Effects of Hygrothermal Cycling on the Chemical, Thermal, and Mechanical Properties of 862/W Epoxy Resin

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Abstract

The hygrothermal aging characteristics of an epoxy resin were characterized over 1 year, which included 908 temperature and humidity cycles. The epoxy resin quickly showed evidence of aging through color change and increased brittleness. The influence of aging on the material's glass transition temperature (T_g) was evaluated by Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA). The T_g remained relatively constant throughout the year long cyclic aging profile. The chemical composition was monitored by Fourier Transform Infrared Spectroscopy (FTIR) where evidence of chemical aging and advancement of cure was noted. The tensile strength of the resin was tested as it aged. This property was severely affected by the aging process in the form of reduced ductility and embrittlement. Detailed chemical evaluation suggests many aging mechanisms are taking place during exposure to hygrothermal conditions. This paper details the influence of processes such as: advancement of cure, chemical degradation, and physical aging on the chemical and physical properties of the epoxy resin.

Introduction

Epoxy resins constitute an important class of matrix material for the aerospace industry. These materials are generally easy to process and can be tailored to provide a wide range of chemical and physical properties (Ref. 1). Composites based on epoxy resins are often used in structural applications where environmental conditions can include cyclic exposure to high heat and humidity. Repeated exposure to temperature/humidity cycles will be referred to as "hygrothermal aging" in this paper. Hygrothermal aging results in changes to the physical and mechanical properties of an epoxy resin (Ref. 2). When the epoxy resin is used as the matrix material in a composite, these changes in resin properties can contribute to microcracking and to reduced strength for matrix dominated composite failure modes.

Hygrothermal aging can induce permanent changes in an epoxy resin through non-reversible mechanisms that alter the chemical structure of the material. This process is called chemical aging. A change in the glass transition temperature (T_g) during aging is one indication of chemical aging in the form of continued cure or thermal degradation in the bulk resin. Chemical aging can also occur as oxidation (which is typically limited to exposed surfaces) and as hydrolysis (which is dependent on moisture absorption). There is a vast body of literature detailing the influence of thermal-oxidative aging on the chemistry of epoxy resins and epoxy based composites. It has been reported that thermal-oxidative environments can be detrimental to the integrity of the resin, and many papers report a reduction in T_g with aging; attributed to oxidative chain scission within the structure (Refs. 3 and 4). The chemical reactions associated with oxidation have been extensively monitored by Fourier Transform Infrared Spectroscopy (FTIR), and a significant database of peak assignments has been collected (Refs. 5 to 9). Hydrolysis also contributes to a decrease in T_g through chain scission within the epoxy structure. As with oxidative aging, hydrolysis effects have can also be monitored by FTIR as a result of changes to the epoxy chemistry (Ref. 10).

Hygrothermal aging can also induce reversible changes in an epoxy resin through reversible mechanisms that alter the physical state of the material without changing the chemical structure. One such effect is shrinkage of the material at temperatures below T_g. The reduction in volume (without change in mass or change in chemical structure) is associated with volume relaxation toward the equilibrium volume. Although free volume within the resin decreases, T_g does not change. This process is called physical aging. Physical aging can cause embrittlement of polymeric materials, including epoxy resins. There is information in the literature indicating that the embrittlement of epoxy resin caused by physical aging is at least partially reversible by exposing the material to an appropriate thermal cycle with a maximum temperature above T_g (Ref. 3). An indication of physical aging is no change in T_g during aging and reversibility of aging effects such as embrittlement or reduced creep rate. Another reversible effect is plasticization due to moisture absorption (Refs. 2 and 11). Plasticization increases free volume within the polymer, which can significantly reduce T_g and alter mechanical properties. It is well known that elevated temperature mechanical properties are affected by moisture absorption from high humidity environments (Refs. 12 to 15).

