contains laminae with different fiber orientation and is subjected to uniaxial tension, individual laminae will fail successively with increasing order of failure strain in the given direction. In a cross-ply laminate, the lamina with its reinforcing fibers aligned perpendicularly to the load direction, i.e., the  $90^\circ$  lamina, will fail first, thus reducing its share of load. The burden of load transfer will be increasing in the unbroken portion of the laminate (4). As a consequence, the "knee" or "yield" point appears in the stress-strain curve due to the reduction of laminate modulus (Figure 1a). Although the term "yield point" will be used hereafter based on the analogy to that in metals, underlying deformation mechanisms are entirely different. What happens in the composite laminate at this point is micro-failure followed by the formation of translamellar crack, not plastic flow or



Schematic diagram of tensile stress-strain curves of a  $90/0/90^\circ$  laminate

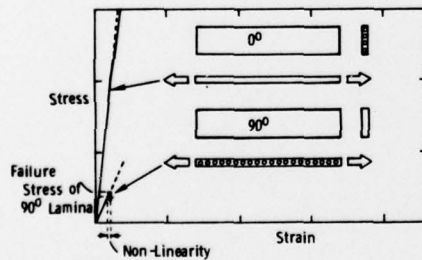
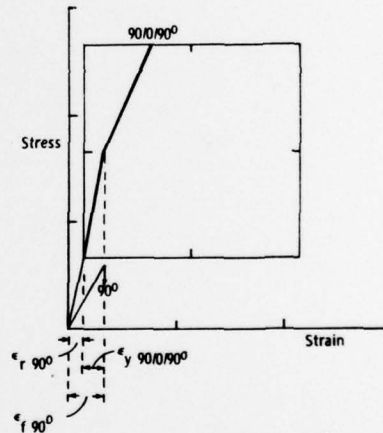


Figure 1a. Schematic diagram of tensile stress-strain curves of a  $0^\circ$  lamina, a  $90^\circ$  lamina and a  $90/0/90^\circ$  laminate.



- $\epsilon_f 90^\circ$  - failure strain of  $90^\circ$  lamina without the restraint of  $0^\circ$  lamina
- $\epsilon_r 90^\circ$  - prestrain in the  $90^\circ$  lamina due to the interlaminar residual stress
- $\epsilon_y 90/0/90^\circ$  - yield strain of  $90/0/90^\circ$  laminate (due to the failure of  $90^\circ$  lamina)

Figure 1b. Approximate relationship between the yield strain of a  $90/0/90^\circ$  laminate and the failure strain of a  $90^\circ$  lamina (without the restraint of a  $0^\circ$  lamina).

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dislocation motion. The yield point is usually far below the catastrophic failure point (which determines ultimate strength or strain) of the laminate. But it is very important in the design concept, since the "design ultimate stress" is related to the maximum laminate stress attainable without the rupture of any lamina (5).

The yield stress or strain is also important from the viewpoint of resistance to water absorption, because the occurrence of cracks for whatever reason will allow water absorption of composites by capillary or conduction mechanisms. Therefore, the absorption of water into the composites can be accelerated at the yield point, possibly resulting in further deterioration of the properties. Considering these facts, the effect of water absorption on the tensile strength of crossply (90/0/90°) laminates will be examined with particular emphasis on the yield point.

#### EXPERIMENTAL PROCEDURE

Three types of glass fiber/epoxy resin composites have been used in this phase of the study. In this report, discussion will be restricted to the data based on E glass/Scotchply 1009 resin system. The E glass/1009 resin laminates were prepared by a commercial manufacturer (3M, St. Paul, MN). The following conditions were used in their curing and postcuring process: cure at 163 C (325 F) for 45 minutes under pressure of 50 psi, postcure at 177 C (350 F) for 4 hours under contact pressure. The laminate supplied consists of three crossplied laminae (lamina thickness  $\approx 0.016$  inch). The laminate has a density of  $2.01 \pm 0.02$  gram/cc and  $74.9 \pm 1.2$  weight percent fiber (6,7).

