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ENVIRONMENTAL EFFECTS ON THE MECHANICAL PROPERTIES OF GLASS FIBER/EPOXY RESIN COMPOSITES

Part I. Effect of Static Immersion in Water on the Tensile Strength of Cross-Ply Laminates

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ABSTRACT

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. 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. 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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INTRODUCTION

The effect of exposure to outdoor environments on the mechanical properties of fiber-reinforced resin composites has been of great concern during the past few years.[1-4] 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.[4,5]

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.

The effect of moisture on the mechanical properties of fiber-reinforced resin composites has been observed by numerous investigators. Experimental studies have shown that moisture absorption affects the strength, stiffness, and toughness of composites in tension, compression, bending, or shear to a considerable degree.[6-21] However, many of these previous studies have been devoted to determining the sensitivity to moisture of each particular material system without developing a satisfactorily clear picture on the mechanism of property change. The main reasons for this are:

(a) complexity of mechanical response of composites; and

(b) uncertainty in the mechanism of moisture absorption in composites.

Before discussing these factors, a review of the structure of composites, specifically continuous glass fiber-reinforced epoxy resin composites is in order. For structural applications in beams, plates, shells, sandwiches, etc., a laminate of continuous fiber-reinforced resin composites is built up with the fibers of each constituent lamina typically oriented in a different direction. In each lamina, closely packed and parallel fibers are embedded in the matrix resin, thus forming a relatively wide interfacial region. For good interfacial bonding, the resin must fill every hill and valley of the microscopically rough surface of the fibers. In order to improve the interfacial bond, a finish (or coupling agent) is usually applied to the glass fiber. Ideally, the finish should be deposited as a continuous layer on the fiber. However, it actually consists of microscopically discrete globules leaving substantial parts of the fiber surface exposed to the other medium (e.g., resin, air, or water).[22] Most composites in practical use contain various types of voids which are caused by entrapped air or volatile components of resin during the processing.[23,24]

At the molecular level, each component of the lamina, i.e., fiber, resin, or finish, has its own complicated structure. The glass fiber is considered a mixture of metal oxides dispersed in a silica network.[25] Its surface composition shows a higher concentration of polarizable ion than the average bulk composition.[22] The epoxy resin consists of micelles or granules of highly cross-linked regions separated by regions of low molecular weight material.[26] Even the silane finish is not a simple layer or globule of monomers. It was found to be composed of a polysiloxane network along with low molecular weight materials and other impurities.[25] The silane finish possesses functional groups which can react theoretically with the fiber or resin, and there are many theories on the molecular interaction between the components at the interface.[22,27-30] Each theory from the "covalent bonding" theory to the "physical friction" theory has its own merits and some supporting evidence, but none explains all of the data.

Up to this point, the structure of fiber-reinforced resin composites has been briefly examined at the level of laminate, laminae, components, and finally molecules. The complexity of mechanical response of composites stems from behavior of this multi-layered structure. For instance, the mechanical properties of the laminate are related to the properties of the constituent laminae through the specification of individual lamina thickness, fiber direction, stacking sequence, and interlaminar bonding strength.[31] By varying these properties, the mechanical properties of the laminate can be "tailored" to the specific design requirements of the structural element.

One complicating factor in the mechanical behavior of laminates is the presence of internal residual stresses. The principal source of this stress in the laminate is thermal incompatibility of the various laminae due to the dependence of thermal properties on the fiber direction.[32-35] When a laminate is made up of laminae with different fiber directions and cooled down from the stress-free cure temperature to room temperature, the laminae with the higher positive thermal expansion coefficient in a given direction try to shrink more than the other laminae. If the dimensional contraction is constrained by strong bonding between the laminae, each lamina of the laminate will be in a state of residual stress. Hereafter, this stress will be referred to as the "interlaminar residual stress." The interlaminar residual stress has a certain influence upon the mechanical properties of each lamina as well as the mechanical interaction between the laminae.[33,36]

In the discussion on the mechanical properties of the laminate, the properties of the lamina are tacitly conceived as average bulk properties in a given direction. In reality, the mechanical properties of the lamina are related to the bulk properties of components through the specification of (a) the volumetric content ratio of components, (b) geometry of fiber packing (mode of packing, interfiber spacing, and fiber diameter), and (c) intralaminar bonding strength at the fiber/resin interface.[31] Here the residual stress develops in each component of the lamina, since the resin attempts to shrink more than the fiber during the processing.[32,37-40] This stress will be called the "intralaminar residual stress." Classical elasticity formulations show that the intralaminar residual stress is highly dependent on the position around the fiber circumference, the interfiber spacing, and the mode of fiber packing.[39,40] The intralaminar residual stress will affect the mechanical properties of each component as well as the mechanical interaction between the components.[40] The bulk mechanical properties of each component are determined by its own (a) molecular weight distribution, (b) molecular architecture, and (c) inter- and intramolecular bonding forces.[41]

Based on the above description, the mechanical response of composites is seen to be rather complex even under the simplest mode of external loading. With the application of external forces, each lamina, each component, and finally each molecule, already prestrained due to the residual stresses, deform further in a mode compatible with bonding restraint. Changes in the mechanical properties of a composite laminate due to moisture absorption may be attributed to:

(a) the change in the structure and properties of each lamina, each component, and each molecule;

(b) the change in the degree of bonding between the laminae, between the components, and between the molecules; and

(c) the change in the level of internal residual stresses.

These three aspects are being explored at each structural level of the composite, starting from the laminate eventually down to the molecules. In this report, the change in the mechanical properties of the laminate by water absorption will be related to the possible change in (a) the structure and properties of each lamina, (b) the degree of bonding between the laminae, and (c) the level of interlaminar residual stress. Although the mechanism of moisture absorption in composites will not be the main concern in this report, its several points of interest will be summarized in the remainder of this section to clarify later discussions.

Water molecules penetrate into the composites, mainly through the matrix resin. In the absence of any defects or stresses, the resin phase will allow the simple diffusion of water molecules. But, in actual composites, the presence of internal residual stresses will influence the diffusion rate, particularly in the region adjacent to the fiber/resin interface.* Under certain conditions, residual stresses might develop microvoids or fissures of a width sufficient to cause capillary action. The capillary phenomenon is also possible through formation of surface cracks during fabrication or subsequent use. In this case, voids along the exposed fiber/resin interface, as well as the resin phase adjacent to the interface play a significant role in water absorption. The presence of macrovoids running parallel to the fiber might also cause conduction of water molecules. Therefore, it is an open question whether the penetration of water molecules into composites occurs according to the law of diffusion or the law of filtration.[42] At the molecular level, the mechanism of water absorption in the resin phase will be governed by the affinity of specific functional groups to water molecules (e.g., polarity, hydrogen bonding characteristics, etc.).

