Aging of polymeric composite specimens for 5000 hours at elevated pressure and temperature

Thomas K. Tsotsis [a,*], Scott Keller [b], Kyejune Lee [b], Jason Bardis [c], Jack Bish [d]

[a]*The Boeing Company, Mail code C078-0208, 2401 E Wardlow Road, Long Beach, CA, USA*
[b]*UCLA, Los Angeles, CA, USA*
[c]*UCSB, Santa Barbara, CA, USA*
[d]*Friedman Research, Santa Barbara, CA, USA*

Abstract

As part of the NASA High Speed Research Program and as a continuation of a test program developed at Boeing — Long Beach [Tsotsis TK, Keller S, Lee K, Bardis J, Bish J. 3000 hours aging of polymeric composite specimens under elevated presssure and temperature. Presented at 44th International SAMPE Symposium 24–27 May, 1999 (closed paper). Tsotsis TK, Keller S, Bardis J, Bish J. Preliminary examination of the use of elevated pressure to accelerate thermo-oxidative aging in composites. Polym Degrad Stab 1999;64:207–12.], the effects of elevated pressures in conjunction with elevated temperatures on the thermo-oxidative stability of polymeric composites are being investigated. This test program examines four different air pressures — 0.101, 0.345, 1.03, and 1.72 MPa (14.7, 50, 150, and 250 psi)—for carbon/epoxy specimens aged at 121°C (250°F) for up to 5000 h. Specimens used were AS4/3501-6 [±45°]2s tensile shear and IM7/8552 [+45°/0°/−45°/90°]$_{2s}$ open-hole compression. The materials used were selected to evaluate the test method and not as candidate materials for use at higher temperatures. The test results show a distinct accelerating effect with the use of elevated pressures especially for the tensile shear coupons. The results also suggest that elevated pressure may be a good tool for significantly reducing screening times for material that will be subjected to long exposures in oxygen-containing environments at elevated temperatures. © 2000 Elsevier Science Ltd. All rights reserved.

1. Background and objectives

Future high-speed commercial transport (HSCT) applications will be likely to require polymeric composites able to withstand long-term (60,000 h) exposures at elevated temperatures between 177 and 232°C (350 and 450°F). In such applications, the long-term stability is essential because the long-term performance of polymer composites at elevated temperature is dictated by thermal and oxidative stability of the materials. Therefore, it is necessary to understand how the materials will behave during the intended service life when considering polymeric composites for long-term applications. The underlying degradation processes leading to material structure changes are not well known, however, it has

been observed [1–6] that elevated pressures increase the degradation rate of polymeric composites. Furthermore. it has been noted [4] that aging at too high a temperature leads to erroneous calculations of material lifetimes because the mode (or relative modes) of degradation change from what would occur at normal usage temperatures.

The degradation mechanism of polymeric composites subjected to thermo-oxidative aging has been observed [1–5,7] to be a combination of surface degradation and microcracks perpendicular to the surface as shown in Fig. 1. The implications of this are that degradation occurs initially at the outer surface of the composite and proceeds inwards via a diffusion mechanism. Simultaneously, microcracks may occur perpendicular to the composite surface depending on the toughness of the material [5]. These microcracks create additional surface area and diffusion paths. Thus, these additional cracks enhance the rate of degradation.

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* Corresponding author. Tel.: +1-562-593-5583; fax: +1-1-562-496-9377.
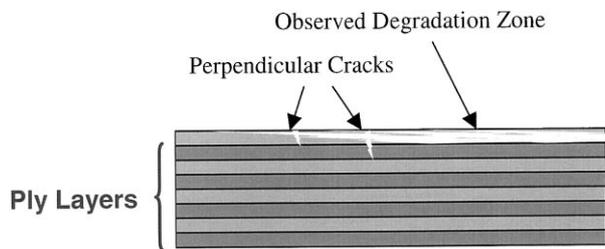*E-mail address:* thomas.tsotsis@boeing.com (T. K. Tsotsis).

Fig. 1. Schematic of transverse microcracking and surface degradation observed in composite laminates as a result of thermo-oxidative aging.

Based on the above understanding, the present work is intended to evaluate the potential of using elevated pressures in conjunction with a 'realistic' use temperature. This work is also an extension of a test program begun by Tsotsis et al. [1,2] in which specimens were aged to 1000 and 3000 h, respectively, at the same conditions used in the present work. This work indicated that elevated pressure had a distinct accelerating effect that will be discussed in greater detail below. An earlier study attempted to quantify the effect of oxygen pressure on polymer degradation through a kinetic model was performed by Ciutacu et al. [8] who applied an Arrhenius model to correlate pressure to mechanical strength loss in glass/epoxy composites. Although the results of Ciutacu et al. agreed well with their model, the dependence on material anisotropy, as noted by Nam and Seferis [10]. which was not considered in their work, does not allow for generalization of their results without further verification.

The recommendations for the type of approach used herein were previously outlined by Tsotsis [3] and more recently by Gates and Grayson [9]. Both of these works stressed the need for accelerating methods that would produce reproducible mechanisms representative of those that would occur during real-time aging at operating conditions.

1.2. Test conditions

In the present work, specimens were aged in air at atmospheric (0.101 MPa or 14.7 psi) pressure and at the elevated pressures of 0.345, 1.03, and 1.72 MPa (50, 150, and 250 psi) all at a temperature of 121°C (250°F). Because the intent of the present work is to accelerate the rate of degradation, no reduced pressures were considered. The temperature of 121°C was selected as the aging temperature for the materials considered, which have glass transition temperatures in the range of 160–190°C (320–370°F). Although aging at 121°C has been observed to degrade very similar resin systems [3], the aging temperature is far enough below the unaged T_g that non-linear effects that occur near the glass transition temperature, T_g, should be avoided.

Non-linear effects are typically observed as a material passes through its glass transition and changes from 'glassy' to 'rubbery' mechanical behavior. These changes most affect mechanical properties such as modulus and toughness, the latter of which has been shown to influence thermo-oxidative aging [5]. Because the modulus change at the glass transition is reflective of chain mobility and free volume in the matrix, non-linearities in the modulus are, it may be assumed, indicative of non-linear changes in these two parameters. In addition, because diffusion rates are related to available free volume and because reaction rates are related to the ability of reactive species to reach reactive sites, chain mobility also influences reaction rate because higher chain mobility increases the likelihood that a reactive site becomes available. Thus aging near or above the glass transition temperature will encounter all of these non-linear changes and thereby make the scaling of any observed changes very difficult if not impossible.

1.3. Test specimens

Compression and shear tests were selected because they are highly reliant on resin properties. By contrast, tensile tests containing 0° fibers are almost completely reliant on the fiber strength. The results of Kerr and Haskins [6] in which tensile coupons that visibly exhibited severe resin degradation had only small decreases in strength even for a $[0°/\pm 45°]_s$ lay-up demonstrate this point. Additionally, Tsotsis [4] directly compares compressive and tensile test properties, among others, for two materials systems similar to those examined in the present study that were exposed to identical aging conditions up to 10,000 h. In these data large drops were observed in compressive properties well before such changes were observed in tensile ones. Similarly, $[\pm 45°]_{2s}$ tensile shear coupons also demonstrated decreases well before $[0°]_8$ ones.