Three sets of specimens (34 in.  $\times$  6-1/2 in.  $\times$  thickness) were used in the water absorption experiments:

- (1) three-ply tension test specimens with the direction of fiber on the outermost lamina perpendicular to the specimen axis (90/0/90°),
- (2) single-ply tension test specimens (0° or 90°), and
- (3) warped two-ply specimens (0/90°).

The following procedure was used for measuring the change in the tensile properties of 90/0/90° laminate by water absorption.

- (1) The 90/0/90° specimens were cut from the plate and completely dried at 50 C under vacuum.
- (2) The dried specimens were immersed in distilled water at 23 C for various periods of time.

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(3) After the moisture was removed from the specimen surface by soft paper tissue, the weight gain of each specimen was measured.

(4) The specimens having a gage length of 4.5 in. were tested in an Instron tester at a crosshead speed of 0.05 in./min.

(5) Yield stress and strain were defined by the position of the knee in the stress-strain curve and ultimate strength by the catastrophic failure point. The tangent modulus of elasticity was estimated from the slope of the stress-strain curve.

The  $90^\circ$ ,  $0^\circ$ , and  $0/90^\circ$  specimens were prepared by removing unnecessary lamina(e) from the  $90/0/90^\circ$  specimens which had been immersed in water for various periods of time. Since the removal of unnecessary lamina was performed by a sharp razor blade within 10 minutes, its effect on the water retention of the specimens was assumed negligible. The  $90^\circ$  and  $0^\circ$  specimens were used in the evaluation of strength and modulus of each lamina. The curvature of  $0/90^\circ$  specimens was estimated from the values of height and span, assuming that the arc of warped specimens was circular. All measured properties were plotted against the weight gain due to water absorption.

#### RESULTS AND DISCUSSIONS

The weight gain of  $90/0/90^\circ$  specimens due to water absorption is shown as a function of immersion time in Figure 2. It clearly shows that equilibrium has not been reached at 2500 hours of water immersion at ambient temperature.

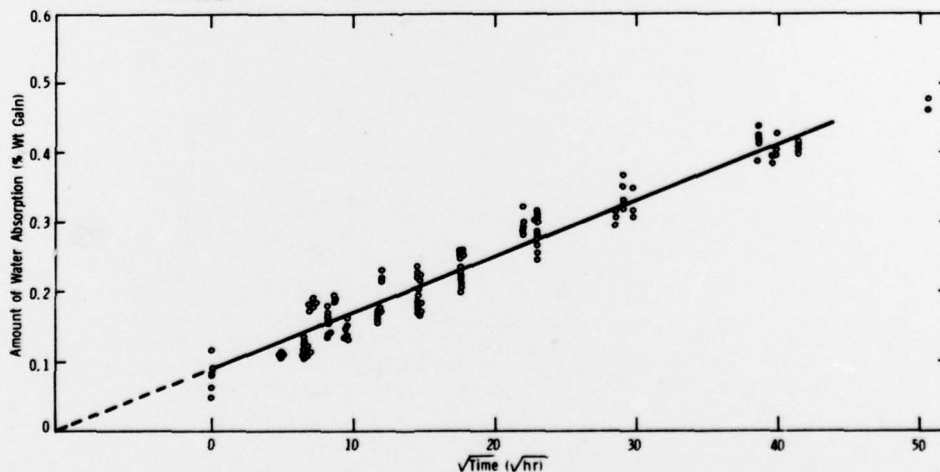


Figure 2. Amount of water absorption of a  $90/0/90^\circ$  laminate versus the immersion time at room temperature.