The intent of this work is to identify potential aging mechanisms within the epoxy matrix of composite materials exposed to environmental conditions representative of the fan section of a modern high bypass jet engine. The 862/W epoxy resin was chosen for this work, as it has been identified as a candidate material for composite engine fan cases and other structures in the fan section of gas turbine engines. Materials used in these engine structures absorb moisture at a rate which is dependent on ambient conditions and length of time that an aircraft is on the ground. During takeoff and after landing materials can be exposed to elevated temperatures as a result of heat transfer from the engine core. During ascent and descent, materials experience thermal cycling as the ambient temperature changes with altitude. There is no unique hygrothermal cycle for all engines and all flight cycles. However, common features of most cycles include: (1) thermal cycling with material in a partially moisture saturated condition; (2) prolonged low temperature isothermal exposure at cruise; and (3) transient high temperature exposures. The cycle used in this work is intended to simulate these conditions as closely as possible while allowing two cycles per day to accumulate a large number of cycles within 1 year. Other potential conditions that could have an effect on material aging (such as fluid exposure, erosion, and repeated ice/hail impact) are not considered in this work. This paper investigates the accumulation of changes in an epoxy matrix material (resin only) after hygrothermal cycling for over a year. Ultimately, the effect of aging on properties of the composite material and on structural performance is the important issue. Composite materials made with carbon fibers and the same epoxy resin discussed in this work are currently being exposed to the same hygrothermal cycle to investigate the effects of hygrothermal aging on composite material properties.

Experimental

Resin Fabrication

Epon 862 epoxy resin was purchased from Hexion Specialty Chemicals. Epikure W curing agent was purchased from Resolution Performance Products. E862/W resin plates were manufactured as 0.609 m by 0.6096 m by 0.635 cm (2 ft by 2 ft by 0.25 in.) thick sheets using a resin transfer molding process. The cure time at maximum temperature was 2.5 hr at 176 °C (350 °F) under 1.03 MPa (150 psi) of pressure.

Aging Cycle

Figure 1 illustrates the thermal/moisture cycle. The materials were aged according to the following cycle: 5 hr soak at 29 °C (85 °F)/85 percent humidity, ramp to –54 °C (–65 °F), 1 hr soak, ramp to 121 °C (250 °F), 2 hr soak, ramp to 29 °C (85 °F). Each cycle requires 12 hr, including the ramp times. It should be noted that the ramp times are not defined by the set points, but rather by the thermal loading of the chamber. The soak times are guaranteed because the timer starts when the chamber reaches the temperature set point.

Thermal Rejuvenation of Physical Aging

Cylindrical tensile specimens were heated to a temperature above T_g then quenched in an effort to reverse the effects of physical aging. Thermal rejuvenation was investigated using specimens aged through 66 cycles, by holding one tensile specimen at 160 °C ($T_g \sim 150$ °C, T_{cure} 177 °C) in an air circulating oven for 10 min. A 20 min ramp to 160 °C was used. Following the 10 min hold, the sample was immediately removed to room temperature to cool. DSC specimens were also thermally treated using the same method to verify that this procedure removed the endotherm observed by differential scanning calorimetry that corresponds to a physically aged sample. The same procedure was followed for samples aged through 581 cycles.
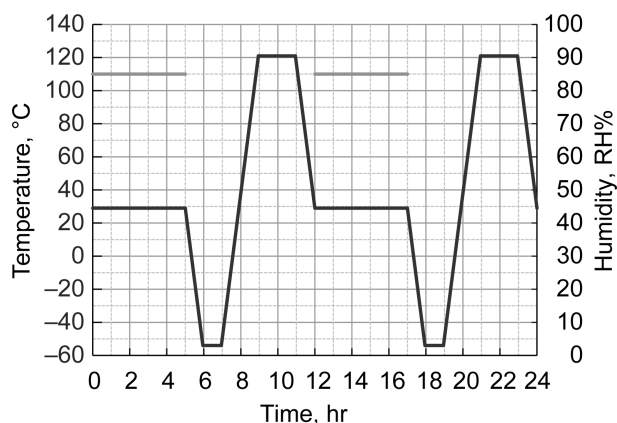

Figure 1.—Aging cycle based on set points.