The lay-ups used were selected for the above-mentioned dependence on matrix properties. $\pm 45°$ tensile shear specimens are highly dependent on the matrix shear strength and modulus. Similarly, open-hole compression coupons are known to be dependent on the matrix yield point and modulus. Previous work [3] has shown that compressive and shear properties show much greater decreases in strength than other properties when subjected to elevated temperature in the presence of air.

The high dependence on matrix properties arises because elevated-temperature aging tends to post-cure and degrade thermosetting polymers by causing increased cross-linking or chain scission, respectively. These molecular-level changes cause the macroscopic matrix mechanical properties to change after long-term exposure at elevated temperatures. The rate of degradation as determined by a reduction in strength or stiffness properties is likely a result of the chain scission reaction

taking precedence over the cross-linking reaction. The rates of these reactions change significantly at or above T_g due to increased molecular mobility above this temperature. Therefore, aging these specimens well below their T_g should approximate the reaction rates that would be experienced in actual use environments that are also well below the material T_g, if long-term usage is desired. It is anticipated that. by keeping the rates and mechanisms approximately the same as those that likely to be experienced by a part in actual use, accurate scaling of the lifetime of such parts may be achieved by accelerating via the use of elevated pressures.

The present work does not address the projection of useful lifetimes from the data gathered herein. Rather, it is the intent to demonstrate feasibility of using an elevated-pressure approach to provide data that may be used to develop accelerated aging models.

2. Experimental methods

2.1. Materials

Two materials were selected in the present study: AS4/3501-6 and IM7/8552 carbon/epoxy both from Hexcel Composites, Magna, UT. The AS4/3501-6 was used to make the $[\pm 45°]2s$ shear specimens and the IM7/8552 used to make the $[+45°/0°/-45°/90°]_{2s}$ quasi-isotropic open-hole compression specimens. All panels were cured according to the manufacturer's recommended cure cycles: UCLA processed the AS4/3501-6 panels and the IM7/8552 panels were processed at Boeing—Long Beach. Shear tests were performed according to ASTM D3039.

2.2. Test conditions

The open-hole compression fixture [11] was based on the specimen and fixture in Boeing BSS 7260, which is the same as the SACMA open-hole compression method [12]. This specification requires a specimen larger than required to provide a uniform far-field stress state. The work of Bardis et al. [11] showed how the open-hole compression specimen used in the Boeing or SACMA method could be scaled to reduce its overall length while still providing adequate grip areas and comparable measured properties. Indeed, testing at UCSB confirmed that the specimen failed properly and that the strengths recorded were consistent with past data for the full-size open-hole compression specimens. The specimen
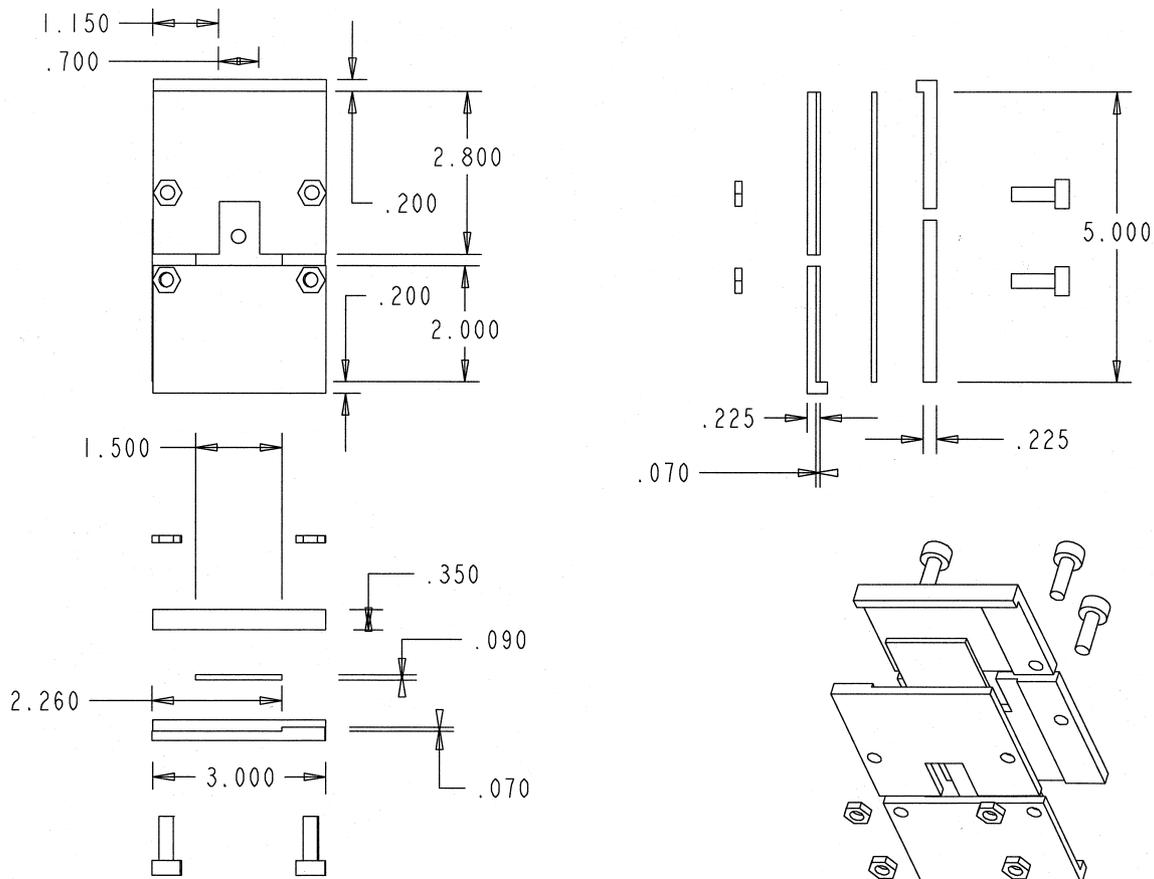

Fig. 2. Exploded view of open-hole compression test fixture.

used was 38.1 mm (1.5 in) wide and contained a 6.35 mm (0.25 inch) diameter hole. Schematics of the specimen and fixture are shown in Figs 2 and 3. Fig. 4 shows the fixture assembled with a specimen.

Pressure chambers manufactured at UCLA were cylinders made from steel with one welded and one bolted end. Pressurized air was continuously supplied to the chambers with the bolted end fitted such that it allowed a slight bleed of air so that the chamber air was continuously refreshed. Calibrated pressure regulators and rupture disks were used to maintain pressure and to prevent over-pressurization from occurring, respectively. Specimens were placed on racks inside the chambers to permit free airflow around each specimen. The chambers were closed and placed in an oven with the ambient-pressure specimens inside the same oven. After each 1000 h of aging time, five specimens from each pressure were removed from the chambers for testing.

