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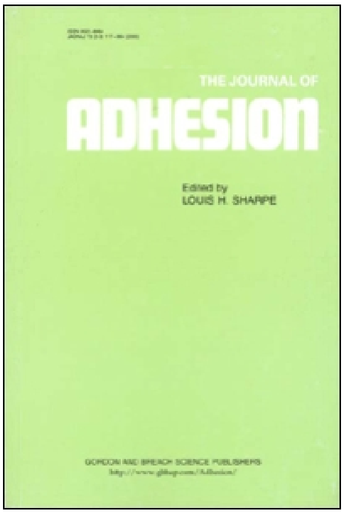

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The Effect of Aging in Water on the Strength of Fiber-Polymer Systems

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The Effect of Aging in Water on the Strength of Fiber-Polymer Systems

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The effect of the exposure time in water on the adhesive strength of fiber/resin systems was studied. To this end, the adhesive strength of single-fiber/matrix systems was measured. Such a system simulates the unit cell of a fiber-reinforced composite. The resin was an epoxy (DGEBA-based) polymer. The substrate used consisted of boron fibers, a steel wire and glass fibers. The samples were kept in water for a period of 1–280 days at room temperature and the adhesive strength was measured also at room temperature. The water absorption of the resin and the impregnated strand produced on the basis of the glass fibers and the resin studied and also the physico-mechanical characteristics of the impregnated strand were studied at the same time. The samples used to determine the adhesive strength were kept in such a contact with water that the latter could penetrate into some of them only at the interface and into the others at the interface and through the resin layer.

It was found that the adhesive strength decreases with increasing amount of water absorbed. On the basis of the results obtained it was concluded that the principal factor that determines the loss in adhesive strength during the aging of the systems in water is the plasticization of the epoxy resin by water.

KEY WORDS Fiber-resin bond strength; aging in water; epoxy resin; boron, glass and steel fibers; impregnated strand; glass-transition temperature; plasticization.

INTRODUCTION

It has been shown[1,2] that upon completion of the process of curing of a resin the adhesive strength of a thermosetting polymer/fiber system varies little when the system is exposed to air. Such stability has been demonstrated for fibers of various nature—glassy, metallic and organic (chemical)—upon their interaction with epoxy, phenol, polyester and some other resins. The experimental results have been obtained in a study of the shear adhesive strength of a fiber-matrix system, which was determined using the method of pull-out of monofibers from the cured polymer.[1,3-7]

There arises the question: How stable is the strength of such joints upon exposure to liquids (water and various solvents)? No such data are available in the literature. Meanwhile, this problem is important from both a theoretical and a practical standpoint. Its theoretical importance is accounted for by the fact that the mechanism of action of moisture in composites and their model joints has not yet been completely elucidated. The practical importance is evident since fibrous composites are widely used in the various liquid media.

The purpose of the present work is to study the effect of the exposure time, t, in water on the adhesive strength τ of the fiber/resin interface. Here, as previously,[1,2,4-7] we studied the adhesive strength in a monofiber/matrix system, which simulates the unit cell of a fiber-reinforced composite.

EXPERIMENTAL

The adhesive used was an epoxy polymer (based on DGEBA) modified by diethylene glycol and cured by triethanolaminotitanate at 160°C for 8 hours. Preliminary measurements showed that under such conditions the adhesive is completely cured and the possible maximum degree of conversion α of the reactive epoxy group present in the polymer is attained ($\alpha = 96\%$). The substrate used consisted of a steel wire of diameter $d = 150$ μm, boron fibers of diameter $d = 140$ μm and glass fibers of alkaline-free glass (an analog of E-glass) of diameter $d = 10$–13 μm. The procedures of fabrication of joints composed of an epoxy resin and "thin" ($d = 7$–30 μm) and "thick" ($d > 100$ μm) fibers and also the methods of their testing and of treatment of the measurement results have been described in detail in the literature,[1,4-7] in particular the procedure of calculation of the average value of adhesive strength τ in experiments in which, apart from specimens that fractured adhesively (at the interface), use was also made of samples which fractured in a cohesive way, *i.e.*, systems in which the fiber is fractured earlier than the interface. Figure 1 presents the types of specimens studied. The time during which the specimens were exposed to water varied from 1 day to 9 months. The basic measurements were made for systems with boron fibers, since upon aging in water the strength of glass fibers decreases and the strength of the wire is almost completely lost because of corrosion.