SCOPE OF WORK

The principal objective of this report is to investigate the effect of water absorption on the mechanical properties of composite laminates. As discussed previously, it is the intent to relate the change in the mechanical properties of the laminate to the possible change in (a) the structure and properties of each lamina, (b) the degree of bonding between the laminae, and (c) the level of interlaminar residual stress. In this section, several technical questions related to these goals will be examined to define a clear boundary of the experimental work.

*Private communication with K. H. G. Ashbee.

First, the type of mechanical properties to be considered must be determined. A wide variety of mechanical properties can be dealt with depending on the specific application of the materials. In this study, the uniaxial tensile strength of the laminate under constant-rate deformation was chosen mainly because of availability and simplicity of the testing technique.

As stated earlier, 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° lamina, will fail first, thus reducing its share of load. The burden of load transfer will be increased in the unbroken portion of the laminate.[43] 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 translaminar crack, not plastic flow or dislocation motion. The yield point is usually far below the

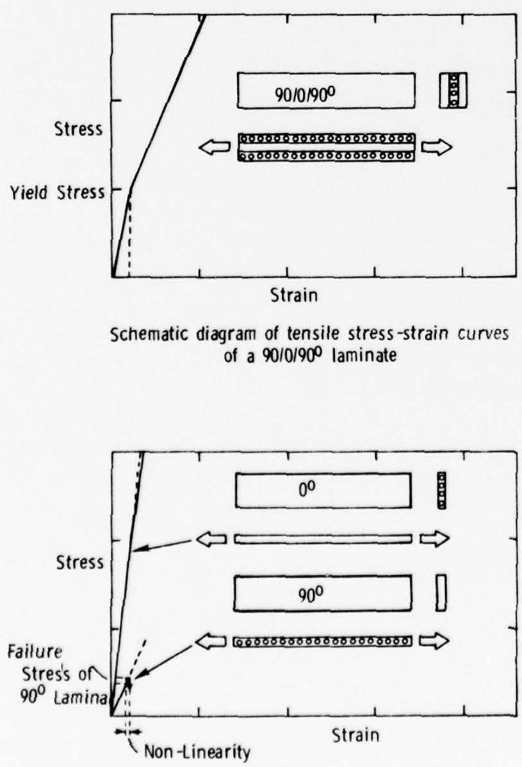


Figure 1a. Schematic diagram of tensile stress-strain curves of a 0° lamina, a 90° lamina, and a 90/0/90° laminate.

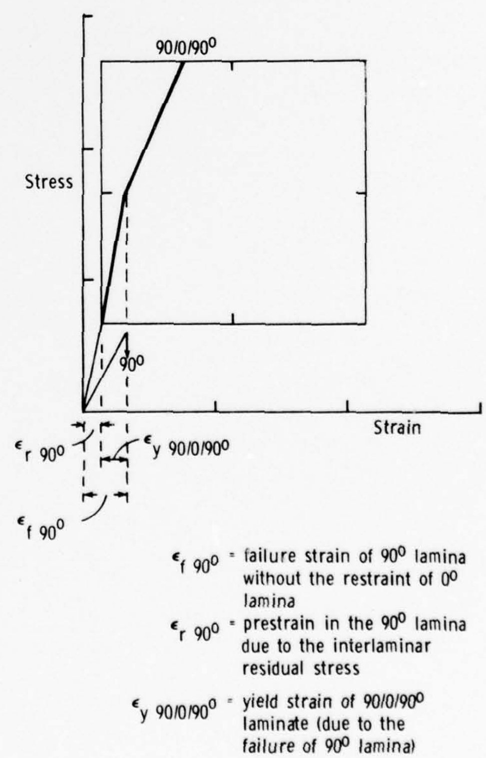


Figure 1b. Approximate relationship between the yield strain of a 90/0/90° laminate and the failure strain of a 90° lamina (without the restraint of a 0° lamina).

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.[31]

The yield stress or strain is also important from the viewpoint of resistance to water absorption. As discussed at the end of the previous section, 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 cross-ply (90/0/90°) laminates will be examined with particular emphasis on the yield point.

A last consideration in the study is to choose between two possible sequences of cutting and water immersion of specimens: (1) cut the specimens first and immerse them in water or (2) immerse the plate first and then cut the specimens.

When the cross section of fiber/resin interface is exposed by specimen cutting, the effect of subsequent water absorption may be more severe than the case of an actual composite structure having no exposed cross section or surface defects. However, the first option was chosen for two reasons:

(1) to minimize the concentration gradient of absorbed water inside the composites, and

(2) to avoid possible "desorption" of water by heat generation during the specimen cutting.

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 cross-plied laminae (lamina thickness $\sim$0.016 inch). The laminate has a density of 2.01 ± 0.02 gram/cc and 74.9 ± 1.2 weight percent fiber.[44,45]

Three sets of specimens (3/4 in. × 6-1/2 in. × 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.

(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°, 0°, and 0/90° specimens were prepared by removing unnecessary lamina(e) from the 90/0/90° 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° and 0° specimens were used in the evaluation of strength and modulus of each lamina. The curvature of 0/90° 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.

In addition to water absorption experiments, a set of 90/0/90° specimens which had been immersed in water was redried at room temperature. Tensile properties of 90/0/90° specimens and the curvature of 0/90° specimens were estimated and plotted against the amounts of water absorption before and after redrying.

RESULTS AND DISCUSSIONS

The weight gain of 90/0/90° 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.

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 increasing amount of water absorption (Figure 3a). Yield stress increased initially and then decreased, thus to form a broad maximum around the point of 0.2% weight gain (Figure 3b). On the other hand, no significant change was observed in the values of moduli before and after yielding due to water absorption (Figure 3c).

