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Mechanical and microstructural properties of notched E-glass/vinyl ester composite materials subjected to the environment and a sustained load

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Abstract

Flaws may be created in composite materials during manufacturing, machining, or use. Composites with such flaws, when subjected to adverse environmental conditions in the presence of a sustained load, may have reduced mechanical properties and changes in their microstructural properties up to failure. E-glass/vinyl ester composite coupons with a single edge flaw have been conditioned for periods up to 7900 h at room temperature and at an elevated temperature, with and without a sustained load, to determine the changes in the tensile properties and damage mechanisms which occur with the introduction of the flaw. The early damage mechanisms have been found to change from transverse matrix cracking, fiber cracking, and occasional edge delaminations for unnotched specimens to the growth of a significant matrix crack from the notch tip and well-defined longitudinal tow debonding for notched specimens.

Keywords: Environmental durability; Mechanical properties; Composites

1. Introduction

Polymeric composites are being investigated for use in a variety of structural applications in which they will be subjected to adverse environmental conditions while under load. Flaws may be created in these composites during fabrication, machining, or use. Although nondestructive evaluation techniques may typically be used to detect flaws prior to use of a component, it is possible that seemingly minor flaws may go undetected until the use conditions have resulted in significant damage growth in the vicinity of a flaw. The combination of moisture and temperature has previously been found [1–6] to degrade the mechanical properties of composite materials by increasing damage growth. The addition of load exacerbates the loss of strength and the amount of damage growth [7]. Little work has previously been done to determine the further changes that occur when the expected use conditions are applied to a composite part with a preexisting flaw [8]. However, study of this combination of conditions is necessary to determine the changes in the moisture uptake and resultant damage, as well as the changes in damage modes, prior to catastrophic failure of a part.

2. Experiments

2.1. Materials

The composite specimens consisted of Derakane 411-350 vinyl ester resin reinforced with E-glass fibers. The fiber architectures studied were woven roving and cross-ply, with and without a chopped strand mat. The composites were fabricated by Seemann's composite resin infusion molding process (SCRIMP). The nominal thicknesses of the panels were 0.36, 0.56, and 0.44 cm, respectively, for the different fiber architectures. The layups were $[0/90]^f_8$, $[90/0]_{2s}$, and $[0/90]_8$, respectively, where superscript f denotes woven fabric. The panels had fiber volume fractions of approximately 52, 54, and 43%, respectively. The materials, which had an ambient cure, were post-cured at 120°C for 2.5 h prior to conditioning.

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2.2. Apparatus

The test setup and procedure have been described elsewhere [7]. Notch lengths were 11% of the sample width for coupons which contained the random mat; for the other fiber architectures, notches were approximately 28% of the sample width. Short notches were cut with a file and razor blade, while long notches were cut with a water jet, diamond wire, and razor blade. The nominal notch tip radius was 0.04 mm. The environmental conditioning was performed in tanks which were specifically created for this purpose. Test coupons were conditioned in water at 21°C and at 57°C for up to 7900 h, although conditioning of most coupons was performed for a maximum of 5000 h. The tanks were initially filled with tap water, which was replaced as it evaporated with deionized water to maintain constant mineral constant. The pH of the water was typically in the range of 8.0–8.6. The sustained load chosen for each fiber architecture was approximately 30% of the failure load of an unconditioned, notched coupon and was provided by specially designed pneumatic load frames; the nominal failure loads were 3.5×10^4 N for $[90/0]_{2s}$ with chopped strand mat, 1.9×10^4 N for $[0/90]_8$, and 2.7×10^4 N for $[0/90]_8^f$. Coupon dimensions and moduli were measured before and after conditioning. All samples were evaluated by optical microscopy, as seen in Figs. 1 and 2. Some coupons were evaluated by scanning electron microscopy, as in Fig. 5, or by optical microscopy of layers after conditioning, as shown in Figs. 3 and 4; others were tested to failure.