Mechanical tests were performed using an MTS hydraulic test machine. Load, crosshead displacement, and ultimate load were recorded for all specimens. Additionally, the $[\pm 45°]_{2s}$ were strain-gauged to record strain during testing to allow for calculation of modulus. Modulus was determined from the linear portion of the stress/strain curve.

All specimens were dried using desiccant at 40°C for at least 2 weeks prior to conditioning in order to record dry weights. Specimen weight changes were verified to have reached equilibrium before removing them from the desiccant and measuring their dry weights.

At each 1000 h interval that weights were measured after aging, the specimens would be removed from the conditioning chambers, placed in desiccant bags, allowed to cool to room temperature, and then weighed. Specimens that were to be conditioned for additional time were returned to the conditioning chambers after they had been weighed. Weight changes were based on the difference from the unconditioned dry weight.

3. Results and discussion

3.1. Tensile shear specimens

Measurements of the ultimate strength changes in AS4/3501-6 $[\pm 45°]_{2s}$ specimens showed significant differences between the different aging conditions. Strength losses were significantly higher for the elevated-pressure samples than for the ambient-pressure ones as shown in Fig. 5. In this figure, an offset is used for the exposure time between the various pressure exposures to improve the readability of the graph. Corresponding weight loss data and modulus change data are shown in Figs. 6 and 7, respectively.
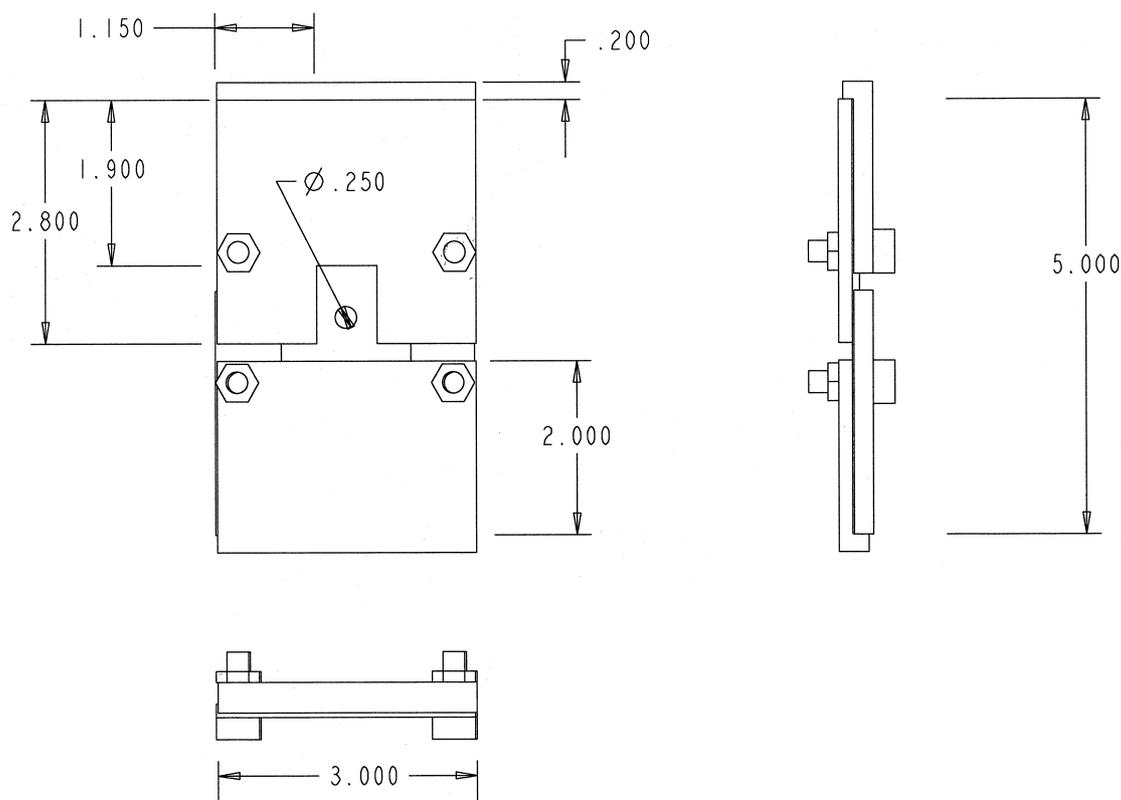

Fig. 3. Drawing of assembled open-hole compression test fixture.

In the tensile shear specimens, the differences in the degradation rates become particularly dramatic after 2000 h exposure as measured by their residual strengths. At this point, the ambient-pressure-aged specimens have virtually no loss in their strength versus the unaged specimens and furthermore show little scatter in their strength values. By contrast, the specimens aged at the elevated pressures all show strength reductions of greater than 15% with the specimens aged at 1.72 MPa having nearly a 50% reduction in strength. Moreover, all of the specimens aged at elevated pressure show a very large scatter in the strength values. This scatter is larger than was observed at 1000 h exposure time.

After 3000 h, both the 1.08 and 1.72 MPa specimens show extremely large scatter bands. The magnitude of these is indicative of both the brittleness of the material after aging and the varying degree to which transverse cracks are likely to have formed in these specimens. Moreover, the strength has decreased to as little as 30% of the unaged value for some specimens aged at 1.72 MPa, though the average strength remained essentially unchanged from its value at 2000 h. The 1.08 MPa specimens
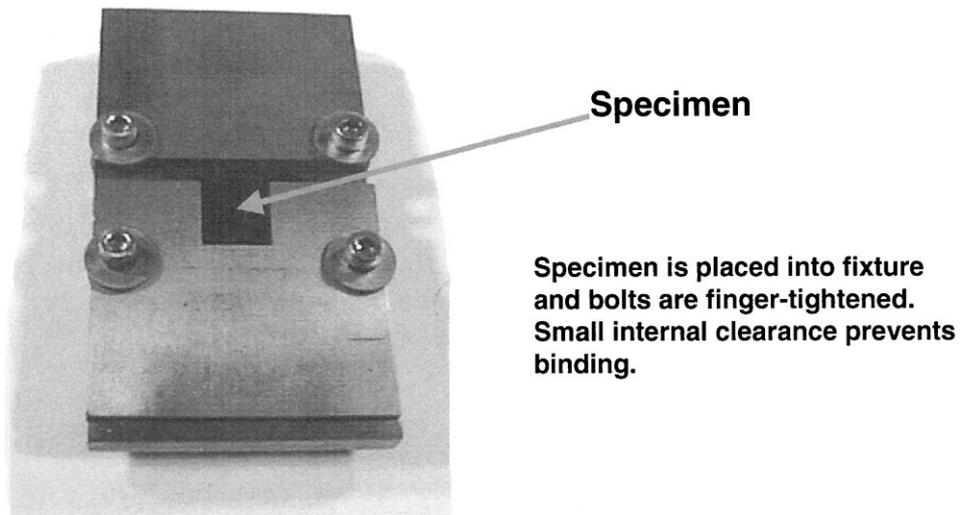