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

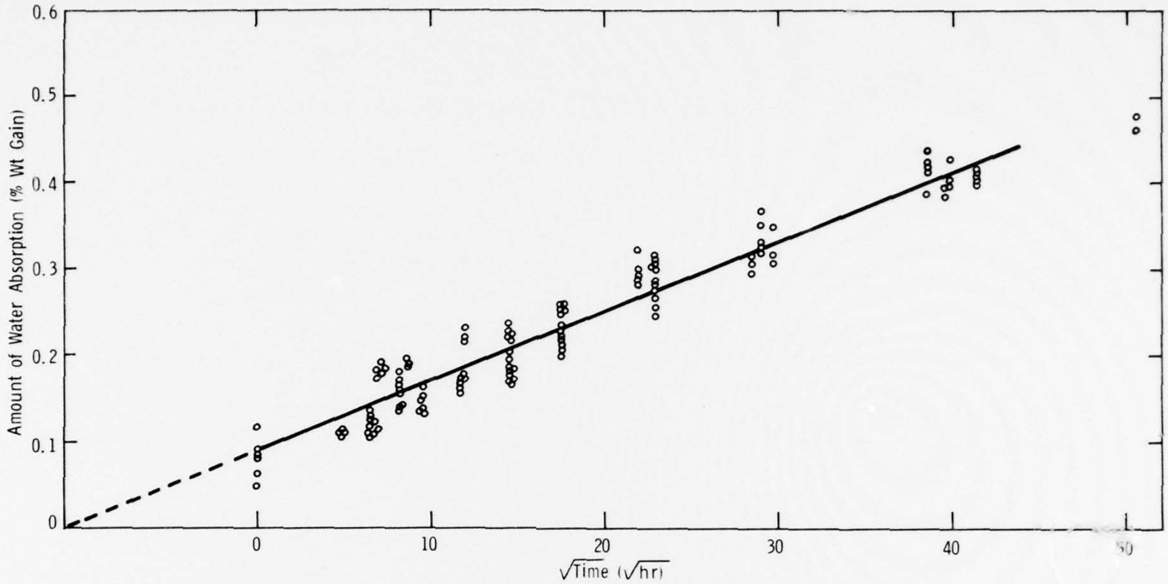


Figure 2. Amount of water absorption of a 90/0/90° laminate versus the immersion time at room temperature.

water absorption. Considering this, discussion in this report 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,[46] a 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 the fibers in the 0° lamina. At catastrophic failure, extensive delamination took place between the 0° and the 90° 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.

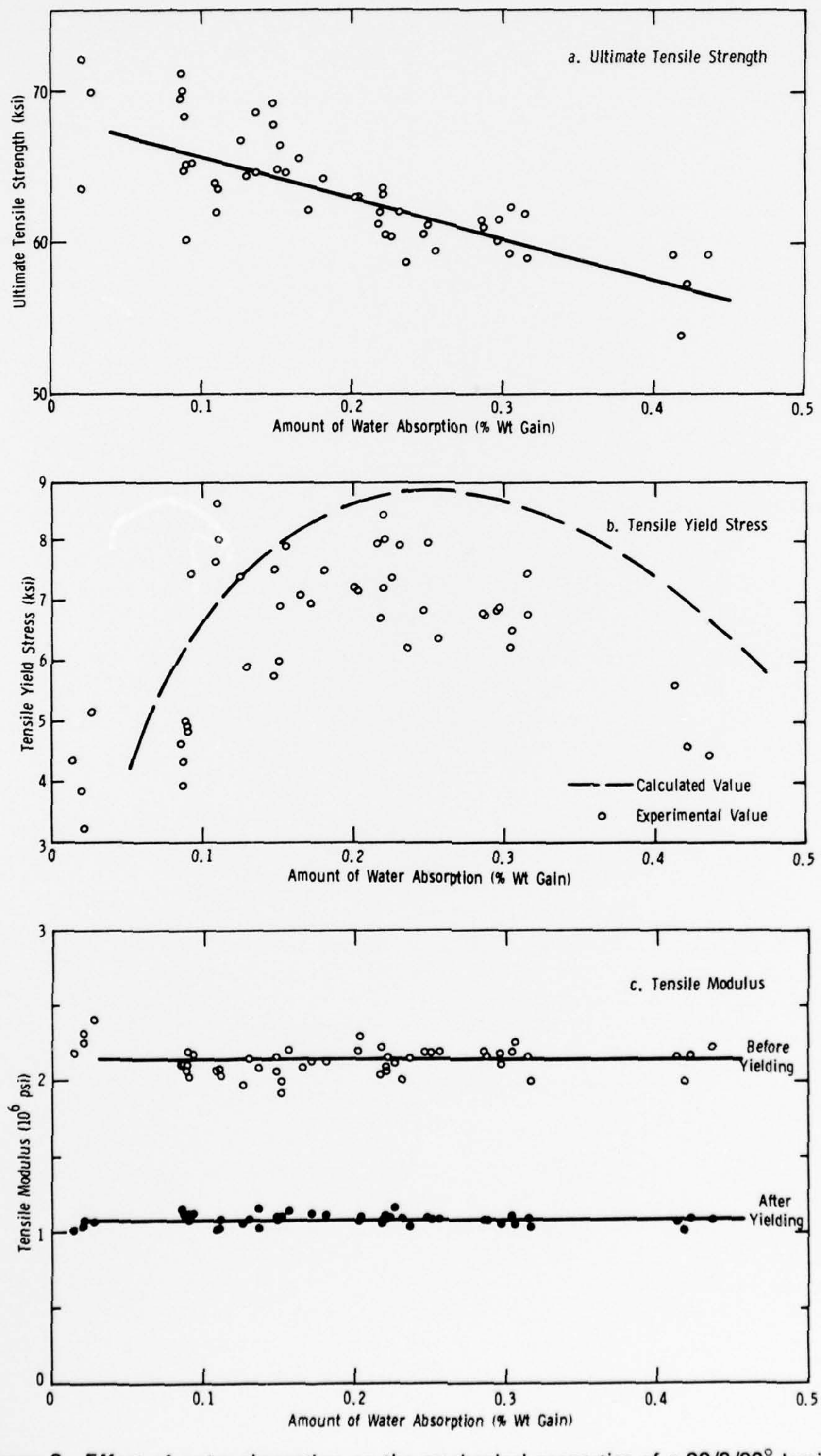


Figure 3. Effect of water absorption on the mechanical properties of a 90/0/90° laminate.

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

(a) failure strain of unrestrained 90° lamina, i.e., transverse ultimate strain of unidirectional lamina,

(b) degree of bonding between 90° and 0° laminae (which controls the degree of load transfer between the laminae), and

(c) prestrain in the 90° lamina due to the interlaminar residual stress.

If the interlaminar bonding is strong enough to preclude delamination before the failure of 90° lamina, the yield strain can be approximated as a difference between (1) the failure strain of 90° lamina without restraint and (2) the prestrain in the 90° 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° 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° Laminate

When a cross-ply laminate made up of 0° and 90° laminae is cooled from the cure temperature to room temperature, 90° laminae having higher thermal expansion coefficients try to shrink more than 0° laminae along the 0° direction (Figure 4). 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 (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° and 90° laminae during the cooling.

Interlaminar residual stress can be partially relieved by the distortion (warpage) of the laminate.[47,48] In actual cross-ply laminates, the distortion is avoided by mid-plane symmetry (e.g., 90/0/90/0/90/0/90°). 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,[39] and only normal stresses are possible: 90° lamina in tension and 0° lamina in compression along the 0° (load) direction. No warpage was in fact observed in the 90/0/90° symmetric laminates used in this study.