3. Results and discussion

Moisture, elevated temperature, and a sustained load result in a significant degradation of mechanical properties beyond that resulting from moisture and elevated temperature alone [7]. The addition of a significant flaw during conditioning further degrades the tensile strength of the material. The presence of a significant notch also alters the early stages of damage growth from matrix microcracking and localized interfacial cracking to a prominent transverse crack from the notch tip and associated tow debonding along the direction of the applied load. A smaller, less easily detectable, edge notch has been found to significantly exacerbate edge delamination, leading to a decrease in tensile strength and a site of significant damage initiation with further use. Significant damage forms much more quickly in the composites in the presence of flaws. The higher conditioning temperature was found to lead to more rapid damage formation in the presence of a flaw, but the nature of the physical damage formed is common to both testing temperatures. Figs. 1 and 2 show the progression of damage in cross-ply coupons with chopped strand mats. These optical micrographs show both transverse matrix cracking, vertical in the figures, and tow debonding, emanating from the notch tip in the direction of the applied load, less apparent since this damage is not superficial. Figs. 3 and 4 show optical micrographs of layers of cross-ply material to which food coloring has been added to highlight damage, particularly the tow debonding. The two major damage components are also more readily apparent in these figures due to the absence of chopped strand mats. Thus, increasing the conditioning temperature and adding moisture are useful tools in accelerating the aging process to more rapidly assess the damage growth patterns and evaluate patterns of long-term damage growth in a composite part. Quantitative evaluation of the extent of crack growth has not been presented here. Although the physical characteristics of the damage are similar for both testing temperatures, the samples exposed to the elevated temperature undergo much greater chemical aging during the conditioning period. The chemical properties of the resin are also affected by the advancement of cure during conditioning.

In order to assess the hypothesis that adding temperature and moisture accelerates damage growth without altering the damage modes, notched coupons have been tested under load and ambient environmental conditions. Fig. 5 shows that the nature of the damage which is created, in the form of a macroscopic transverse crack and tow debonding, is similar at room temperature and at an elevated temperature, with or without submersion in moisture. It is more extensive for the case of the woven fabric coupons exposed to moisture than for those subjected to ambient conditions due to interaction of the more complex pathways, formed by undulations of tows and greater intermingling of fibers with different orientations, with the absorbed moisture. This difference is not apparent in either of the cross-ply materials.

The presence of moisture may have dual effects on the damage growth. The further moisture absorption resulting from the presence of a flaw may enhance crack growth, particularly interfacial crack growth, by increasing degradation of relatively weak areas of a composite. At the same time, the moisture results in redistributed stresses which may effectively blunt cracks, leading to a reduction in the degree of damage which forms under the influence of an external load. Hence, the macroscopic crack seen in Fig. 8 in the direction of the applied load would be expected to be sharper than those seen in Figs. 1 and 2. The transverse crack is less pronounced in the woven coupon seen in this figure than in Figs. 5 and 6, which show coupons for which conditioning included submersion. However, based on a comparison of the appearances of coupons after conditioning, which indicate that other coupons without a chopped strand mat also show less superficial transverse cracking than those coupons with a chopped strand mat, this difference is believed to be due to the presence of that mat in the coupons shown in Figs. 1 and 2 rather than to the difference in the moisture conditions. The chopped strand mat present in some of the material studied also affects the paths of moisture