Fig. 4. View of open-hole compression test fixture with a specimen placed into fixture.
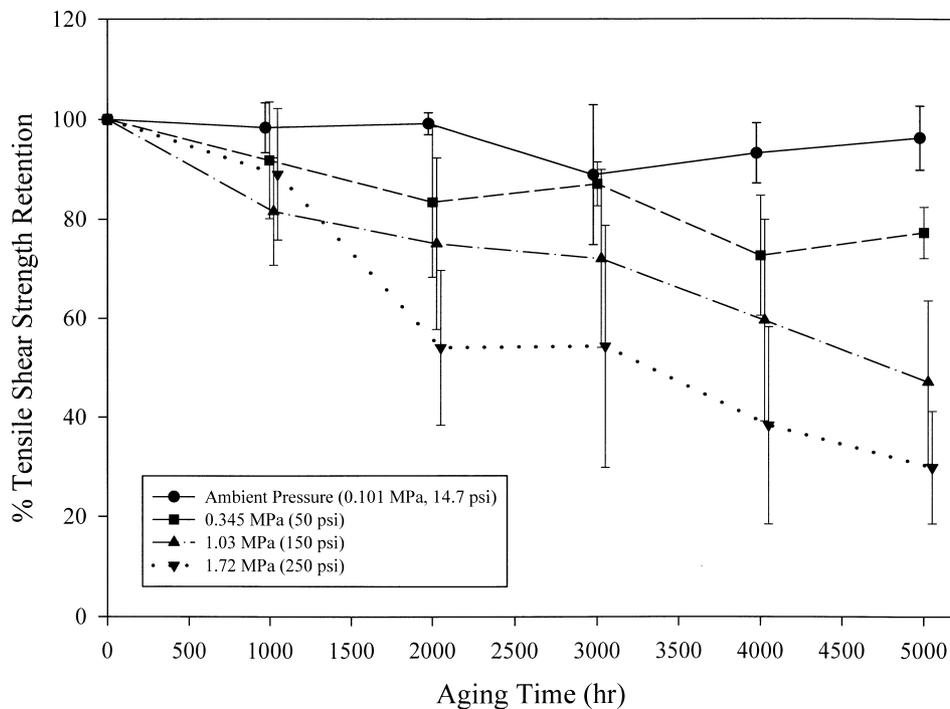

Fig. 5. Percent tensile shear, $[\pm45°]_{2s}$, retention versus aging time in air at various pressures and 121°C for AS4/3501-6 test specimens.
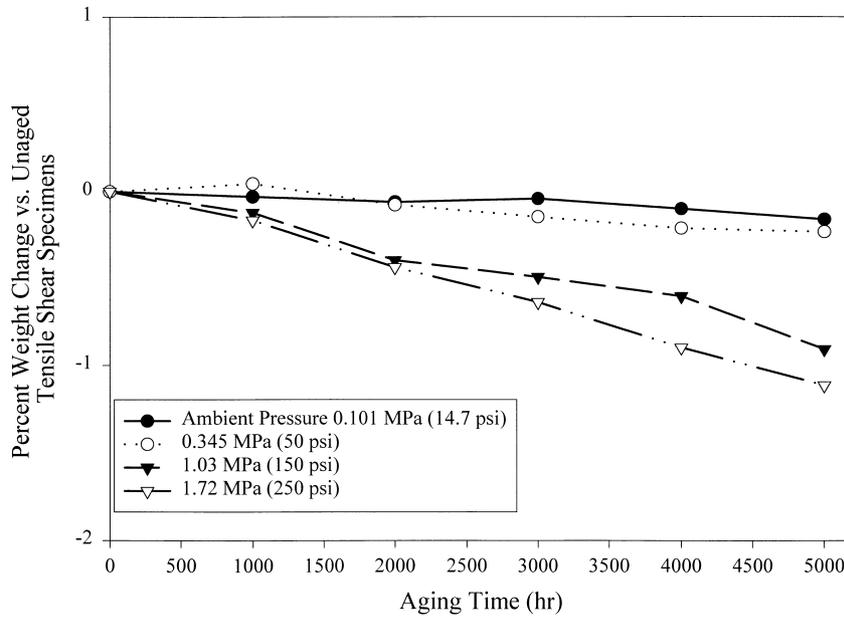
Fig. 6. Percent weight change in tensile shear specimens, $[\pm 45°]_{2s}$, versus aging time in air at various pressures and 121°C for AS4/3501-6.
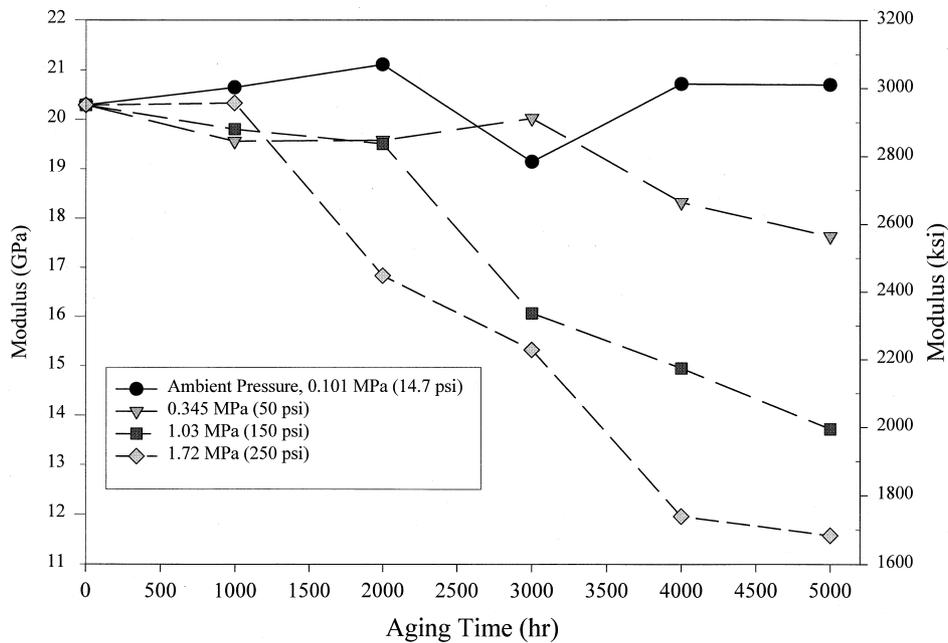

Fig. 7. Modulus of tensile shear, $[\pm 45°]_{2s}$, specimens versus aging time in air at various pressures and 121°C for AS4/3501-6.

appear to have a fairly steady reduction in strength with time, whereas the 0.101 and 0.345 MPa specimens show little trend through 3000 h.