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The results of tension tests show that, at room temperature, water absorption affected the level of both yield stress and ultimate strength of 90/0/90° laminates to a considerable degree. Ultimate strength decreased continuously with an increasing amount of water absorption (Figure 3). Yield stress increased initially and then decreased, thus to form a broad maximum around the point of 0.2% weight gain (Figure 4). On the other hand, no significant change was observed in the values of moduli before and after yielding due to water absorption (Figure 5).

As shown earlier, the yield stress is very important from the viewpoint of both the design limit of the material and the resistance

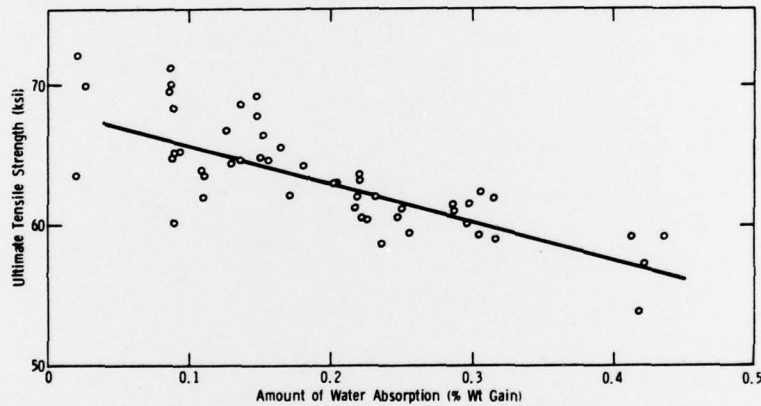


Figure 3. Effect of water absorption on the ultimate tensile strength of a 90/0/90° laminate.

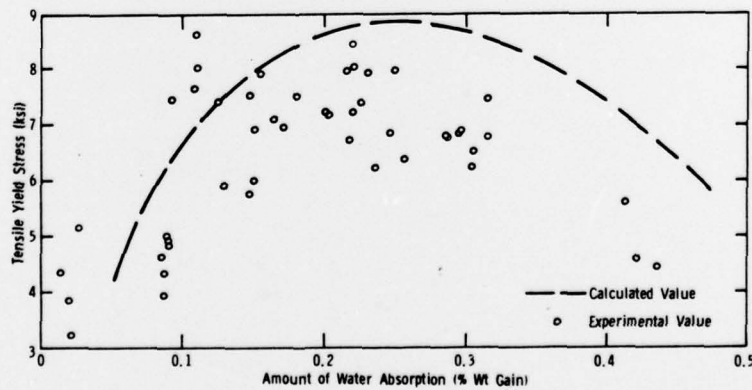


Figure 4. Effect of water absorption on the tensile yield stress of a 90/0/90° laminate.

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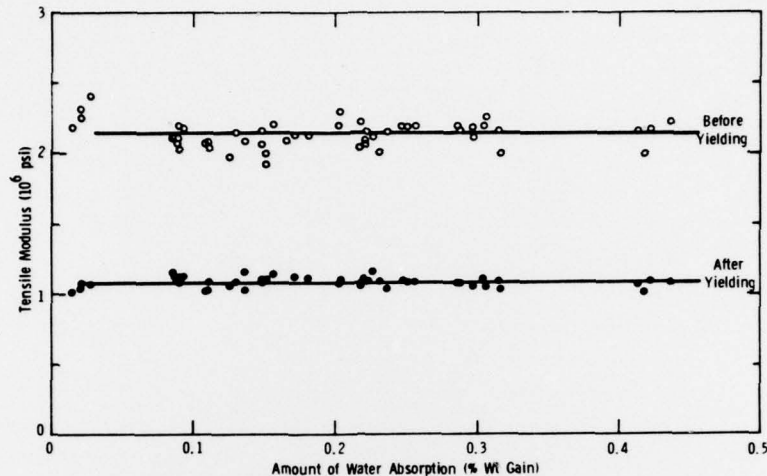


Figure 5. Effect of water absorption on the tensile modulus of a 90/0/90° laminate.

to water absorption. Considering this, discussion will be directed to the effect of water absorption on the yield stress. Since the yield stress is closely associated with the failure of the 90° lamina, discussion will start with the description of the deformation mechanism of 90/0/90° laminates in uniaxial tension.