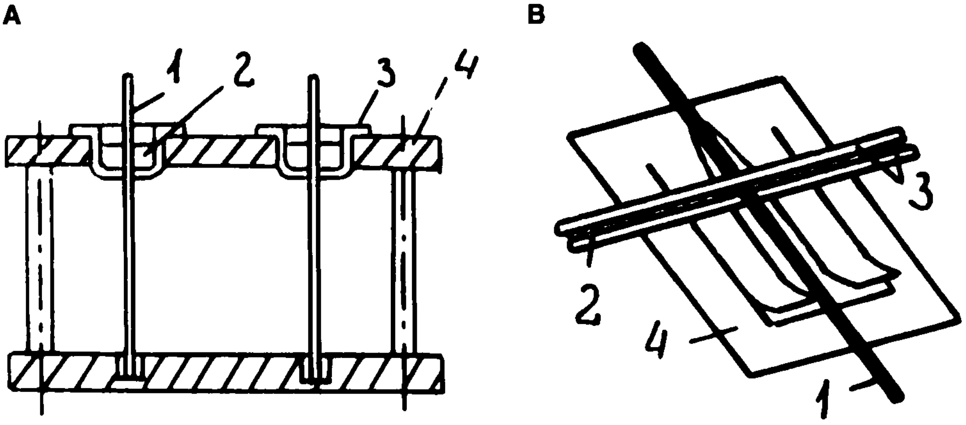


FIGURE 1 (a) Fabrication of specimens with fibers of diameter $d \geq 100$ μm: (1) fiber; (2) polymer; (3) aluminum mould; (4) supporting plate. (b) Preparation of samples with fibers of diameter $d = 6$–40 μm: (1) "thin" fiber with $d = 6$–40 μm; (2) polymer; (3) "thick" fibers—supports for resin; (4) frame with "whiskers."

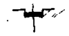
In order to elucidate the mechanism of penetration of the liquid into the adhesive joint, it is desirable to study samples in which various paths of liquid diffusion are possible. Such a possibility appears in a study of systems composed of polymers and "thick" ($d \geq 80-100$ μm) fibers, which are fabricated using the method of casting into moulds (Fig. 1a). The manner in which such samples come into contact with the liquid and the possible paths of penetration of the liquid into the system are shown in Table I.

The specimens in aluminum moulds were either floating (Nos. 2 and 3) or completely submerged in water (No. 4). In order to prepare specimens in which the resin surface could be completely accessible to the liquid (Nos. 6 and 7), the aluminum moulds used for casting were covered with an antiadhesive (silicone) coating. Such moulds were easily removed after the resin had been cured. All the samples under study were kept at room temperature. No special conditions were created in order to maintain a constant temperature. Possible small (21 ± 3)°C natural oscillations in temperature in our experiments are practically unimportant since water penetrates (diffuses) into the samples that are under the same temperature conditions. (Note also that, as will be shown at a later time, the most significant factor appears to be the total amount of water absorbed rather than the kinetics of its absorption).

Since the values of adhesive strength measured in our experiments depend significantly on the shape and size of samples (see, for example,[1,8-14]), the measurements were conducted, as a rule, with the length of the joints ℓ being varied within as wide a range as possible (see Fig. 2).

Apart from measurements of adhesive strength, we also studied the water absorption W_{res} of the resin and also the water absorption W_{com} of impregnated strands produced from the same resin and glass fibers of alkaline-free composition, and the effect of water absorption on their strength, F, and rigidity, G. The water absorption was determined by weighing, the weighing accuracy achieved being ±0.00005 g. This made it possible to weigh the resin and impregnated-strand samples with an accuracy not lower than $1 \times 10^{-2}\%$ and to determine water absorption with an accu-

TABLE I
Methods of exposure of specimens to water
(boron fiber/epoxy resin joints)

Position of specimen	Symbols in Figs. 2 and 4	Possible paths of water penetration	
		Along the interface	Through the resin bulk
1. (*) 	●	—	—
2. 	⊙	From below	—
3. 	ø	From above	From above
4. 	X	From below and from above	From above
5. (*) 	⊡	—	—
6. 	◬	From below	From below
7. 	■	From below and from above	From below and from above

(*)Control specimens (exposed to air).