The differential contractive strain of 0° and 90° laminae was estimated two-dimensionally by measuring the degree of warpage in an unbalanced laminate. In

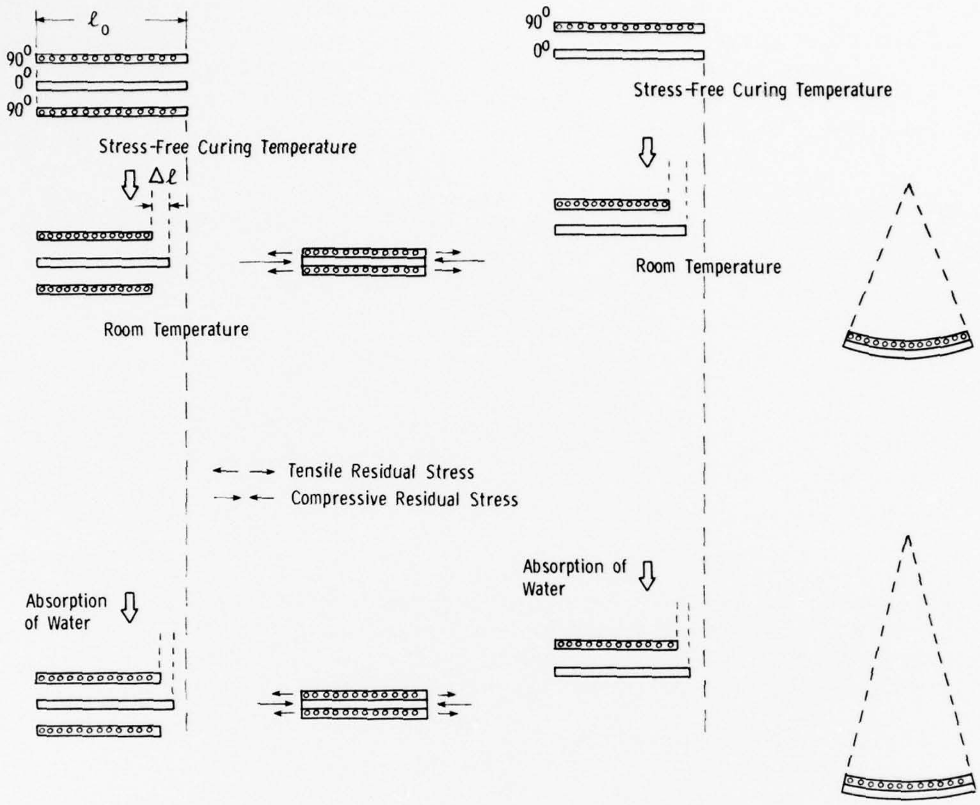


Figure 4. Interlaminar residual stresses for balanced and unbalanced laminates.

order to obtain unbalanced 0/90° laminate specimens, the top laminae were removed from the 90/0/90° laminate specimens which had been immersed in water for various periods of time. The curvature of each 0/90° specimen was measured, assuming the arc of the now warped specimen to be circular. The results showed that the curvature of 0/90° specimens decreased in a nearly linear fashion with an increasing amount of water absorption (Figure 5).

There have been several equations derived to relate the curvature of unbalanced laminates to the differential thermal strain of the constituent laminae.[47-51] The simplest of these is Timoshenko's bimetallic thermostat equation which was derived from the strain energy consideration[50] (Figure 6). Although this equation ignores the local shear interaction between the laminae, it was shown to agree reasonably well with the more rigorous solutions[51] 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 (Figure 7). 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.

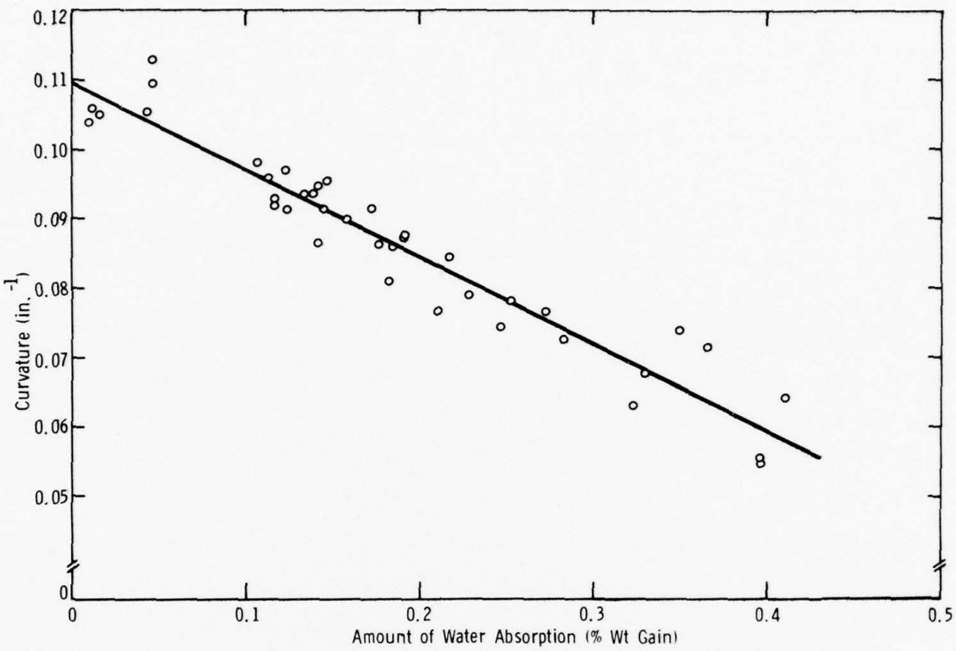
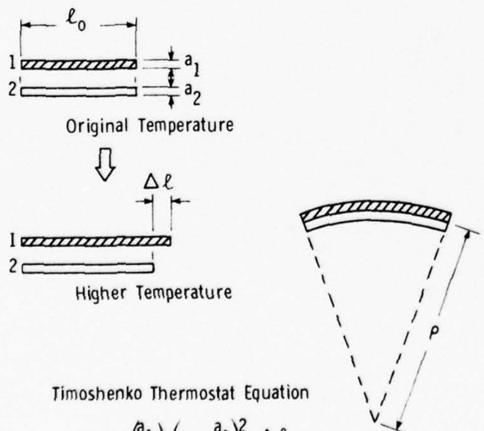


Figure 5. Effect of water absorption on the curvature of a 0/90° laminate.