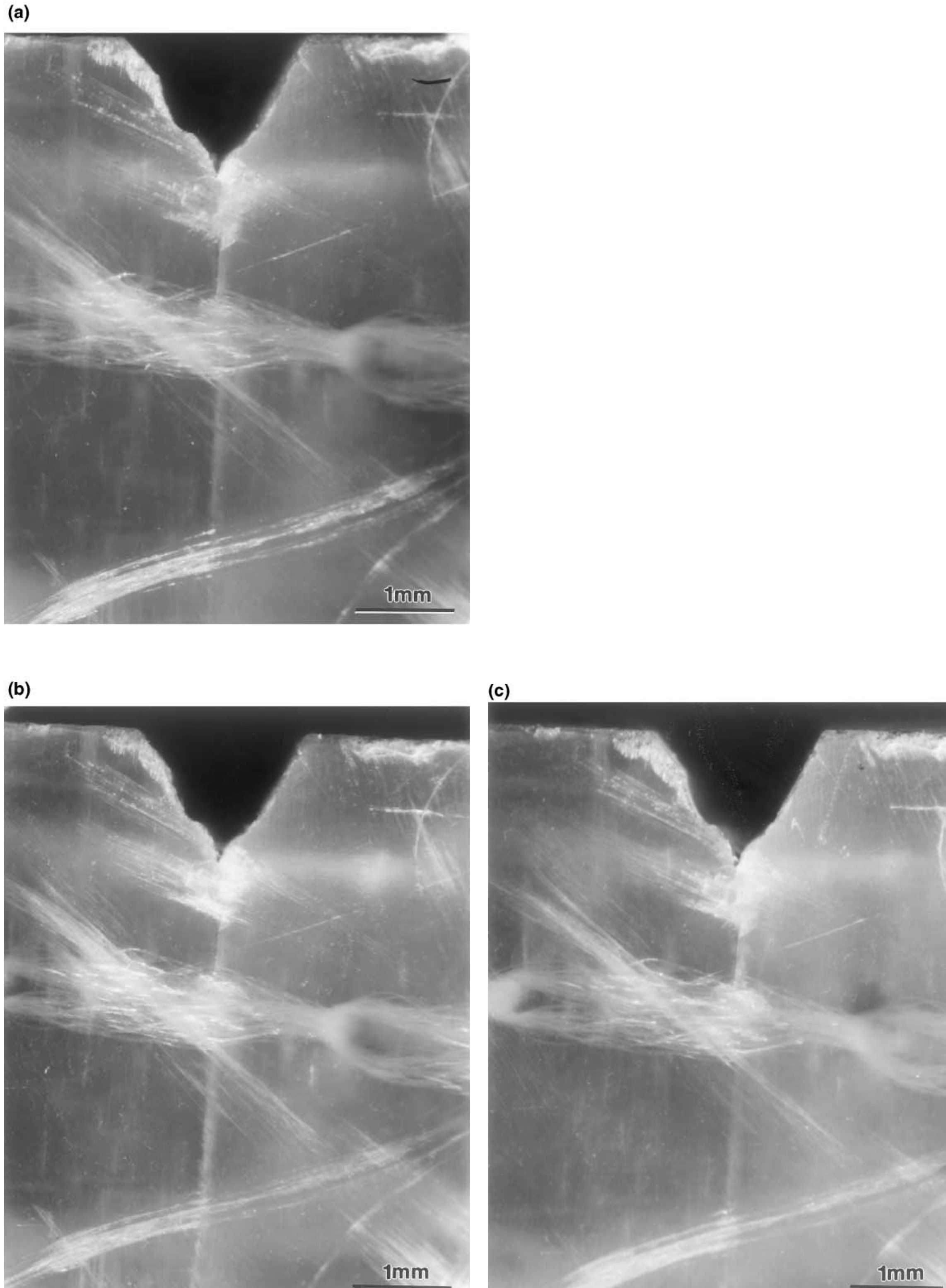

Fig. 1. Optical micrographs of $[90/0]_{2s}$ coupon conditioned at 21°C and 145 MPa for (a) 2500 h, (b) 3000 h, and (c) 5500 h. Layup has chopped strand mat.