Compared to other angle-ply laminates (8), 90/0/90° laminate exhibits a relatively simple failure pattern under uniaxial tension. As the laminate is deformed, all laminae which have been prestrained due to interlaminar residual fabrication stresses are subjected to the same external (in-plane) strain. In the initial stage of loading, the whole laminate deforms linearly. With continued loading, the strain (sum of prestrain and external strain) reaches a critical level and a translaminar failure by cracking parallel to fibers occurs in the 90° lamina. The failure of 90° laminae reduces their share of load and transfers it to the unbroken portion of the laminate. As a consequence, the stress-strain curve showed a marked reduction of laminate modulus above the yield point (Figure 1a).

During the testing it was observed that the successive occurrence of translaminar cracks (accompanied by acoustic emission) started in the 90° laminae slightly past the yield point. Delamination was rarely observed at the junction between the interlaminar region and the translaminar crack. When the crack density became relatively high in the 90° laminae, cracks started to form also in the 0° lamina. After "whitening" of all laminae by densely populated cracks, the laminate continued to deform until catastrophic failure occurred across

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the fibers in the  $0^\circ$  lamina. At catastrophic failure, extensive delamination took place between the  $0^\circ$  and the  $90^\circ$  laminae. The whole failure pattern described above was not affected by water absorption despite the change in the level of yield stress and ultimate strength.

Based on the observed behavior, it can be assumed that the yielding of the  $90/0/90^\circ$  laminate is governed primarily by the failure of  $90^\circ$  lamina. Since the  $90^\circ$  lamina is prestrained by the interlaminar residual stress and strained externally by the applied load with the restraint of the  $0^\circ$  lamina, the following properties play a dominant role in the determination of yield strain of the laminate:

- (1) failure strain of unrestrained  $90^\circ$  lamina, i.e., transverse ultimate strain of unidirectional lamina,
- (2) degree of bonding between  $90^\circ$  and  $0^\circ$  laminae (which controls the degree of load transfer between the laminae), and
- (3) prestrain in the  $90^\circ$  lamina due to the interlaminar residual stress.

If the interlaminar bonding is strong enough to preclude delamination before the failure of  $90^\circ$  lamina, the yield strain can be approximated as a difference between (1) the failure strain of  $90^\circ$  lamina without restraint and (2) the prestrain in the  $90^\circ$  lamina, assuming negligible Poisson's ratio effects (Figure 1b).

Therefore, the possible change in each of the above-mentioned properties must be investigated to explain the change of yield strain (or stress) by water absorption. So far the effect of water absorption on the interlaminar residual stress and the failure stress of  $90^\circ$  lamina has been examined by separate experiments. The results are presented in the following three sections.

#### A. Effect of Water Absorption on the Interlaminar Residual Stress of $90/0/90^\circ$ Laminate

When a crossply laminate made up of  $0^\circ$  and  $90^\circ$  laminae is cooled down from the cure temperature to room temperature,  $90^\circ$  laminae having higher thermal expansion coefficients try to shrink more than  $0^\circ$  laminae along the  $0^\circ$  direction. If the same dimensional contraction is dictated by strong bonding between the laminae, each lamina will be in a state of interlaminar residual stress. Although the isothermal contraction of each lamina due to the resin shrinkage in the curing process can influence the residual stress, its effect is probably negligible because of stress relaxation at the high curing temperature

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(e.g., 163 C in E glass/1009 resin system); the interlaminar residual stress results mainly from the restraint against the differential contractive strain of  $0^\circ$  and  $90^\circ$  laminae during the cooling.