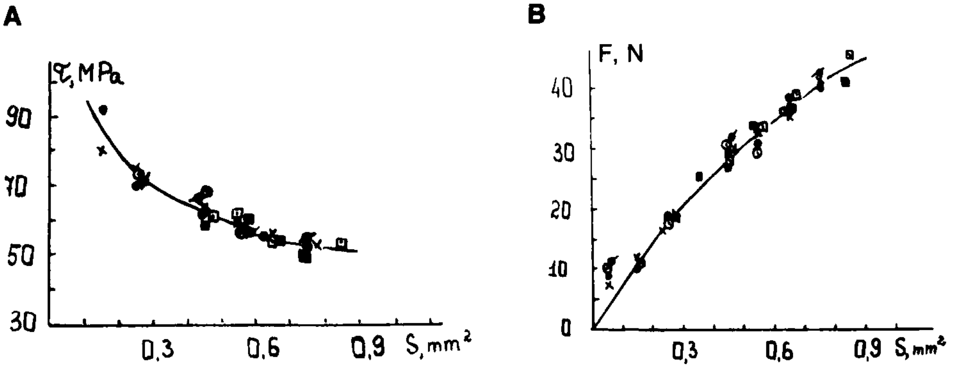


FIGURE 2 The adhesive strength of boron fiber/epoxy resin joints (a) and the force required for the fracture of joints (b) as a function of the specimen size. $S = \pi d\ell$, where S is the surface area of joints (the area of contact between the fiber and the matrix); d is the fiber diameter; ℓ is the length of the joint (the length of the fiber portion immersed in the matrix). For the symbols, see Table I. The exposure time in water is 1 day.

racy not worse than 0.2%. The quantity W_{res} was measured using resin disks removed from aluminum moulds upon completion of the determination of the adhesive strength of the control samples. The value of water absorption is given by

$$W_{res} = \frac{\Delta m}{m_0} = \frac{m_t - m_0}{m_0}$$

where m_0 is the total mass of several tens of resin disks prior to exposure to water; m_t is their mass after exposure to water during the time t. The water absorption of impregnated strands was measured on samples identical to those used for determination of the rigidity. The rigidity, G, was measured on a torsional pendulum, which is analogous to the one described in the literature.[15,16] The samples were bundles of filaments impregnated with the epoxy resin under study. The length of the specimens was 30 cm. Since the rigidity of a resin-impregnated filament depends on the mass of the resin, for each of the 25 specimens that were exposed to water we constructed plots of $\Delta M/M_0$ *vs.* t and $\Delta G/G_0$ *vs.* t, where $\Delta M = M_t - M_0$ and $\Delta G = G_t - G_0$; M_0, M_t, G_0 and G_t are the mass and rigidity of the ith specimen before and after exposure to water for the time t, respectively ($i = 1, 2, \ldots 25$).

RESULTS AND DISCUSSION

Figures 2–5 and Tables I and II present the results reflecting the kinetics of variation of the adhesive strength during the aging of the joints in water. It turned out (Fig. 2a and b) that after a short-term (one day) exposure to water the strength of all the systems remains practically unchanged. The adhesion varies little even after a 10-day exposure of the joints (Figs. 3 and 5). The adhesion then begins to decrease. A noticeable decrease in systems with boron fibers discontinued after 3 months (Figs. 3 and 4). Further exposure to water practically does not change the strength of the systems (and, hence, the force required for their fracture). From Fig. 4 it follows

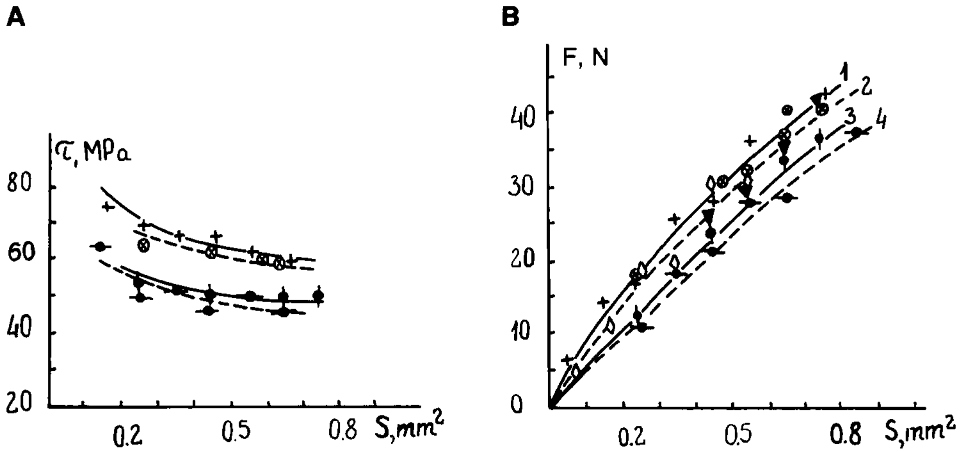


FIGURE 3 The effect of the time of exposure to water on adhesive strength (a) and the force required for the fracture of the joint (b). The exposure time in water: + = control specimens; ◇ = 10 days; ⊗ = 3 weeks; ▼ = 7 weeks; ⧫ = 3 months; ◆ = 6 months. System No. 4.