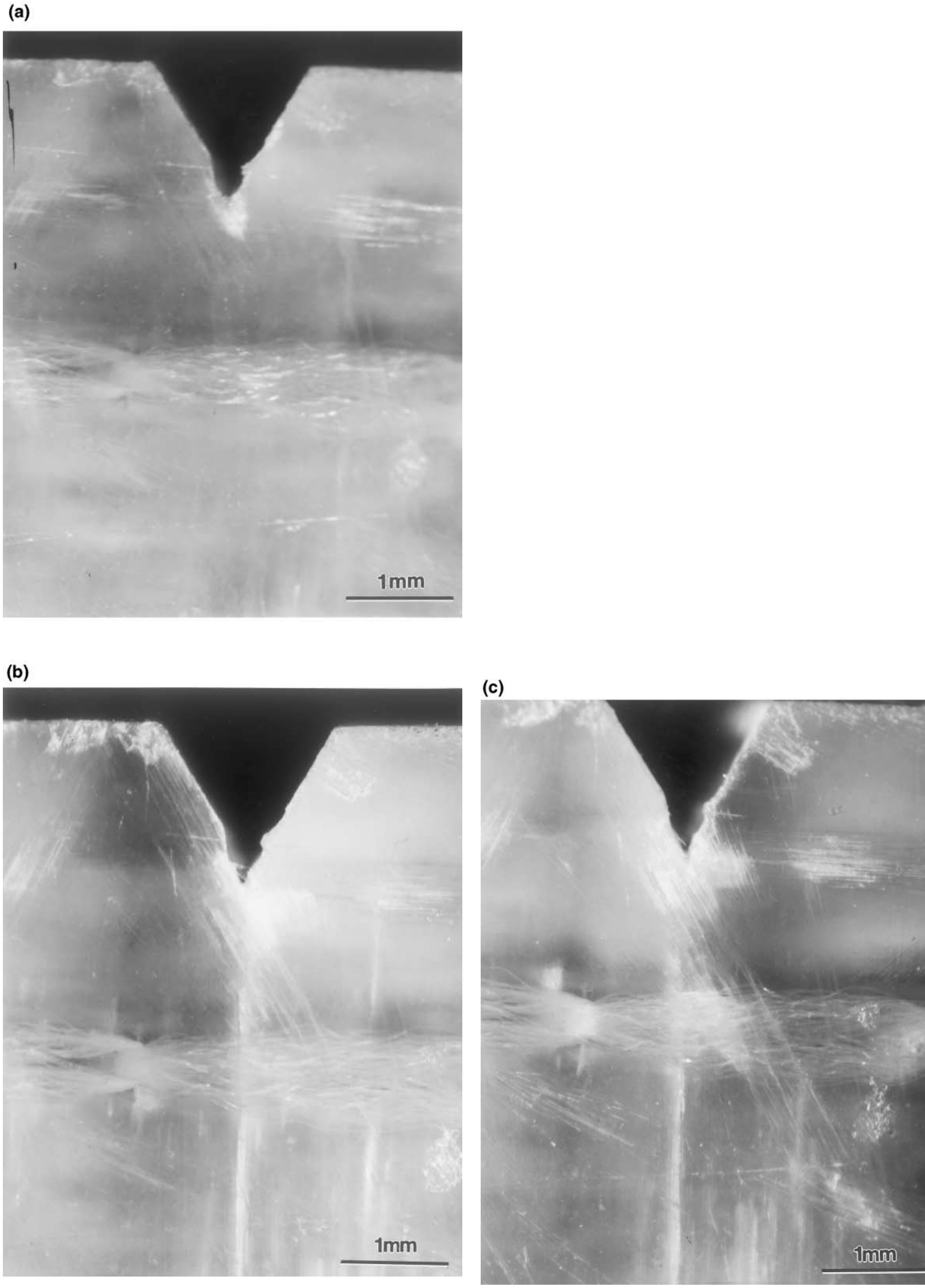

Fig. 2. Optical micrographs of $[90/0]_{2s}$ coupon (a) prior to conditioning and of coupon conditioned at 57°C and 145 MPa for (b) 300 h and (c) 900 h. Layup has chopped strand mat.

uptake by increasing the number of complex pathways for moisture movement, similar to one of the effects of microcracking; however, the damage modes remain similar for these notched coupons. The similarity in the patterns of damage regardless of the precise environmental conditions has demonstrated the validity of accelerating physical macroscopic changes in composites in order to assess the potential long-term problems in a relatively short period of time.

The average load required for tensile failure of an unnotched, unconditioned $[90/0]_{2s}$ coupon with chopped strand mats was 4.5×10^4 N, while that of a notched, unconditioned specimen was 3.5×10^4 N. Decreases in failure load for this fiber architecture with the addition of a notch were greater for higher temperatures and longer conditioning periods, with a nominal decrease of 13% for samples conditioned at room temperature for 3000 h and a nominal decrease of 47% for samples conditioned at 57°C for 1000 h. The modulus was not found to change significantly with conditioning; average values for nonnotched and notched coupons were 27 and 28 GPa, respectively, with S.D. of 1.5 and 2.8 GPa, respectively.

Fig. 7 shows scanning electron micrographs of $[0/90]_8^f$ coupons conditioned at 21 and at 57°C, respectively. These figures show that transverse cracking occurs in matrix-rich regions and along fiber/matrix interfaces. The woven material shows splitting within tows, as