The mechanical strength drop-off also corresponds to the weight loss data shown in Fig. 5. In this figure, weight changes correlate directly to the level of air pressure applied: as the pressure is increased, the amount of weight loss also increases. It may appear that an exception to this is observed at 1000 h aging where a slight weight increase is seen in the 0.345 MPa specimens. This is a likely result of the two competing reactions

taking place due to oxidation: oxygen uptake leading to weight gain and chain scission leading to weight loss. Based on this hypothesis. only after greater than 1000 h do the 0.345 MPa specimens have a weight loss rate sufficiently great to overcome the weight gain due to oxygen uptake.

The two pressures showing the greatest weight loss — 1.03 and 1.72 MPa — also show the greatest property loss. More significant is the fact that at these two pressures the weight losses are significantly greater than at the other lower pressures. Because large property losses

accompany these weight losses, it may be inferred that the mechanisms that cause weight loss, such as transverse microcracking. are also increasing weight loss. Such synergy has been observed previously [2,3].

The change in modulus shown in Fig. 7 closely follows the trends seen for the strength and weight change shown in Figs. 5 and 6, respectively. In fact, the modulus change is more sudden than for either strength or weight loss with a sharp increase in the rate of decrease of modulus occurring after 1000 h for the specimens aged at 1.72 MPa and after 2000 h for the specimens aged at 1.03 MPa.

The decrease in strength and modulus is consistent with the degradation mechanism shown in Fig. 1. As the outer layers of the resin degrade, the fibers are no longer able to carry load or at least are less able to carry load than without any resin degradation. This reduces the strength by reducing the number of load-carrying fibers. Because the modulus is calculated from the initial cross-sectional area and the effective load-carrying area has been reduced as described above, the modulus also will decrease as observed.

This mode of degradation is also consistent with the observations on the failure modes of the $[\pm45]_{2s}$ coupons. These specimens showed greater evidence of 'blooming' of the fibers after failure as aging times increased. Furthermore, a greater amount of powder was seen on the surfaces of the specimens as aging time increased. Both the tensile shear and open-hole compression specimens showed a discoloration due to aging in which the specimens assumed a brownish tinge instead of the as-cured black.

3.2. Open-hole compression specimens

The open-hole compression data, like the tensile shear data, also show a marked increase (from approximately 3% at 1000 h to 5% after 4000–5000 h) in the amount of scatter in the ultimate strength values versus the unaged data, especially at the higher pressures as shown in Fig. 8 but by a far smaller degree than the tensile shear specimens. The causes for this are multiple. The postcure of the resin increases the modulus and correspondingly the brittleness of the resin, however this is not the principal reason for the observed behavior as is discussed below.

Thermo-oxidative aging does degrade an outer layer of the material as well as causing transverse microcracks as noted above. Although the outer-layer degradation may be somewhat uniform (because the medium through which the air (oxygen) diffuses is largely uniform along the length of the specimens), the formation of the transverse microcracks is likely dependent on local defects and stochastic processes. As such, the amount and location of these microcracks will necessarily vary from specimen to specimen. The increased scatter in the data at longer exposure times suggests that the potential for large strength degradation increases significantly with increasing thermo-oxidative aging.

Despite some misleading trends observed at earlier times (discussed below), after 5000 h there is a strong correlation between pressure and open-hole compression strength loss vs. aging time as shown in Fig. 8, with the exception of the ambient pressure results also discussed below. The observation that significant degradation
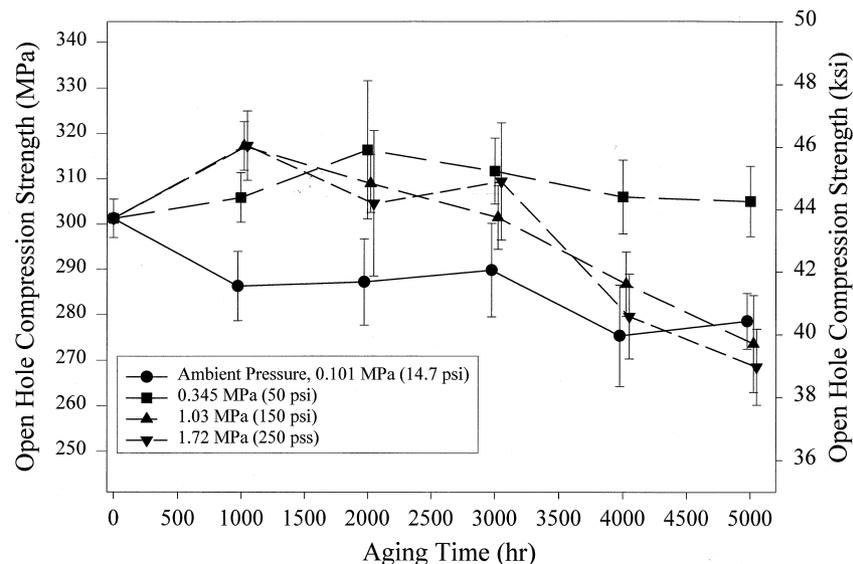

Fig. 8. Open-hole compression strength change versus aging time in air at various pressures and 121°C for 1M7/8552 test specimens.

occurs only after several thousand hours is consistent with earlier work [4]. However, in the present case, the elevated pressures appear to cause this point to occur after 3000–4000 h instead of after 5000 h.

The lack of degradation up to 3000 h may be explained by the fact that the plies that dominate mechanical behavior—the 0° plies—are at least one ply removed from the specimen surface. Because of this, until this ply becomes degraded, there will likely be no significant decrease in open-hole compression strength. The results shown in Fig. 8 are consistent with this hypothesized mechanism.

It appears, from the observed behavior, that degradation around the hole relative the the effect of the original hole itself does not appear to have a strong effect on the measured behavior. As will be discussed later, the measured relationship between the modulus and the weight change support this point as no significant changes in modulus are noted until a critical weight loss has been reached. It is, however, possible that degradation around the hole does indeed contribute to this property loss. Unfortunately, the results gathered herein are not sufficient to determine this effect.

Results from open-hole compression tests of the aged IM7/8552 specimens shown in Fig. 8 showed no significant changes in strength from the unaged specimens after 3000 h exposure. Indeed, an increase in strength was noted for all of the elevated pressure samples. The ambient pressure data are misleading. These specimens were aged in a separate oven from the other specimens and were inadvertently exposed to temperatures above 121°C as well as to greater convective currents than the other specimens. The temperature excursion above 121°C occurred in the first 1000 h of aging. After this

excursion had been discovered, an improved temperature controller was placed on this oven and further such anomalies were avoided as noted by the reduced weight change rate after the first 1000 h. Unfortunately, the exact temperature excursion and duration were not identified. For these reasons, the ambient-pressure specimens showed greater strength degradation than the others.