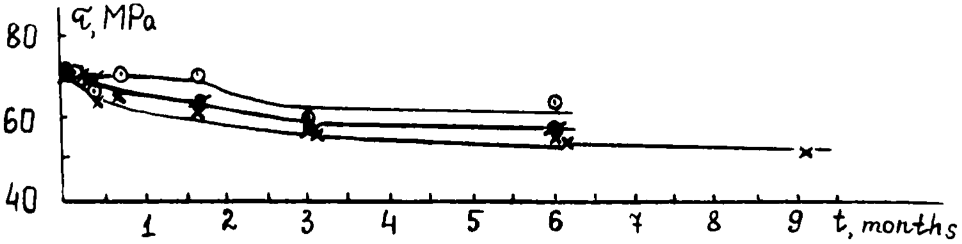


FIGURE 4 The adhesive strength of boron fiber/epoxy resin joints after exposure to water. S = 0.25 mm². For the symbols, see Table I.

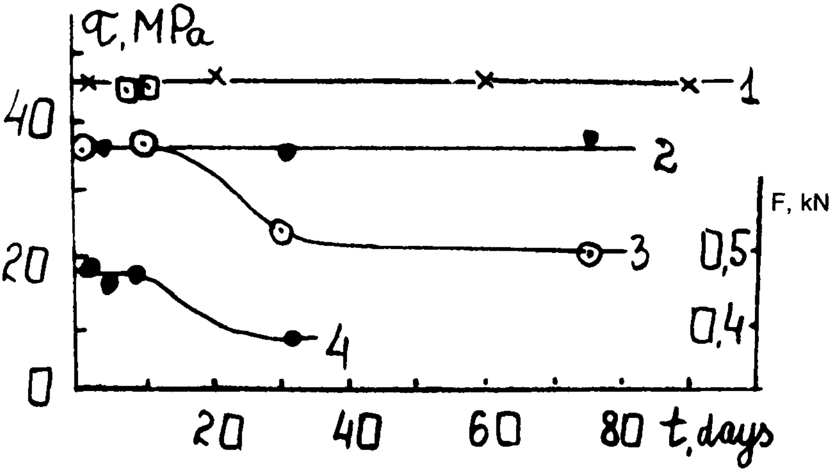


FIGURE 5 The adhesive strength of fiber/epoxy resin joints (1–3) and the strength of an impregnated strand based on the same resin and alkaline-free glass fibers (4) as functions of the time of exposure to water and air: (1) joints with a steel wire, d = 150 μm; S = 0.95 mm². Exposure: χ = air; □ = water; (2) and (3) joints with glass fibers, d = 10–13 μm; $S = 8 \times 10^{-3}$ mm². Exposure: (2) air; (3) water.

TABLE II
The adhesive strength of an epoxy resin/alkaline-free glass fiber system (glass fiber with d = 10–13 μm) upon exposure to water

Exposure time, days	$\alpha^{(*)}$, percent	n_τ	n_σ	τ_0, MPa$^{(**)}$	n_τ/n_σ
0 (control)	92	56	47	35.5	1.12
10	92	39	48	36.6	0.81
30	91	43	23	23.8	1.90
75	93	45	16	20.3	2.80

(*)α, percent = degree of curing of the resin. The values of α were determined using silver ion titration.
(**)$S = 8 \times 10^{-3}$ mm^2.

that the kinetics of the loss in strength, τ, depends on the diffusion path of water into the sample. For system No. 2 in which the liquid can reach the interface only along the fiber, the loss begins only in 1.5 months after the beginning of the exposure. In systems Nos. 3 and 4, when water penetrates both along the fiber and through the resin bulk, during the first weeks of exposure there is observed a more intensive loss of the strength. The paths of penetration of the liquid in these systems (Nos. 3 and 4) are almost the same. The kinetics of variation of the strength during the first 1.5–2 months is also similar. (It should be noted that the scatter of values is 8–10%. The differences observed for different systems are only insignificantly beyond this range. We may, therefore, speak with confidence only of the tendencies of the changes observed).

From Figs. 3 and 4 it follows that the maximum loss in adhesive strength during the exposure of systems composed of an epoxy resin and boron fibers to water does not exceed 15–20% (with respect to the strength of the control sample exposed to air). The loss is at a maximum for systems Nos. 4 and 7.

Upon exposure of the steel wire/epoxy resin joints studied to water the adhesive strength at the interface (just as in joints with boron fibers) remained constant during 10 days (see Fig. 5). After a 2-week exposure the adhesive strength could not be determined with any degree of reliability since, because of the corrosion of the wire, upon loading of the specimens most of them suffered cohesive fracture across the wire.