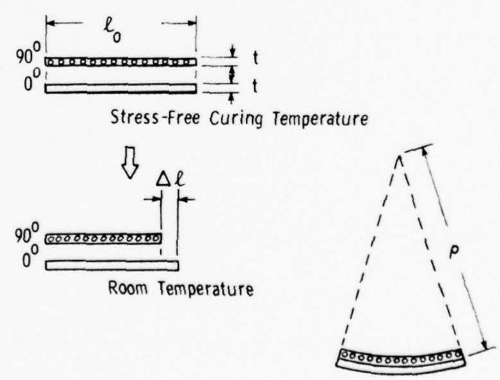


Timoshenko Thermostat Equation

$$\frac{6\left(\frac{a_1}{a_2}\right)\left(1+\frac{a_1}{a_2}\right)^2 \frac{\Delta\ell}{\ell_0}}{(a_1+a_2)\left[\frac{E_1}{E_2}+4\left(\frac{a_1}{a_2}\right)+6\left(\frac{a_1}{a_2}\right)^2+4\left(\frac{a_1}{a_2}\right)^3+\frac{E_2}{E_1}\left(\frac{a_1}{a_2}\right)^4\right]}=\frac{1}{\rho}$$

$\Delta\ell/\ell_0$ = differential strain
a = thickness
E = modulus
$1/\rho$ = curvature

Figure 6. Timoshenko's bimetallic thermostat equation.



$$\frac{24\frac{\Delta\ell}{\ell_0}}{2t\left[\frac{E_{90^0}}{E_{0^0}}+14+\frac{E_{0^0}}{E_{90^0}}\right]}=\frac{1}{\rho}$$

$\Delta\ell/\ell_0$ = differential strain of 0^0 and 90^0 laminae
t = thickness
E = modulus
$1/\rho$ = curvature

Figure 7. Relationship between the curvature of a 0/90° laminate and the differential strain of the constituent laminae.

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 8).

Using the average values of moduli of 90° and 0° laminae, the differential strain ($\Delta\ell/\ell_0$) of 0° and 90° laminae was calculated from the curvature of 0/90° specimens. Figure 9 shows that the differential contraction of 0° and 90° laminae due to the cooling decreased linearly with increasing amount of water absorption. This phenomenon could be explained by one or more of three factors.

(1) Reduction in the modulus of 90° 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.[16,52])

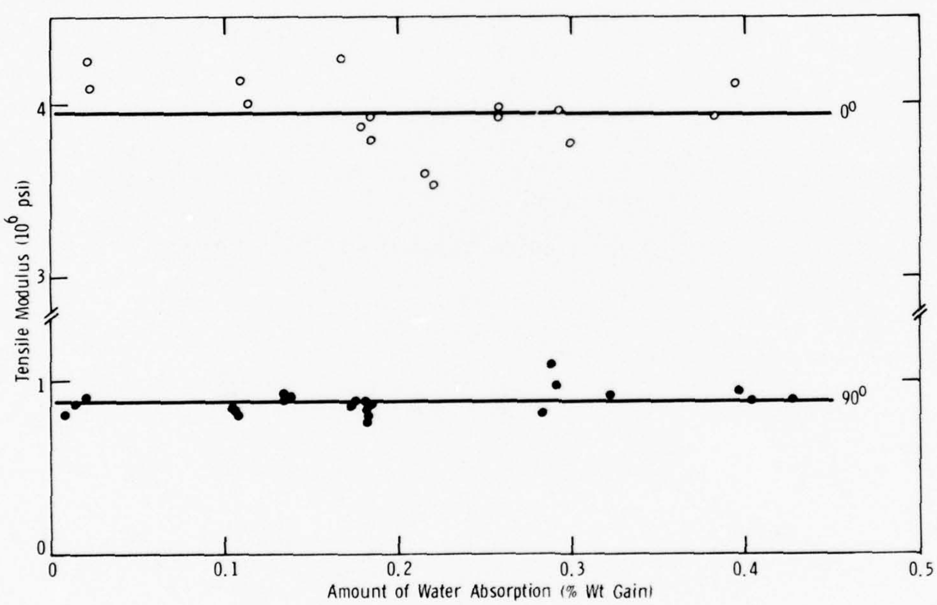


Figure 8. Effect of water absorption on the tensile modulus of 0° and 90° laminae.

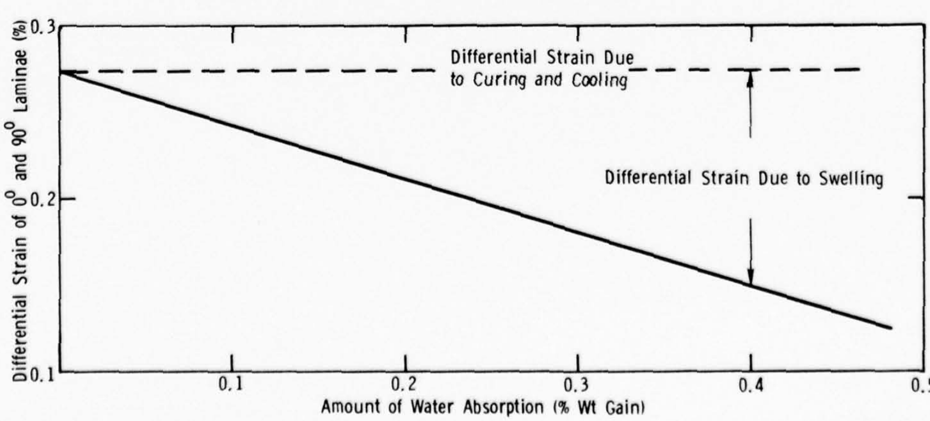


Figure 9. Effect of water absorption on the calculated differential strain of 0° and 90° laminae.

(2) Reduction in the degree of interlaminar bonding.

(3) Development of differential expansive strain of 0° and 90° 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° lamina by water absorption (Figure 8). Presumably, the reduction in the glass transition temperature of matrix (1009) 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° 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° and 90° 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.[12] 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 (Figure 4). The second and third factors will be investigated by additional experiments in the immediate future.

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[33] (see Appendix). 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:

$$\sigma_{r\ 90^\circ} = [(\Delta\ell/\ell_0)\ E_{90^\circ}]/1 + (E_{90^\circ}\ A_{90^\circ}/E_{0^\circ}\ A_{0^\circ})$$

where ($\Delta\ell/\ell_0$ = 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

*In fact, the whole failure pattern (including the tendency toward coupling between the delamination and the translaminar crack formation) was affected by water absorption in the other composite laminate which was not discussed in this report.

calculated from the interlaminar residual stress ($\sigma_{r\ 90°}$) in a straightforward manner. The tensile prestrain in the 90° lamina calculated as above showed a linear decrease with increasing amount of water absorption (Figure 10). It reflects clearly the relaxation of interlaminar residual stress by water absorption.