Interlaminar residual stress can be partially relieved by the distortion (warpage) of the laminate (9,10). In actual crossply laminates, the distortion is avoided by mid-plane symmetry (e.g.,  $90/0/90/0/90/0/90^\circ$ ). In this case the distortional forces exerted by all individual laminae will be balanced and no out-of-plane warping will occur. Therefore, in-plane shear stresses cannot occur in the individual laminae (11), and only normal stresses are possible:  $90^\circ$  lamina in tension and  $0^\circ$  lamina in compression along the  $0^\circ$  (load) direction. No warpage was in fact observed in the  $90/0/90^\circ$  symmetric laminates used in this study.

In this work, the differential contractive strain of  $0^\circ$  and  $90^\circ$  laminae was estimated two-dimensionally by measuring the degree of warpage in an unbalanced laminate. In order to obtain unbalanced  $0/90^\circ$  laminate specimens, the top laminae were removed from the  $90/0/90^\circ$  laminate specimens which had been immersed in water for various periods of time. The curvature of each  $0/90^\circ$  specimen was measured assuming the arc of the now warped specimen to be circular. The results showed that the curvature of  $0/90^\circ$  specimens decreased in a nearly linear fashion with increasing amount of water absorption (Figure 6).

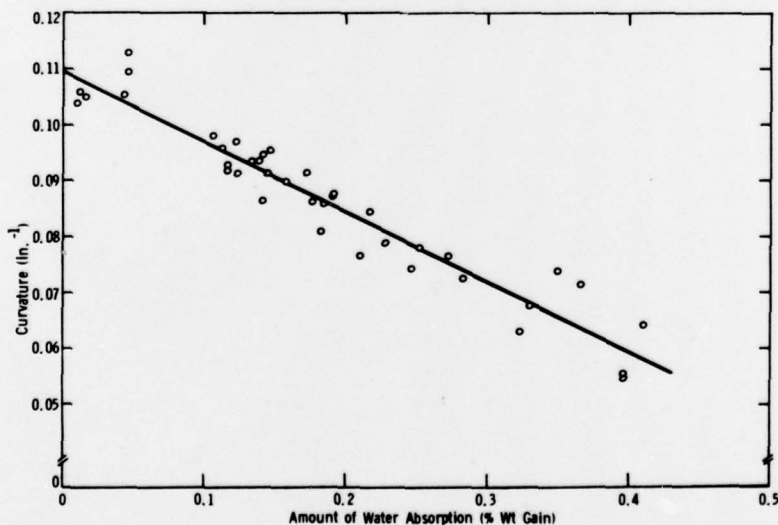


Figure 6. Effect of water absorption on the curvature of a  $0/90^\circ$  laminate.

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There have been several equations derived to relate the curvature of unbalanced laminates to the differential thermal strain of the constituent laminae (9,10,12,13). The simplest of this is Timoshenko's bimetallic thermostat equation which was derived from the strain energy consideration (12). Although this equation ignores the local shear interaction between the laminae, it is shown to agree reasonably well with the more rigorous solutions (13) for a very thin laminate (with the thickness-to-length ratio less than 0.05). Since the 0/90° specimens of this study have a thickness-to-length ratio less than 0.005, Timoshenko's equation was used in the calculation of differential strain of 0° and 90° laminae (14). The following assumptions were made:

- (1) linear elastic behavior of 0° and 90° laminae,
- (2) perfect bonding between the laminae, and
- (3) negligible Poisson's effect.

The calculation of differential strain requires values of tensile moduli of 90° and 0° laminae. The modulus of each 90° and 0° lamina which was removed from the immersed laminate specimens was measured and found to be relatively unchanged by water absorption up to the point of 0.4% weight gain (Figure 7). Using the average values of moduli of 90° and 0° laminae, the differential strain ( $\Delta l/l_0$ ) of 0° and 90° laminae was calculated from the curvature of 0/90° specimens. Figure 8 shows that the differential contraction of 0° and 90° laminae due to cooling decreased linearly with increasing amount of water absorption. This phenomenon could be explained by one or more of three factors.