Upon aging of resin/glass fiber joints in water there is observed a monotonic decrease of the strength after 10-day exposure. By the end of the 2.5-month period the loss in τ attained 43% (Table II). It should also be noted that as the exposure time is increased the ratio of the number of specimens fractured adhesively (n_τ) to the number of specimens fractured cohesively (n_σ) undergoes a change; the value of n_τ/n_σ increases appreciably (Table II). Such an increase is natural since under the action of water the strength of the fiber decreases to a smaller extent than does the adhesive strength at the interface. It should be stressed here that the joints studied by us are in very unfavorable conditions: the length of the joints is very small—200–250 μm when they are bonded to glass fibers and 1–2 mm when bonded to steel and boron fibers. Therefore, the liquid can penetrate to the interface along the fiber and also through the bulk of the resin. In real reinforced plastics the loss of strength during aging under comparable conditions must be less than in the systems studied in the present work. Analysis of numerous works, whose authors studied the aging and water resistance of reinforced plastics, shows it to be the case in reality.

Figure 6 shows the manner in which the water absorption of a pure resin varies in water (system No. 4). It is seen that the saturation of the resin with water is attained in 2.5–3 months. Comparison of the results of measurements of the water absorption and of the variation of adhesive strength (see Fig. 7) shows that the loss in adhesion in this system correlates with the amount of sorbed water and with the degree of saturation of the specimens with water.

The water absorption of impregnated strands (Fig. 6) is practically complete by the end of the 1.5–2-month period. By this time, and even a little earlier, the rigidity of the specimens and their strength cease to vary (Fig. 8) and so does the glass-transition temperature of the resin (Fig. 8).

The rigidity and strength of the samples saturated with moisture are less by a factor of 25 and 20%, respectively, than those of the unexposed (dry) samples, and the glass-transition temperature of the resin (in the plastic) diminishes by 32–35° during the aging in water.

From Fig. 5 it also follows that the character of the curves describing the loss in the strength of glass fiber plastic and in the adhesive strength in its unit cell is the same.

Using the data obtained, we will try to answer the question: How could the loss in the adhesive strength of resin joints exposed to a liquid be accounted for? To do this, let us consider the possible causes of the loss in adhesion.

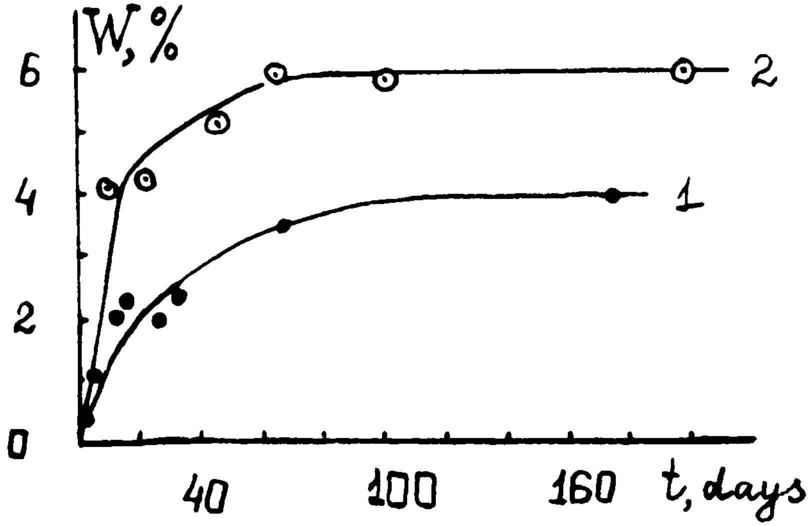


FIGURE 6 The water absorption of an epoxy resin (curve 1) and of an impregnated strand based on the same epoxy resin (curve 2) upon exposure of specimens to water at 20°C. In plotting curve 2 the amount of sorbed water is referred to the mass of the resin.