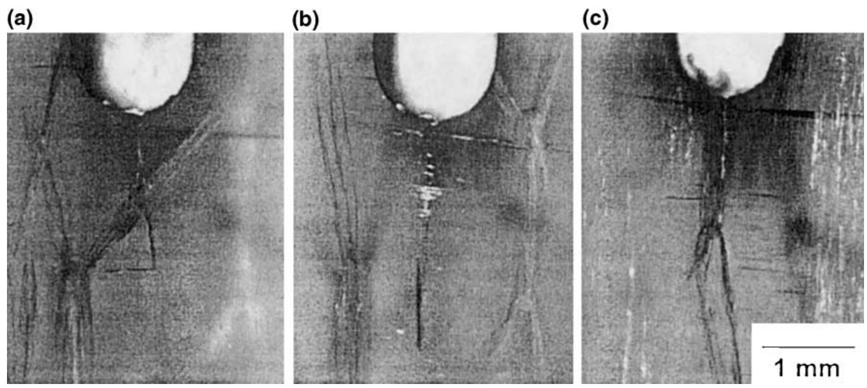

Fig. 3. Optical micrographs of $[0/90]_8$ coupon conditioned at 21°C and 48 MPa for 3001 h. Depth from tool surface: (a) 0.6 mm, (b) 1.1 mm, and (c) 2.4 mm. Food coloring has been used to highlight damage.

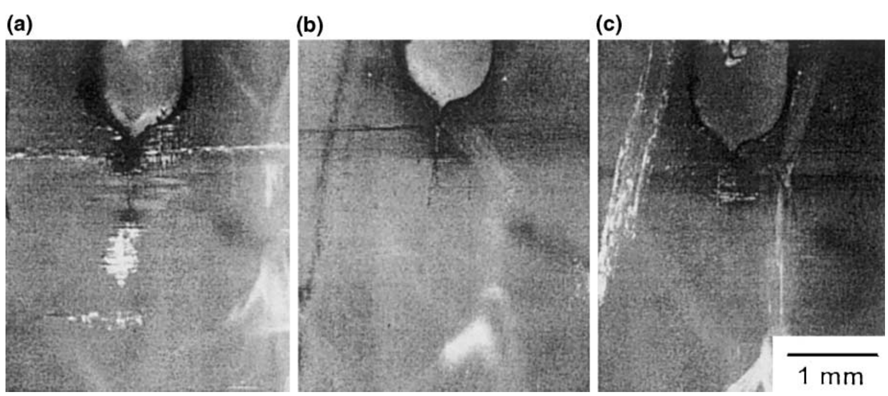

Fig. 4. Optical micrographs of $[0/90]_8$ coupon conditioned at 57°C and 48 MPa for 1006 h. Depth from tool surface: (a) 0.4 mm, (b) 2.2 mm, and (c) 2.6 mm. Food coloring has been used to highlight damage.

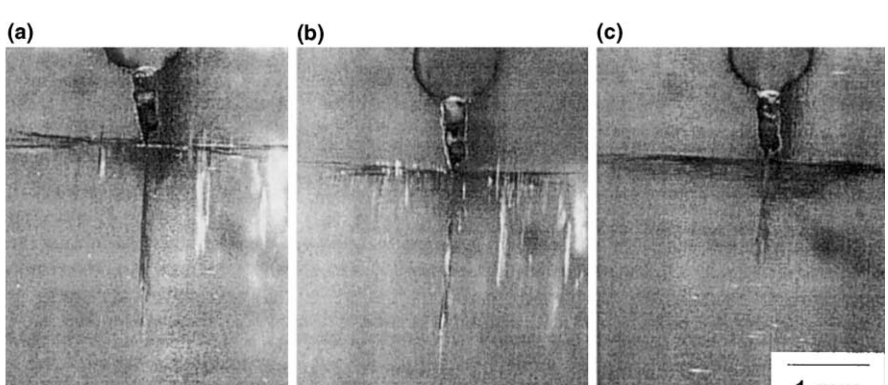

Fig. 5. Optical micrographs of $[0/90]_8^f$ coupon conditioned at 21°C and 97 MPa for 3034 h. Food coloring has been used to highlight damage. Depth from tool surface: (a) 0.8 mm; (b) 1.0 mm; (c) 1.5 mm.

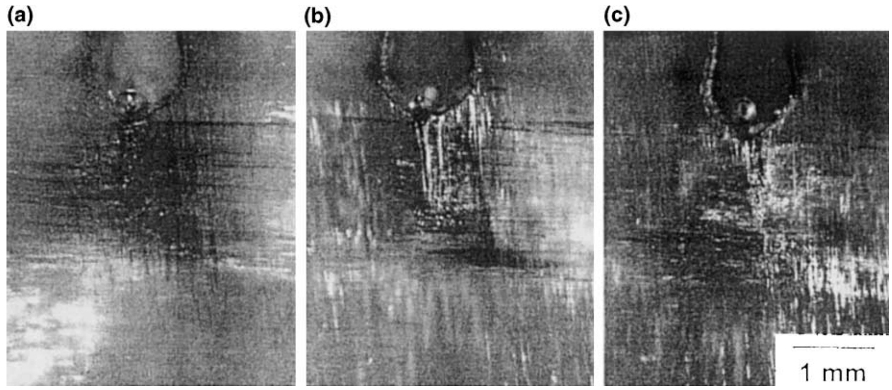

Fig. 6. Optical micrographs of $[0/90]_8^f$ coupon conditioned at 57°C and 97 MPa for 1190 h. Food coloring has been used to highlight damage. Depth from tool surface: (a) 0.4 mm; (b) 0.7 mm; (c) 1.7 mm.