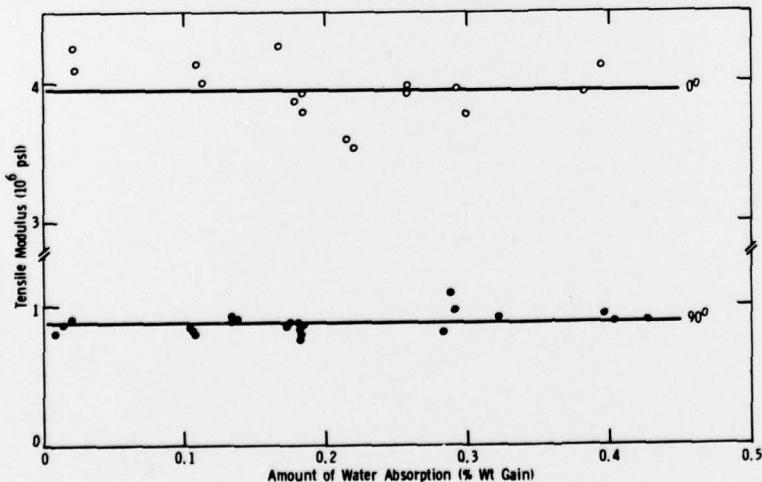


Figure 7. Effect of water absorption on the tensile modulus of 0° and 90° laminae.

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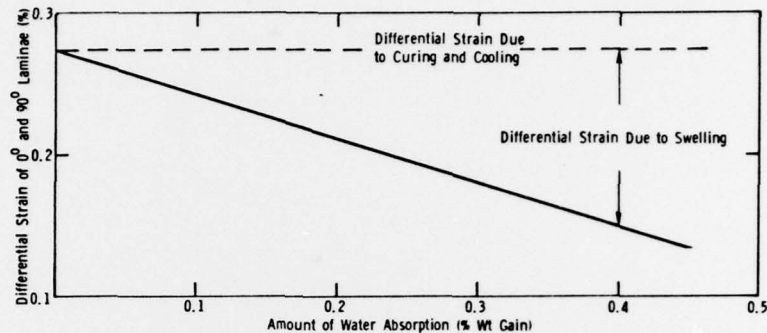


Figure 8. Effect of water absorption on the calculated differential strain of  $0^\circ$  and  $90^\circ$  laminae.

(1) Reduction in the modulus of  $90^\circ$  lamina due to the "plasticization" of matrix resin by water absorption. (Water molecules can disrupt intermolecular bonding forces of resin thus shifting its transition temperatures.)

(2) Reduction in the degree of interlaminar bonding.

(3) Development of differential expansive strain of  $0^\circ$  and  $90^\circ$  laminae by swelling of matrix resin.

The first factor can be dismissed based on the data which showed no significant change in the modulus of  $90^\circ$  lamina by water absorption (Figure 7). Presumably, the reduction in the glass transition temperature of matrix resin by water absorption has a negligible effect on the value of room temperature modulus up to the point of 0.4% weight gain (1.6% weight gain in the resin phase assuming no water absorption of other components).

In the unbalanced laminate, general or intermittent loss of interlaminar bonding can partially relieve interlaminar residual stress, thereby resulting in a lower degree of warpage. Loss of interlaminar bonding by water absorption is theoretically possible through debonding between the fiber (of  $0^\circ$  lamina closest to the interlaminar region) and resin at the interlaminar region. However, with weaker interlaminar bonding, it is also possible for the cracks to propagate more easily along the interlaminar region under external force. In the laminate system reported here, no increasing tendency toward coupling between the delamination and the translaminar crack formation could be observed with increasing amounts of water absorption. This fact discredits indirectly a possible contribution of the second factor to the reduction of calculated differential strain of the  $0^\circ$  and  $90^\circ$  laminae.