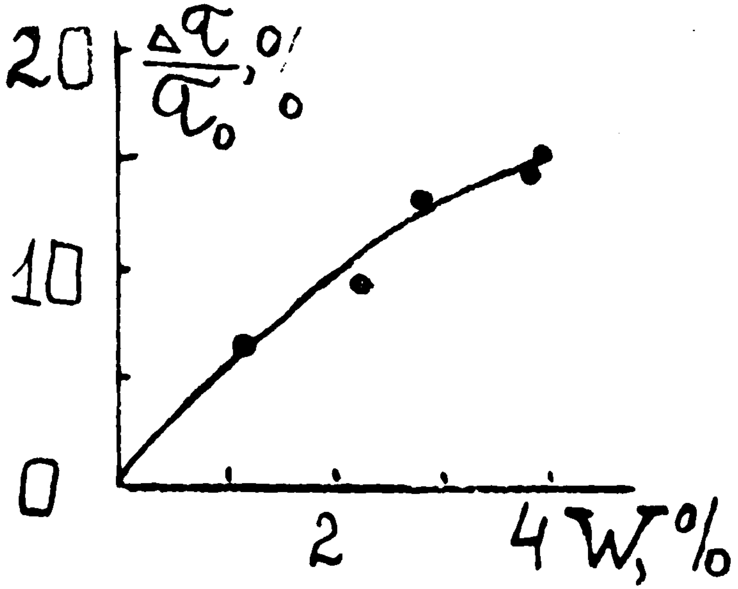


FIGURE 7 The decrease of the adhesive strength as a function of the water absorption of the resin. For boron fiber/epoxy resin joints. System No. 7.

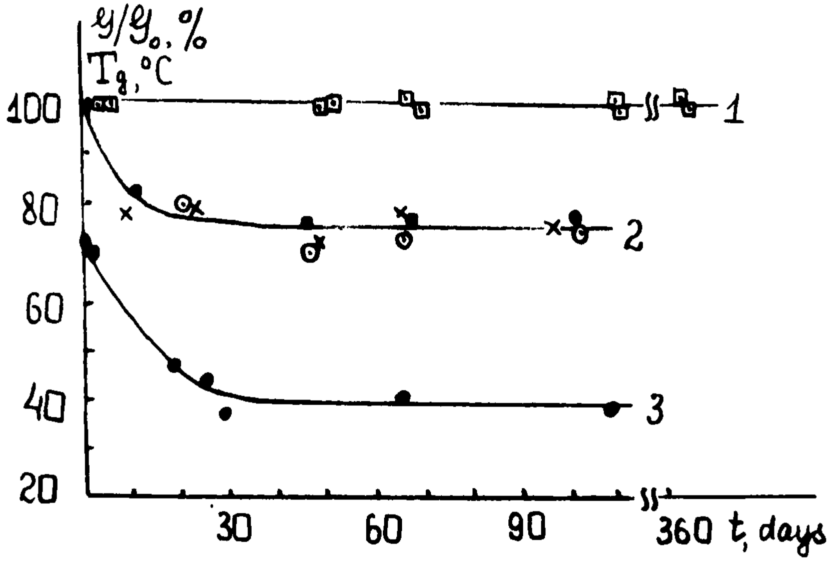


FIGURE 8 The effect of the time of exposure to water on the rigidity (2) of an epoxy impregnated strand and on the glass transition temperature of the resin (3). Curve 1 represents the variation of the rigidity of specimens exposed to air. The symbols on curve 2 refer to specimens with different initial rigidity.

1. Below the glass transition temperature of the resin, at the polymer-filler interface there exists an inhomogeneous field of residual stresses. These stresses are operative during the entire aging process. It is known (see, for example, Refs. 17–19) that the prolonged action of the stress brings about a loss in strength. If this factor were predominant in the case under consideration, the loss in strength after the samples were exposed to water would not exceed the loss of their strength upon aging in air, since the water absorption brings about a decrease in residual stresses (see below). The data given in Fig. 5 show that the action of residual stresses cannot be the main cause of the loss in adhesive strength.

2. Strictly speaking, the exposure process may be accompanied by a change in the chemical structure of the resin as a result of further structuring and degradation processes. The processes of formation and breakdown of the network cross-links may also be accompanied, generally speaking, by the formation and rupture of the bonds at the interface. Apart from this, the structural change may also lead to a change in the physico-mechanical properties of the polymer and, hence, to a change in residual stresses and, as a result of the latter, to a change in the value of adhesive strength measured.

In the case studied the samples were cured to a possible maximum degree, α. Neither additional curing nor destruction of the polymer were observed during the exposure (see Table II): the value of α remained unchanged upon prolonged exposure of the specimens to air and upon exposure to water. Hence, this factor too does not play a significant role in the loss of the strength observed during the aging in the liquid.