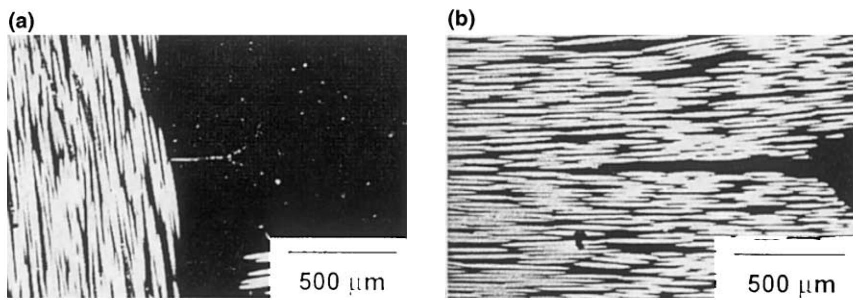

Fig. 7. Scanning electron micrographs of $[0/90]_8^f$ coupons conditioned at 97 MPa. (a) 21°C; 3966 h; (b) 57°C; 1381 h.

depicted in Fig. 7(b). Moisture ingress was found to play a greater role in the damage patterns seen in this material than in those of the other fiber architectures by resulting in greater spreading of damage among tows and greater interaction of damage between tows of different orientations.

The application of a tensile load to notched coupons subjected to adverse environmental conditions alters the nature of moisture absorption. Moisture absorption is often assumed to follow a pseudo-Fickian path in these materials, where Fick's law is given in one dimension by

$$\frac{\partial m}{\partial t} = \frac{\partial}{\partial z}\left(D_z \frac{\partial m}{\partial z}\right),$$

where D is the diffusion coefficient and m is the concentration of moisture. However, for this material, samples subjected to an applied load or to elevated temperature have an altered pattern of moisture absorption, as shown in Fig. 9. Experimental values were obtained for this figure simply by monitoring changes in coupon mass, while the curves shown denote Fick's law, where the diffusion coefficients have been determined from the experimental values. Even coupons exposed to moisture at room temperature with no load do not reach an equilibrium moisture level after a year of conditioning. This material may be expected to follow a two-stage moisture absorption pattern, as has been found for other polymeric composites [9]. However, the differences in the nature of the moisture uptake for the different sets of conditions studied here indicates that the equilibrium moisture levels, the actual level of absorbed moisture compared to the equilibrium level, and the degree of material loss are highly dependent on the test conditions at a given time. These differences contribute significantly to the differences seen in the mechanical and damage properties observed. Thus, accelerating damage growth in the manner described here is a very good qualitative tool, but not necessarily an accurate quantitative one, for estimating the long-term behavior of this material.

The decrease in mass due to loss of residuals and loss of material due to damage growth are countered to an extent by the mass gain due to moisture absorption. With a notch or other macroscopic flaw, as with matrix microcracking, the increased surface area may result in increased damage growth and, by extension, to greater mass loss. As demonstrated in Figs. 1–8, these variations in patterns of moisture uptake do not significantly alter the nature of macroscopic damage growth in the notched coupons.

4. Conclusions

An edge notch, in combination with a sustained load and adverse environmental conditions, leads to more rapid degradation of mechanical properties of E-glass/vinyl ester composite materials than unnotched materials. An edge notch has been found to create a