With present information, the third factor is most likely to explain the reduction of differential contraction. Ishai observed anisotropic volume expansion behavior of unidirectional composites by water absorption (15). According to his results, a glass fiber/epoxy resin lamina shows a considerable volume expansion perpendicular to the fiber direction by water absorption. Likewise, the same lamina shows a negligible amount of volume expansion along the fiber direction. Based on his data, it can be postulated that the differential expansion of 0° and 90° laminae due to swelling has the opposite effect on the differential contraction due to cooling.

Without taking into account the differential expansion due to swelling observed by Ishai, the reduction of interlaminar residual stress arising from the reduction of differential contraction of 0° and 90° laminae by water absorption can be estimated. The interlaminar residual stress was calculated two-dimensionally using a simplified model by Thompson (14,16). His analysis showed that the interlaminar residual stress in the 90° lamina along the 0° direction (as an integrated average value through the lamina thickness) is given by:

$$\sigma_{r\ 90^\circ} = [(\Delta l/l_0) E_{90^\circ}]/1 + (E_{90^\circ} A_{90^\circ}/E_{0^\circ} A_{0^\circ})$$

( $\Delta l/l_0$  = differential strain of 0° and 90° laminae, E = modulus, A = cross-sectional area). The tensile prestrain ( $\sigma_{r\ 90^\circ}/E_{90^\circ}$ ) in the 90° lamina could be calculated from the interlaminar residual stress ( $\sigma_{r\ 90^\circ}$ ) in a straightforward manner. The tensile prestrain in the 90° lamina calculated as above showed a linear decrease with increasing amount of water absorption. It reflects clearly the relaxation of interlaminar residual stress by water absorption.

#### B. Effect of Water Absorption on the Failure Stress of Unrestrained 90° Lamina

Under uniaxial tension, the 90° lamina specimen deforms linearly in the initial stage; however, it later exhibits nonlinear deformation before final failure (Figure 1a). The results based on testing of 90° lamina removed from the laminate show that the magnitude of nonlinearity increased substantially by water absorption (Figure 9). On the other hand, the failure stress (or strain) of 90° lamina showed a broad maximum around the point of 0.2% weight gain, as in the case of yield stress of 90/0/90° laminate. The complete explanation of these phenomena will comprise future work ("The Effect of Static Immersion in Water on the Tensile Strength of a Lamina").

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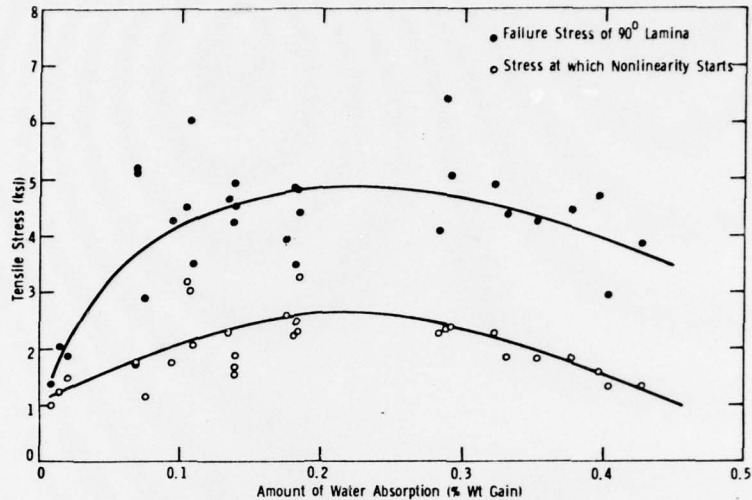


Figure 9. Effect of water absorption on the failure stress of a 90° lamina without the restraint of a 0° lamina.