3. When the systems are in the liquid, the molecules of the latter diffuse into the bulk of the polymer and along the interface. Being at the interface, they can be adsorbed and/or displace from it the resin molecules (preferential adsorption). This may lead to a decrease in τ values. Moreover, penetrating to the interface, the liquid may produce a disjoining pressure. This may also contribute to the rupture of the bonds and to an increase of the defectiveness of the joint and, hence, to a loss in its strength. However, the supposition that the main factor responsible for the loss of the adhesive strength τ is the presence of liquid molecules at the interfacial boundary is in disagreement with data on the dependence of the kinetics of the loss in τ on the path of moisture penetration (see Fig. 4). Indeed, as has already been said, the molecules of the liquid can arrive at the interface by diffusing along it and through the bulk of the resin (from the bulk of the resin). Then, in order to explain the data of Fig. 4, it should be assumed that the interface itself is not the path of "facilitated" propagation of the liquid, a channel along which the water molecules move freely, and that the diffusion from the bulk of the resin to the interface occurs at a rate which, in any case, is not less than that along the fiber. Indeed, if the rate of surface diffusion along the fiber (along the interface) were substantially higher than the velocity of penetration from the bulk, the kinetics of variation of the adhesive strength (up to saturation) would be either the same for all the systems or at a minimum in system No. 3. However, this is not observed in experiments. When the water molecules have a chance to arrive at the interface not only directly along the fiber but also from the bulk—systems Nos. 3, 4, 6, 7—the loss in adhesive strength begins much earlier than in system No. 2, where only surface diffusion is possible.

However, the supposition concerning the low rate of diffusion of water molecules along the interface contradicts the existing conceptions: according to the data reported in Ref. 20, the velocity of propagation of water molecules along the fibrous reinforcing filler (glass fiber/polymer interface) is 10^3–10^4 higher than the rate of diffusion into the polymer matrix. Moreover, one more thing must be added. The lower end of the joints studied by us, *i.e.*, the end to which is applied an external force upon loading, is the site of maximum stress concentration. It has been shown[21] that in the vicinity of this point (upon application of an external load) there is a "singularity," *i.e.*, this end may be regarded as the site of a dangerous defect. It can be shown that this singularity also results from the action of temperature-induced residual stresses. If we assume, as is usually done, that the fracture begins at the site of maximum stress concentration, then the conditions that exist exactly at this site (at the site of the dangerous defect) become especially important. From this standpoint, in the case under discussion, the adhesive strength and the kinetics of its variation will be determined by the presence of water molecules at the site where the fiber emerges from the joint. At this site, in all the systems, except system No. 3, the molecules of the liquid are present from the beginning of exposure. Therefore, the kinetics of variation of adhesive strength in all the systems studied (except system No. 3) should have been the same. If the rate of diffusion of water molecules along the interface is high, then the kinetics of the loss of adhesive strength in system No. 3 must not differ from that in all the other systems. But if the rate of diffusion along the interface is low, then in system No. 3 the value of τ must remain unchanged for a long time. As we see, experiment reveals a different picture. Thus, the results obtained cannot be accounted for only by the presence of the molecules of the liquid at the interface.

4. While penetrating into the polymer, the liquid can plasticize it. Plasticization of epoxy matrices by water has been extensively studied (see, for example, Refs. 22–26). Plasticization is known to lower the glass transition temperature of polymers. Such a decrease must cause a loss in adhesive strength and also in the physico-mechanical characteristics of the polymer measured at room temperature, since in such a case the test temperature is found to be closer to the glass transition temperature of the polymer, and upon approach to the region of transition of the polymer from the glassy state to the rubbery state its mechanical characteristics and the adhesive strength of the joint composed of the given polymer and fibers,[1,27] and any other substrates,[28] are also diminished. Thus, plasticization can cause a loss in adhesive strength. On the other hand, plasticization lowers the rigidity of the polymer, its elastic modulus and, hence, residual stresses[29] that arise at the interface when the joints are cooled from the curing temperature to the measurement temperature (due to the difference in the mechanical and thermal characteristics of the adhesive and substrate). The decrease of residual stresses causes an increase in adhesive strength in fiber/polymer joints.

The possibility of the formation of bonds directly between the plasticizer molecules and the substrate, which must contribute to an increase in adhesive strength, cannot be excluded either.

Thus, three competing mechanisms are operative upon plasticization of the polymer. One of these contributes to the decrease of τ values and the other two to their increase. As a result, the value of adhesive strength can vary in a complicated way, in particular along a curve with a maximum. This is what is observed, for example, when epoxy resins are modified by active diluents.[1]

The plasticization mechanism is supported by the data of Fig. 4 and by the direct relationship (noted earlier (Fig. 7)) between the loss in adhesive strength and the amount of sorbed water.

The amount of sorbed water is determined by the diffusion (through the bulk of the resin) rather than by the capillary (along the interface) saturation. This has been convincingly demonstrated by Brewis *et al.*,[30] who studied the process of penetration of water into adhesive-bonded Al/epoxy resin/Al joints using the tracer technique, and found that water penetrates the joint predominantly through the bulk of the resin, and that the diffusion at the interface does not make a substantial contribution to the water absorption of the specimens.