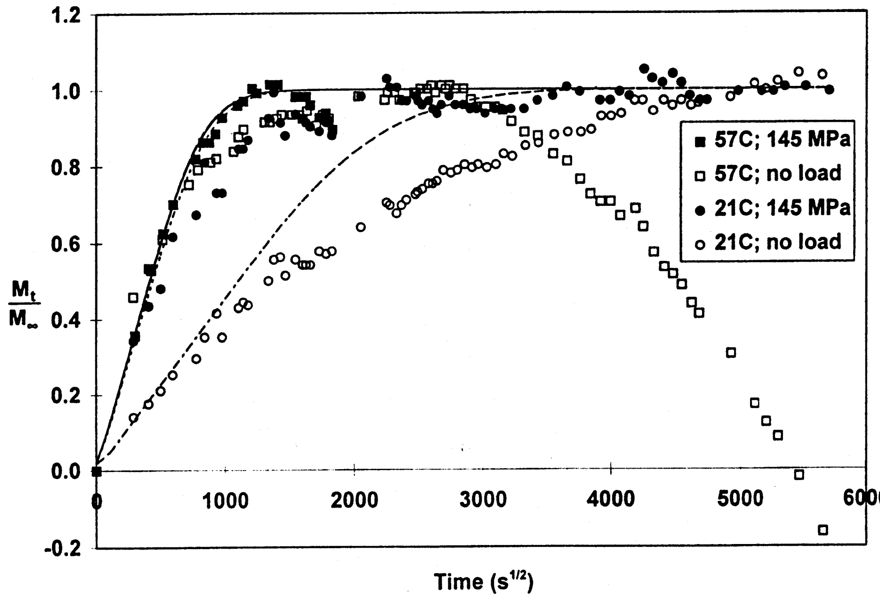

Fig. 9. Moisture absorption in notched $[0/90]_{2s}$ coupons with chopped strand mat.

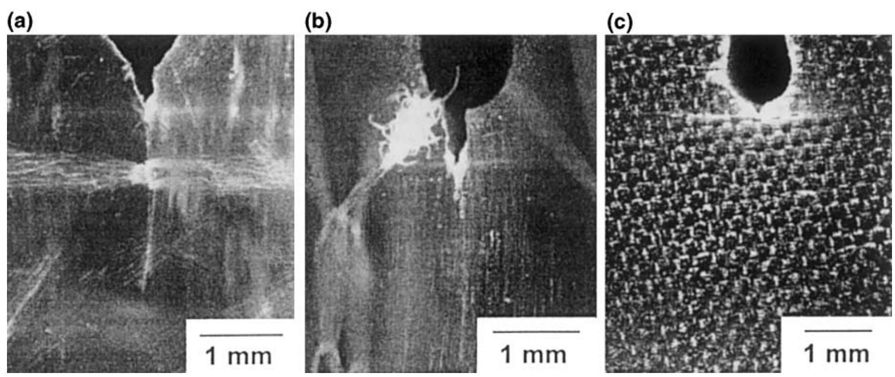

Fig. 8. Optical micrographs of coupons subjected to ambient conditions for 3000 h, (a) 145 MPa; layup is $[90/0]_{2s}$; (b) 48 MPa; layup is $[0/90]_8$; (c) 97 MPa; layup is $[0/90]_8^f$.

two-stage pattern of damage growth, consisting of a significant transverse crack and longitudinal interfacial cracking, in samples conditioned with a sustained load. This damage evolution differs from that found in coupons subjected to similar conditions, but without a significant pre-existing flaw. Moisture uptake in notched coupons typically does not follow a Fickian form, especially for samples subjected to elevated temperatures or a sustained load. In these coupons, increases in mass due to absorbed moisture compete with decreases in mass due to the loss of residuals.

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References

[1] G.S. Springer, B.A. Sanders, R.W. Tung, J. Comp. Mater. 14 (1980) 213.
[2] F.-K. Chang, I. Shahid, R.A. Engdahl, J. Reinf. Plast. Comp. 8 (1989) 106.
[3] C.Y. Lee, M. Pfeifer, B.S. Thompson, M.V. Gandhi, J. Comp. Mater. 23 (1989) 819.
[4] T.K. Tsotsis, Y. Weitsman, J. Reinf. Plast. Comp. 9 (1990) 420.
[5] A. Haque, S. Mahmood, L. Walker, S. Jeelani, J. Reinf. Plast. Comp. 10 (1991) 132.
[6] C.A. Bigelow, Exp. Mech. 29 (1989) 90.
[7] S.E. Buck, D.W. Lischer, S. Nemat-Nasser, J. Comp. Mater. 32 (1998) 874.
[8] P. Lee-Sullivan, G. Lu, Wear 176 (1994) 81.
[9] T.K. Tsotsis, Y. Weitsman, J. Mater. Sci. Lett. 13 (1994) 1635.