Before going further, it is worthwile to comment on the use of the term "swelling." Although a limited plastic flow has been observed at a crack tip,[53] the molecular mobility of epoxy resin on the whole has been found to be very low at ambient temperature.[54] Therefore, volume expansion of epoxy resin due to water absorption is quite limited at room temperature and far different from the classic case of "network swelling" by solvent (e.g., swollen gel).[55] However, since the magnitude of volume expansion due to water absorption can be comparable to that of volume constraint by residual stress in the composites, the use of the term swelling has been employed in a more general sense.

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 11). 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 essentially comprises future work ("The Effect of Static Immersion in Water on the Tensile Strength of a Lamina"). However, it is worthwhile to note several important factors which can induce the change of failure stress.

As discussed in the Introduction, intralaminar residual stresses develop in the fiber and matrix components of the lamina since the resin attempts to shrink more than the fiber during processing. The level of intralaminar residual stress is strongly dependent on the geometry of fiber packing.[38-40] Schneider[39] and Haener et al.[40] independently calculated the distribution of residual stress around the fiber assuming hexagonal array of reinforcing fibers and linear elastic behavior of components. Their results show that normal stresses in the resin phase between fibers are predominantly compressive in the radial direction and tensile in circumferential and axial directions. Shear stresses were shown to vary along the fiber circumference, but were of a significantly lower magnitude than the normal stresses.

Under external force, the distribution of intralaminar residual stress is influenced additionally by "stress concentration" or "strain magnification" effects.[56] Because of this complicated stress distribution, it is a difficult task to establish the effect of intralaminar residual stress on the failure stress of 90° lamina (in other words, transverse strength of unidirectional lamina). Moreover, the micro level failure behavior of the lamina transverse to the fiber direction is still not clearly understood.[57] Based on the preceding discussion, it can be seen that the change in the transverse strength of lamina by water absorption has a remarkably complex nature. Water absorption will certainly affect the distribution of stress (both residual and external) and the failure pattern of the lamina.

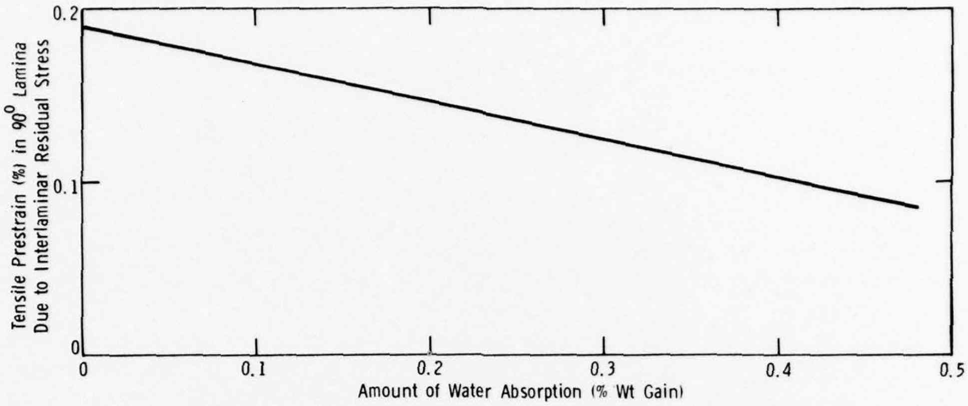


Figure 10. Effect of water absorption on the calculated tensile prestrain in the 90° lamina of a 90/0/90° laminate.

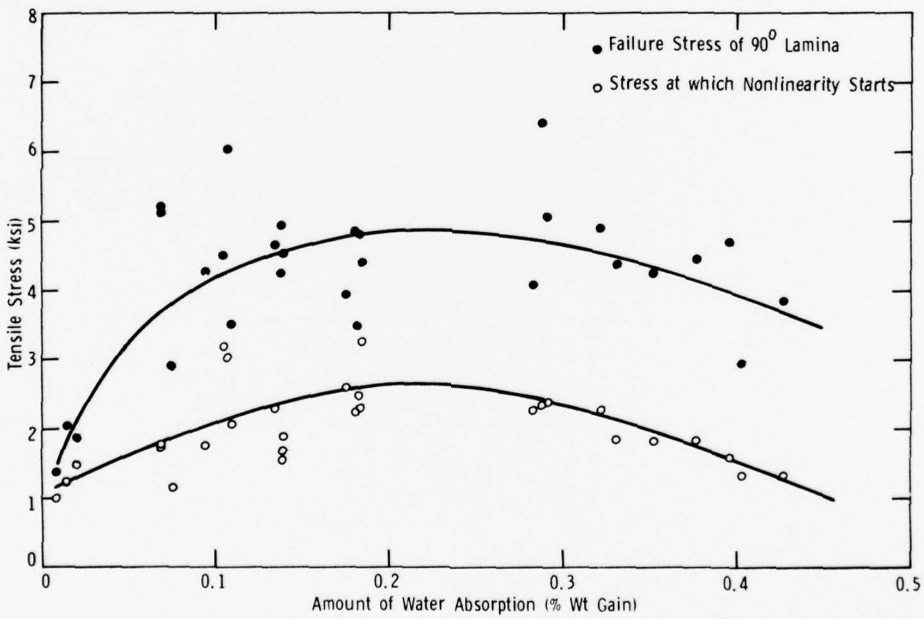


Figure 11. Effect of water absorption on the failure stress of a 90° lamina without the restraint of a 0° lamina.

Despite the complexity, speculation is possible in a qualitative sense concerning the effect of water absorption on transverse tensile strength, assuming that the failure initiates either in the resin phase or at the fiber/resin interface.[57] If water absorption induces volume expansion of matrix resin between fibers, it should relax pre-existing intralaminar residual stress in all directions. The residual stress will become less compressive radially and less tensile circumferentially and axially. The reduction of tensile circumferential and axial stresses (as a prestress) will contribute to the local recovery of tensile strength of matrix resin in the lamina. If the bonding strength at the fiber/resin interface remains stronger than the locally increased strength of resin after water absorption, the recovery of resin strength will result in the increase of transverse tensile strength of the lamina.

The decrease of compressive radial stress around the fiber will reduce the degree of friction between the components (less squeezing of fiber by resin) thus weakening fiber/resin bonding. In addition, the interfacial bonding between the fiber and resin can be destroyed by diffused water molecules.[17-19,58,59] Water molecules can be stored in the macro- and microvoids at the interface. After the lapse of substantial time, isolated pockets of water molecules may surround the fibers and extend along the fibers. Ashbee attributed the failure of fiber/resin bonding to osmotic pressure generated at the interface by water-soluble constituents leached from the glass fiber.[58,59] When the strength of the fiber/resin bonding becomes lower than the resin strength after water absorption, the lamina failure will initiate at the fiber/resin interface instead of resin phase. Therefore, weakening of fiber/resin bonding by water absorption will result in the decrease of transverse tensile strength of the lamina. The maximum of the failure stress of 90° lamina which were observed in the present study may result from overlapping of those two opposing tendencies.