#### C. Prediction of the Change in Yield Stress of 90/0/90° Laminate Induced by Water Absorption

In the two previous sections, the effect of water absorption on the interlaminar residual stress of 90/0/90° laminates and the failure stress of unrestrained 90° lamina was discussed. Based on a two-dimensional model, the tensile prestrain in the 90° lamina due to the interlaminar residual stress was predicted to decrease continuously with increasing amount of water absorption. The change in tensile failure stress or strain of 90° lamina without the restraint of 0° lamina by water absorption was also observed. The next task is to predict the change in yield stress of a 90/0/90° laminate based on the above information.

As shown in the earlier discussion, if the interlaminar bonding is perfectly strong (no delamination before lamina failure), the yield strain of 90/0/90° laminate can be approximated as  $\epsilon_f 90^\circ - \epsilon_r 90^\circ$ , assuming negligible Poisson's effect (Figure 1b). The yield stress of 90/0/90° laminate can be obtained from the values of  $\epsilon_f 90^\circ$ ,  $\epsilon_r 90^\circ$ , and  $E_{90/0/90^\circ}$  (Figure 10). Although the values of  $\epsilon_f 90^\circ$  could be estimated directly from the stress-strain curve, the calculated values of  $\sigma_f 90^\circ / E_{90^\circ}$  were used instead to minimize the effect of nonlinearity.

The calculated change in yield stress of 90/0/90° laminate by water absorption is shown as a dotted line in Figure 4. While based on limited data, the prediction of the change in yield stress is relatively

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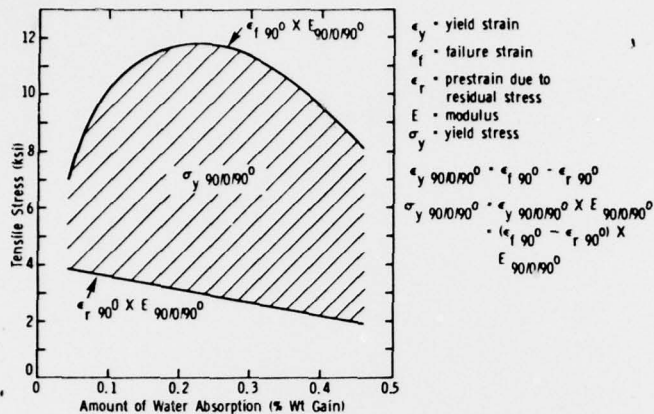


Figure 10. Effect of water absorption on the calculated yield stress of a 90/0/90° laminate.

close at lower amounts of water absorption. But with greater water absorption, the experimental data points deviate from the prediction somewhat. Aside from the experimental errors (weight gain, curvature of 0/90° specimens, failure stress of 90° lamina, etc.), two factors are considered to be responsible for the deviation:

- (1) higher degree of nonlinearity in the stress-strain curve of 90° lamina with increasing amounts of water absorption, and
- (2) possible change in the Poisson's ratio of 90° lamina by water absorption.

#### CONCLUDING REMARKS

This report describes studies to determine the effect of water absorption on the tensile strength of 90/0/90° laminates. The ultimate strength was found to decrease continuously with increasing amounts of water absorption. Yield stress first increased and then decreased to form a broad maximum. Investigation focused on the question of how and why the yield stress shows a maximum at a certain amount of water absorption.

Based on the curvature measurements of unbalanced laminates and the two-dimensional theoretical model, a prediction was made of the relaxation of interlaminar residual stress in the laminate by water absorption. The change in failure stress of an unrestrained 90° lamina arising from water absorption was also observed experimentally. Assuming perfect bonding between the laminae, the change of yield stress could be predicted by overlapping the decrease of interlaminar residual stress (as a prestress) and the change of failure stress of 90° lamina.

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It was found that the relaxation of interlaminar residual stress by water absorption does not result from the reduction of modulus of 90° laminae. Presumably, the reduction in the glass transition temperature of the matrix resin by water absorption (plasticization) has negligible effects on the value of room temperature modulus. Anisotropic volume expansion of the laminae by water absorption is considered to be responsible for the relaxation of interlaminar residual stress.

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