In contrast to the shear tests, the results of the open-hole compression tests indicate that aging for up to 3000 h is insufficient for the observation of any degradation of this mechanical property in IM7/8552 composites. This result is consistent with previously observed behavior [3] for aging at 177°C of a similar material (Hexcel G30-500/6376 carbon/epoxy) in which a significant reduction in open-hole compression strength was not observed until 5000 h exposure.

Unlike the strength results the weight loss data shown in Fig. 9 strongly correlate with the applied pressure. These data do support the hypothesis that the elevated pressure indeed accelerates degradation. Indeed, the weight loss at 1.72 MPa is nearly twice that at 0.345 MPa after 3000 h. Because the mechanical test data described above clearly show that there is not yet any corresponding significant change in mechanical strength accompanying the weight loss, it may be inferred that most of the degradation has occurred in the outer ply of material.

Additionally, the open-hole compression specimens did not display any change in failure mode with increasing aging time as did the tensile shear coupons. Like the tensile shear specimens, however, a change of the specimen color from black to brownish was observed.
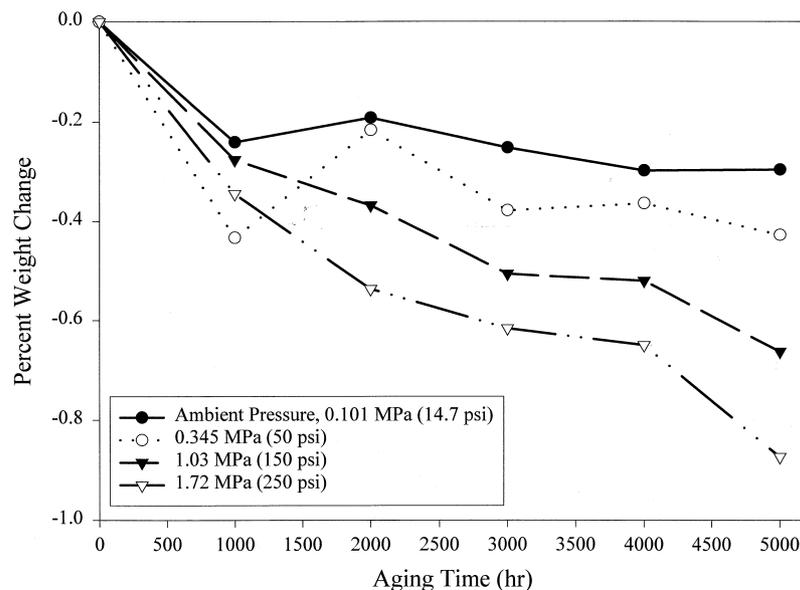

Fig. 9. Percent weight change in open-hole compression specimens versus aging time in air at various pressures and 121°C for 1M7/8552.

Furthermore, an increased amount of powdered resin was also noted with increasing aging times. These observations are consistent with the degradation mechanism postulated above.

3.3. Trends and accelerated aging

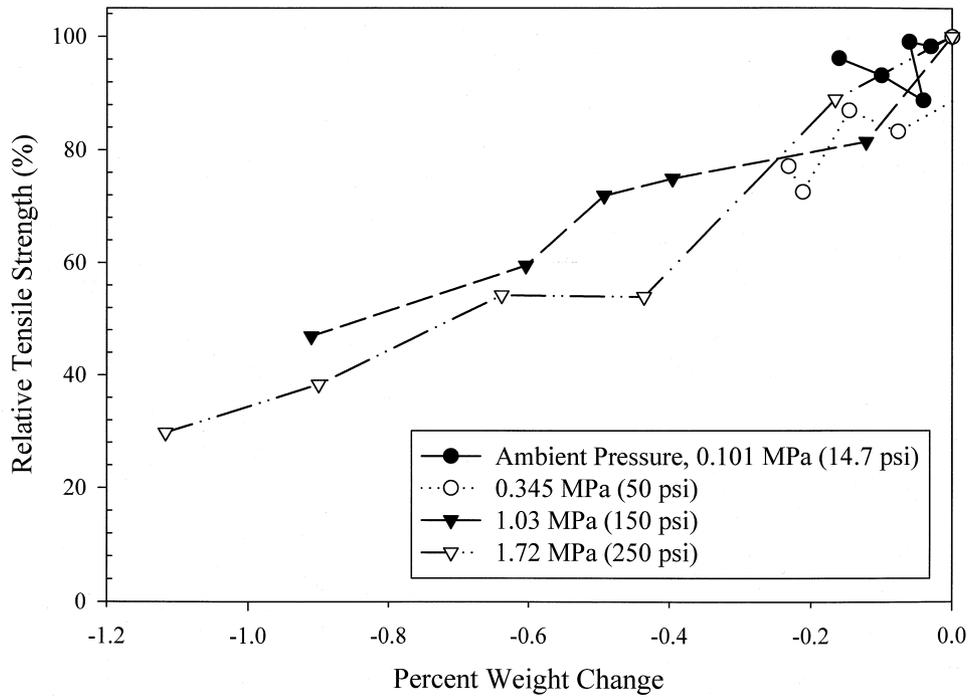Although the data presented above show the basic effects of elevated pressure on aging. some of the means

Fig. 10. Relative tensile shear [$\pm45°$]$_{2s}$, strength retention versus percent weight change for aging in air at various pressures and 121°C for AS4/3501-6 test specimens.
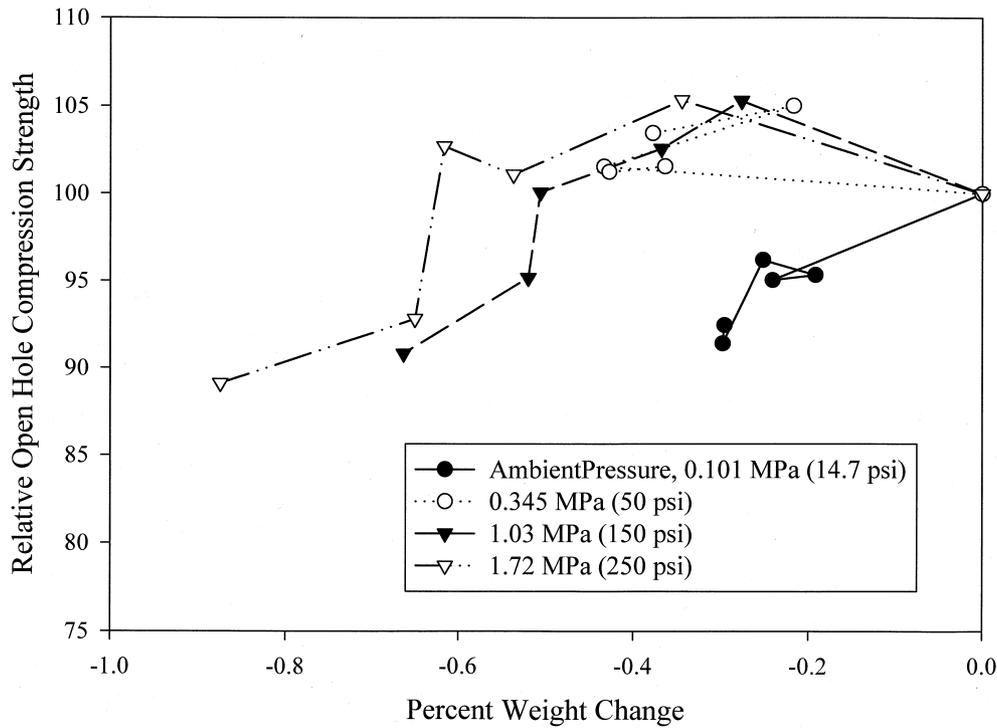