In order to prove that the loss in τ observed by us may be associated only with a change in the physico-mechanical characteristics of the adhesive (because of plasticization), and can occur without active participation of water molecules in the interaction through the interface, one must carry out an independent experiment in which there could be observed an equivalent change of the physico-mechanical characteristics of the polymer, with the nature of the interaction (the nature of the forces) remaining unchanged at the interface. As an example of the closest model, let us consider the variation of the mechanical properties of the polymer and its adhesive strength with a change in the test temperature. Figure 9 shows the curves of the relative variation of the adhesive strength and rigidity of the polymer studied with increasing test temperature. The quantities τ and G were measured within a temperature range of 5–150°C on samples identical to those used for studying the effect of water. From this figure it follows that the values of τ and G fall off almost identically with increasing temperature. The similar manner of variation of the

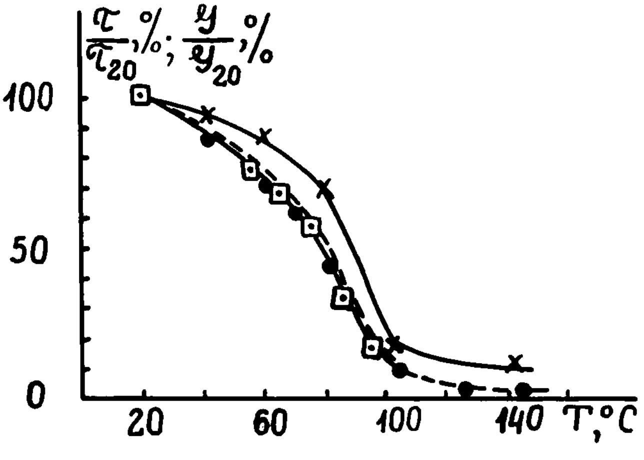


FIGURE 9 Temperature dependence of adhesive strength (□, χ) of fiber/epoxy polymer systems and of the rigidity (·) of an impregnated strand based on this resin. Systems: ▣ = with boron fibers; χ = with alkaline-free glass fibers. The values of τ and G at 20°C are assumed to be equal to 100%.

adhesive strength and mechanical properties of the polymer has also been observed earlier by us.[1,12]

As we have seen (see Fig. 8), the plasticization of the polymer during exposure to water lowers the glass-transition temperature from 73 to 38°C. Such a decrease is equivalent to a rise in temperature of 35°C. From Fig. 9 it follows that with such a temperature rise the loss in adhesive strength in systems with boron fibers is 25–28%. The rigidity of the polymer resin practically falls off as well. It has been shown above (Figs. 3 and 4) that the maximal loss in τ during prolonged exposure of these systems to water does not exceed 20%. Thus, only the change of the mechanical properties of the polymer (without any additional change in the interaction at the interface) outweighs the effect of water. Of course, the change of the mechanical characteristics of the polymer with increasing test temperature is not identical with their change upon plasticization by water. However, in our opinion, the data obtained provide sufficiently convincing evidence in favor of the determining role of plasticization.

The conclusion arrived at is valid for the aging of epoxy resin/boron fiber joints in water. Boron fibers are only slightly subject to the action of water. (In our experiments, after 160-day exposure to water the average strength of boron fibers remains practically unchanged).

Upon prolonged exposure to water of systems consisting of the same resin and alkaline-free glass fibers the loss in adhesive strength is 43% (see Table II). The glass transition temperature of the resin falls off in such a case by 35°C (Fig. 8). However, from Fig. 9 it follows that the equivalent (35°C) rise of the test temperature lowers the adhesive strength of glass fiber/resin systems by only 17%. Probably, in this case, the processes associated with the effect of water on the surface of glass fibers play a substantial role. (It is known that the prolonged action of moisture leads to a chemical damage of glass, its leaching.) This problem requires a more detailed investigation.

CONCLUSIONS

We studied the effect of the time of exposure to water on the interfacial shear strength of systems composed of various fibers (glass, steel and boron) and an epoxy resin and on the physico-mechanical characteristics of impregnated strands based on glass fibers of alkaline-free composition and the same resin. We have found that:

1. The kinetics of the variation of adhesive strength in systems with different paths of water diffusion is different.
2. The loss of adhesive strength becomes greater with increasing amount of sorbed water.
3. The kinetics of variation of the strength of an impregnated strand and of the adhesive strength of glass fiber/resin systems are similar.
4. The main factor that determines the loss in adhesive strength upon aging of the joints in water is the plasticization of an epoxy resin by water.

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