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,[33,50] the tensile prestrain in the 90° lamina due to the interlaminar residual stress was predicted to decrease continuously with increasing amount of water absorption (Figure 10). The change in tensile failure stress or strain of 90° lamina without the restraint of 0° lamina by water absorption was also observed (Figure 11). 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 $\varepsilon_{f\ 90°} - \varepsilon_{r\ 90°}$, assuming negligible Poisson's effect (Figure 1b). The yield stress of 90/0/90° laminate can be obtained from the values of $\varepsilon_{f\ 90°}$, $\varepsilon_{r\ 90°}$, and $E_{90/0/90°}$ (Figure 12). Although the values of $\varepsilon_{f\ 90°}$ could be estimated directly from the stress-strain curve, the calculated values of $\sigma_{f\ 90°}/E_{90°}$ were used 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 3b. While based on limited data, the prediction of the change in yield stress is relatively 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.

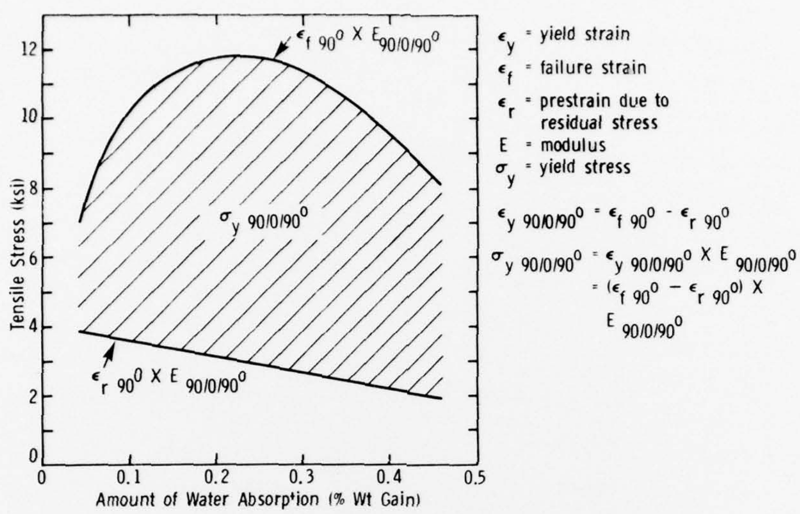


Figure 12. Effect of water absorption on the calculated yield stress of a 90/0/90° laminate.

D. Reversibility of the Effects of Water Absorption

As shown in the previous sections, water absorption affects the levels of (a) yield stress and ultimate strength of 90/0/90° laminate, (b) curvature of 0/90° laminate, and (c) failure stress of unrestrained 90° lamina. Whether these changes can be recovered by drying is of more than practical importance. If the effect of water absorption on the above-mentioned properties is reversible irreversible loss of inter- and intralaminar bonding strength cannot be a factor contributing to the observed property changes.

In this work, the reversibility was checked in the yield stress and ultimate strength of 90/0/90° laminate and the curvature of 0/90° laminate. Each property was measured after water immersion followed by redrying and plotted against the amounts of water absorption before and after redrying (Figure 13). The change induced by initial water immersion in each property (Figures 3a, 3b, 5) was taken as a broad band covering scattered data and projected as a shaded area. Despite broad baselines, the changes in all three properties due to water absorption were found to be recoverable by drying.

Based on the proved relationship between the yield stress of 90/0/90° laminate and failure stress of unrestrained 90° lamina (Figure 12), the above experimental observation indicates indirectly that the change of failure stress of 90° lamina due to water absorption is probably also reversible. Two conclusions can be drawn from the findings on the reversibility.

(1) The development of differential expansive strain of 0° and 90° laminae by swelling of matrix resin is reversible.

(2) No irreversible change is induced by water absorption in the degree of inter- and intralaminar bonding of these composites.