Fig. 11. Relative open-hole compression strength retention versus percent weight change for aging in air at various pressures and 121°C for 1M7/8552-6 test specimens.

for assessing the quantitative effects of the aging accleration are needed. As a step in this analysis, Figs. 10 and 11 show comparisons between the relative strength retention for the tensile shear and the open-hole compression specimens vs. weight loss, respectively. Additionally, Fig. 12 compares the relative open-hole compression modulus to percent weight change. Subsequently, Figs. 13 and 14 show the relative weight-change rates for the tensile shear and
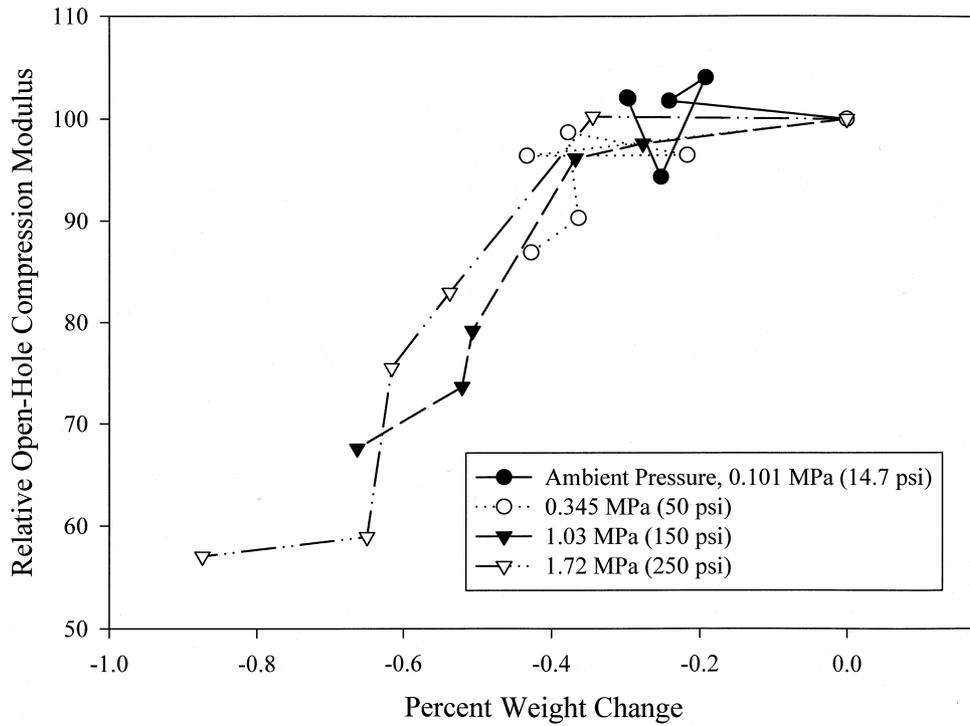

Fig. 12. Relative open-hole compression modulus retention versus percent weight change for aging in air at various pressures and 121°C for 1M7/8552 test specimens.
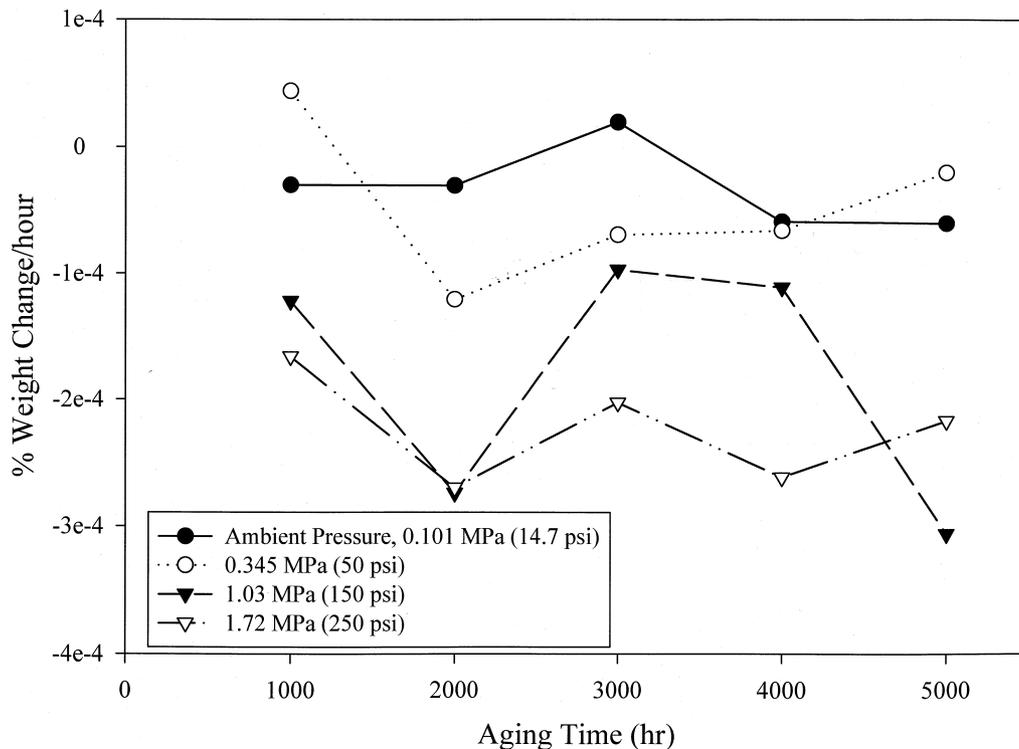

Fig. 13. Weight-change rate of tensile shear, [±45°]₂ₛ, specimens versus aging time in air at various pressures and 121°C for AS4/3501-6.
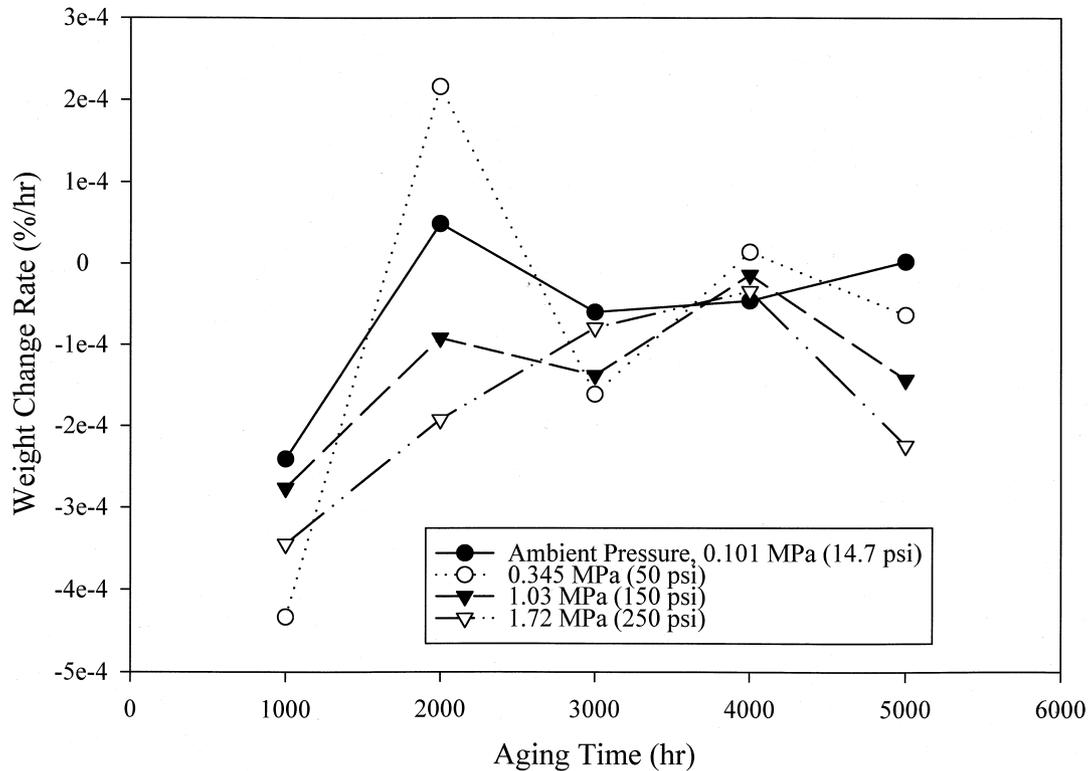
Fig. 14. Weight-change rate of open-hole compression specimens versus aging time in air at various pressures and 121°C for 1M7/8552.

open-hole compression specimens vs. exposure time, respectively.

The comparisons of strength retention vs. weight loss in Figs. 10 and 11 show very close consistencies between all of the exposure pressures for both the tensile shear and open-hole compression data. The only exception to this is the ambient-pressure open-hole compression specimens that were, as mentioned previously, aged in a chamber separate from the other specimens. Figs. 10 and 11 suggest that the increased pressures accelerate aging in a similar fashion that would be expected from long-term exposures at lower pressures. Clearly, longer-term exposures at lower pressures are required to fully verify this hypothesis. Additionally, a larger data set is needed to determine to what degree the measured differences in strength retention for similar weight losses at different pressures are pressure dependent.

The change in relative open-hole compression modulus with weight change shown in Fig. 12 also shows a strong consistency in trends between the different pressures. The results shown in Fig. 12 also support the hypothesis that, because the 0° plies are located at least one ply in from the outer layer, significant changes in mechanical properties do not occur until the degradation reaches the first 0° layer. This effect is seen in the lack of modulus decrease until the weight change has surpassed 0.4%. This effect was observed for all pressure levels.

In Figs. 13 and 14 the weight-change rates are compared for both sets of specimens. These figures show

no clear trends, however. The hypothesized thermo-oxidative mechanism given above in which competing weight-gain (oxygen uptake) and weight-loss (chain scission) mechanisms occur may partially explain the lack of clear trends. Furthermore, any transverse microcracking that may occur will also tend to distort the relative weight-change rates observed at different pressures.

In summary, it appears that weight-change rates, at least for the exposure times considered herein, are not reliable indicators for determining aging acceleration.

4. Conclusions and recommendations

The results presented herein conclusively show the benefits of using elevated pressure to accelerate the oxidative aging of polymeric composites. In particular, the decrease in tensile shear strength after 2000 h and 1.72 MPa is similar to that observed after 5000 h exposure to 177°C at ambient pressure for a similar material [4]. For both materials amid specimen configurations, pressure is seen to directly affect the degree of weight loss. Thus the elevated pressure indeed accelerates the rate of degradation in a measurable way.

The large difference between the tensile shear and the open-hole compression strength changes is not only attributable to the different lay-ups used, but also to the type of resin system used. As noted above, earlier work [2,3] showed that for resin systems containing similar

chemical structures degradation was less pronounced for the tougher resin system. In this case, the 8552 is a toughened epoxy whereas the 3501-6 is an untoughened one. It is expected that similar degradation mechanisms are present in the two materials, but that the increased resistance to microcracking in the toughened resin delays the onset and retards the growth rate of microcracks. This conclusion is based on the similarity of the resins used in earlier work [2,3] and the present work.

Additionally, at longer times the same trends in strength and weight loss became apparent in the toughened 8552 open-hole compression specimens as were observed in the 3501-6 tensile shear coupons. These results support the proposed hypothesis for the delayed onset of significant property losses in the open-hole compression specimens being a result of having the primary load-carrying ply one layer in from the outer layer thereby requiring time for significant degradation to reach this layer. The reduced amount of transverse cracks for the toughened versus the untoughened material likely also partially accounts for the observed differences.

Comparisons of strength retention and weight loss showed good consistency in the qualitative and quantitative trends for the various pressures measured for both sets of test specimens. These results suggest that it may be possible to use such data to construct accelerated-aging curves to calculate long-term strength retention from short-term aging at elevated pressures in a predictable manner.

The data presented herein may be used to begin a preliminary modeling effort to characterize the interaction of oxidation and mechanical property loss. Longer term, a more comprehensive database will be required using higher-temperature materials potentially suitable for HSCT usage such as K3B (E. I. duPont de Nemours, Wilmington, DE) and PETI-5 (NASA—Langley, Langley, VA) aged at elevated pressures in order to provide useful predictive aging models. The similarity in mechanisms observed for the epoxy materials evaluated herein to the degradation mechanisms of high-temperature polyimides such as PMR-15 [7] strongly suggests that the methodology used in the present work may be extended to these other high-temperature polymeric material systems.

The results, in general, suggest that elevated pressure may be a good tool for significantly reducing screening times for material that will be subjected to long exposures in oxygen-containing environments at elevated temperatures. Because HSCT candidate materials need to have 60,000 h of useful structural life — equivalent to nearly 7 years continuous aging — the use of an accelerating method such as that proposed herein is essential.

Additional work to develop quantitative predictive models using the proposed methodology will be required.

Another factor that will need to be considered in any HSCT application is atomic oxygen. At high cruise altitudes, potential exposure to atomic oxygen that may be present would need to be accounted for to fully characterize any material system.

Acknowledgements

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