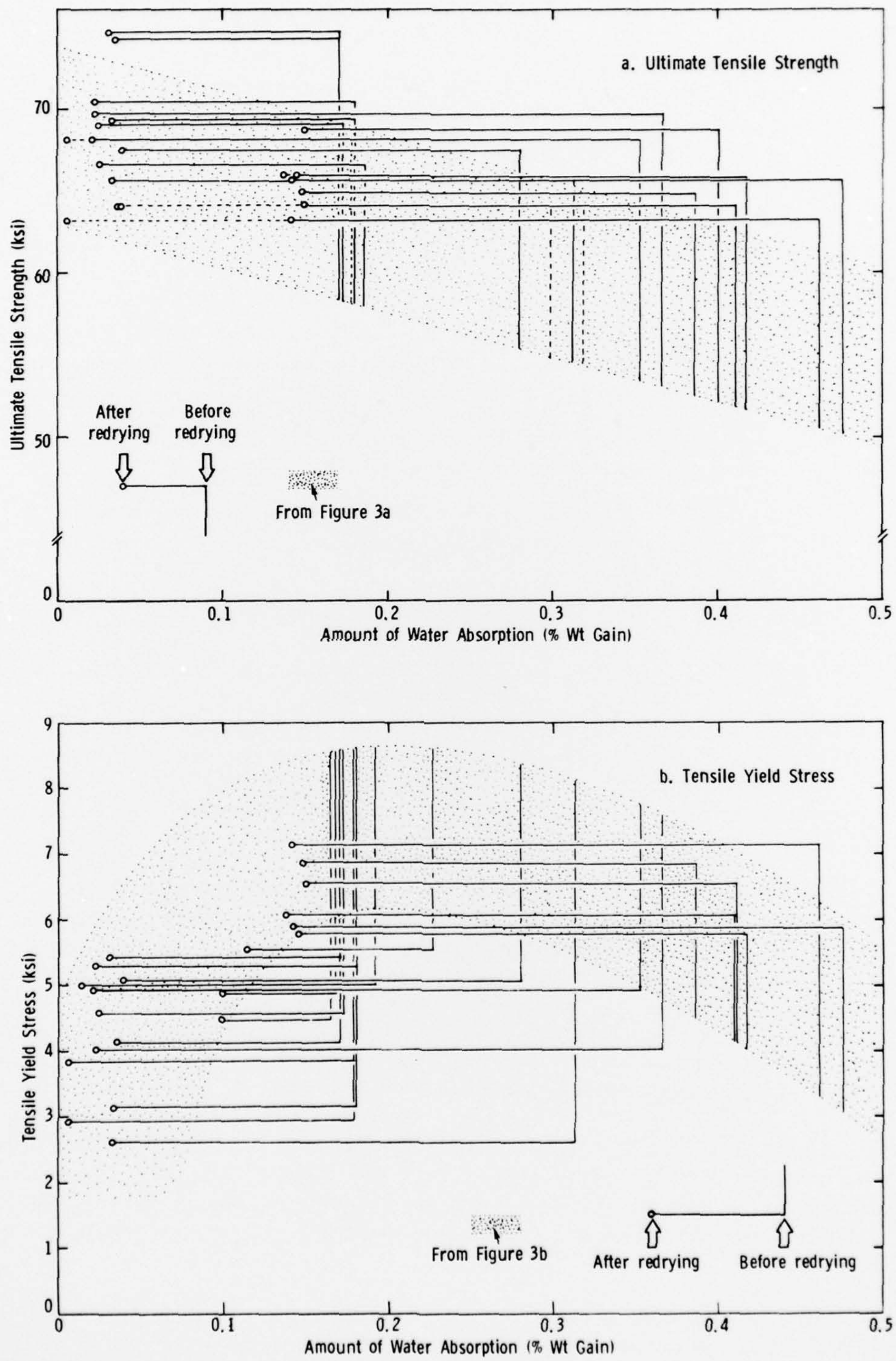


Figure 13a and b

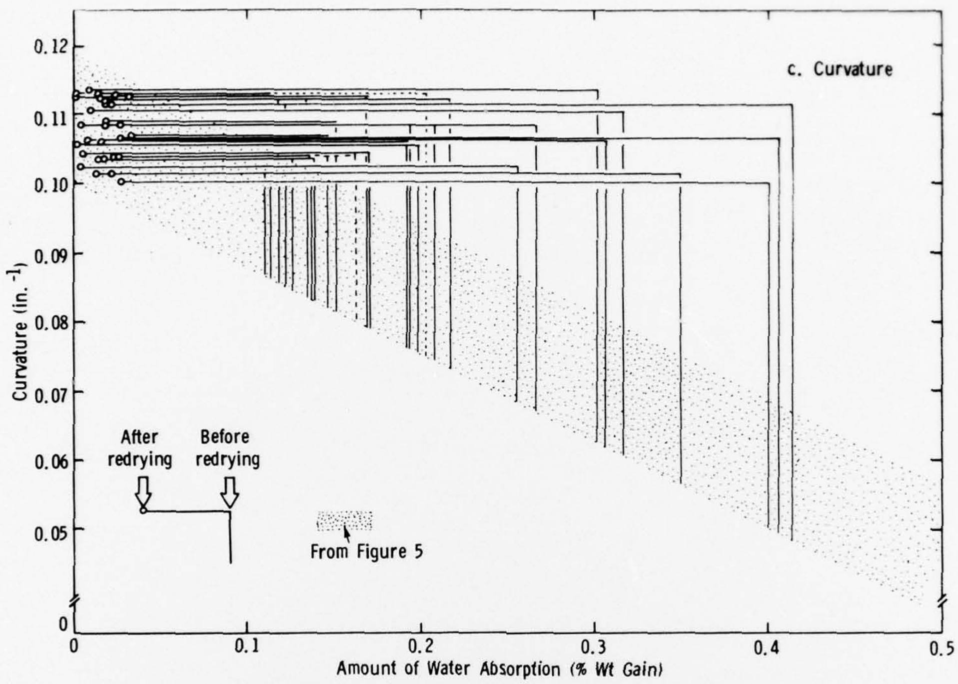


Figure 13a-c. Effect of water desorption on the tensile properties of a 90/0/90° laminate.

However, it should be noted that reversible change in the degree of inter- and intralaminar bonding is theoretically possible and therefore may contribute to the change of bulk mechanical properties of composites. Ashbee observed that fiber/resin bonding could be weakened by the exposure to water and subsequently restored by thorough drying. This recovery was found to be shorter-lived when the exposure to water was resumed, suggesting that the recovery was due to the restoration of compressive residual stress around the fiber rather than that of interfacial adhesion.[58]

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 inter-laminar 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.

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 effect 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.

The changes in the above-mentioned properties by water absorption were found to be reversible. No irreversible change by water absorption is expected in the degree of inter- and intralaminar bonding.

APPENDIX

In our work, interlaminar residual stress in the balanced cross-ply laminate was estimated by a two-dimensional analytical technique.[33] The following assumptions were used in the analysis:

(1) perfect bonding between laminae,

(2) straight alignment of laminae,

(3) linear elastic behavior of the lamina below stress-free curing temperature ($\sim T_g$ of matrix resin),

(4) constant modulus of the lamina below stress-free temperature,

(5) negligible transverse strength of the lamina above stress-free temperature, and

(6) negligible Poisson's effect.

More rigorous solutions can be found in the work of Hahn and Pagano.[35]

Consider a laminate made up of 0° and 90° laminae (Figure 4). The laminae are stress-free and have the same dimensions at the curing temperature. Upon cooling, the 90° laminae attempt to contract more than 0° laminae in the 0° direction. Since the same dimensional contraction of the laminae are required, 90° laminae are in tension and 0° laminae are in compression upon cooling.

$$\ell_{90^\circ}/(\ell_{90^\circ})_o + \varepsilon_{r\ 90^\circ} = \ell_{0^\circ}/(\ell_{0^\circ})_o - \varepsilon_{r\ 0^\circ}$$

$$(\ell_{90^\circ})_o = (\ell_{0^\circ})_o = \ell_o$$

where $(\ell)_o$ = dimension of the lamina at stress-free curing temperature

ℓ = dimension of the lamina without the restraint of the other laminae laminae at room temperature

ε_r = residual strain.

Linear elastic behavior of lamina postulates:

$$\varepsilon_{r\ 0^\circ} = \sigma_{r\ 0^\circ}/E_{0^\circ} \qquad \varepsilon_{r\ 90^\circ} = \sigma_{r\ 90^\circ}/E_{90^\circ}$$

where σ_r = residual stress

E = modulus of elasticity.

The tensile force in 90° laminae is equal to the compressive force in 0° laminae:

$$\sigma_{r\ 0^\circ} A_{0^\circ} = \sigma_{r\ 90^\circ} A_{90^\circ}$$

where A = area fraction.

Solving for $\sigma_{r\ 90^\circ}$,

$$\sigma_{r\ 90^\circ} = (\ell_{0^\circ} - \ell_{90^\circ}/\ell_o) \cdot E_{90^\circ}/1 + (E_{90^\circ} A_{90^\circ}/E_{0^\circ} A_{0^\circ}).$$

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ENVIRONMENTAL EFFECTS ON THE MECHANICAL PROPERTIES OF GLASS FIBER/EPOXY RESIN COMPOSITES - Part I. Effect of Static Immersion in Water on the Tensile Strength of Cross-Ply Laminates - Byung-Lip Lee, Robert W. Lewis, and Robert E. Sacher

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Key Words

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Epoxy resins

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. 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. 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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