Characterization

A modulated Differential Scanning Calorimeter (DSC/Model No Q1000, TA Instruments) was used to determine the glass transition temperature of the cured epoxy. A small (4 to 9 mg) square specimen was weighed into a crimped aluminum DSC pan. The tests were performed under air and nitrogen, using a 5 °C ramp rate from –90 to 370 °C. Glass transition temperatures were also obtained by Dynamic Mechanical Analysis (DMA/Model No 2980, TA Instruments). The T_g was identified as the intersection of lines tangent to the drop in E', as described in ASTM D 7028, Standard Test Method for Glass Transition Temperature (DMA T_g) of Polymer Matrix Composites by Dynamic Mechanical Analysis. FTIR data was collected using a Thermonicolet 380 FTIR. Data was collected using attenuated total reflectance, which is a surface characterization technique. Tensile tests were performed using a modified version of ASTM standard D-638, Standard Test Method for Tensile Properties of Plastics. The large resin sheets needed for ASTM D-638 were difficult to fabricate out of the E862 resin due to cracking, bubbling and warping. Therefore smaller specimen geometries were chosen. These cylindrical specimens had a 0.3175 cm (0.1250 in.) length by 0.3175 cm (0.1250 in.) diameter gauge section, large transition radii, and long 2.54 cm (1 in.) ends for gripping, Figure 2 (Ref. 16).

Data Verification Through Reanalysis of All Samples at the End of Aging Program

The aging program described above subjected resin coupons to continuous heat and humidity cycles over a two year period. During that time, samples were removed monthly from the aging chamber. Half of the samples were characterized by the described methods, and half were stored in a desiccator to be characterized at the end of the two year program. The purpose of setting aside aged samples for concurrent characterization was to identify any deviation from the original data trends that may be associated with instrument drift, contamination, calibration, or other factors that could alter output over the initial two year time frame of the tests. After examining the aged samples consecutively over the course of a week, we found the trends in DSC and DMA remained unchanged from trends found over the two year analysis period. FTIR results were comparable to those examined on a monthly basis. This was anticipated as this technique evaluates material surface chemistry rather than a phase transition which is based on calibration from a standard. Tensile experiments were not repeated.
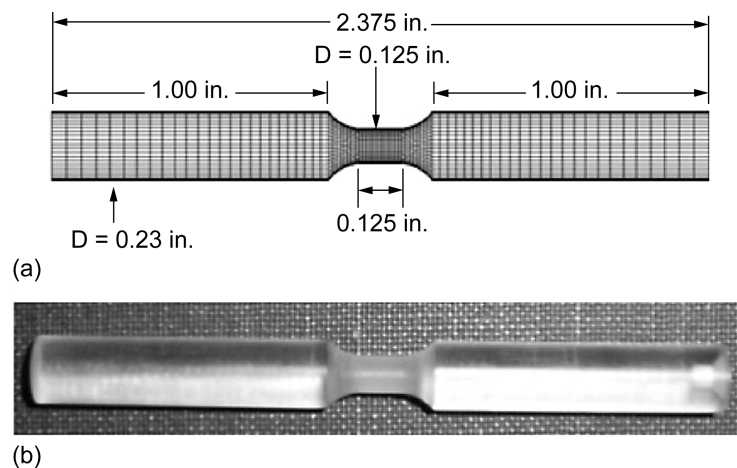
Figure 2.—(a) Tensile specimen dimensions; (b) resin tensile specimen.

Results and Discussion

The chemical structure of Epon 862 epoxy, and "W" diamine curing agent are illustrated in Figure 3.

A representative chemical structure of the crosslinked 862/W resin is illustrated in Figure 4. The crosslinking reaction can lead to a structure which includes secondary amines, epoxy, and hydroxyl functionalities.

Evidence of chemical aging was quickly observed through darkening of the resin samples throughout the aging process, Figure 5. The color change has been noted throughout the literature and is attributed to modification of the resin surface chemistry (Ref. 17). Although the color change was drastic, the responsible chemical degradation is limited to the sample surface. Changes to the surface chemistry will be described in the next several sections.
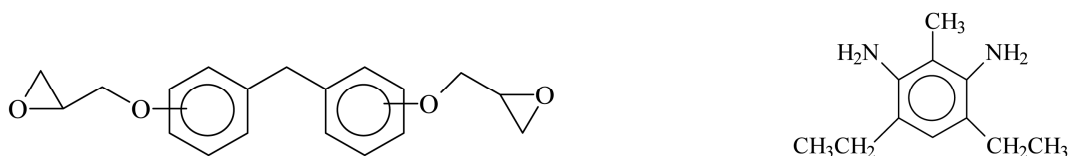
Figure 3.—Chemical structure of Epon 862 epoxy (Bisphenol-F/epichlorohydrin), and W curing agent (diethyltoluenediamine).
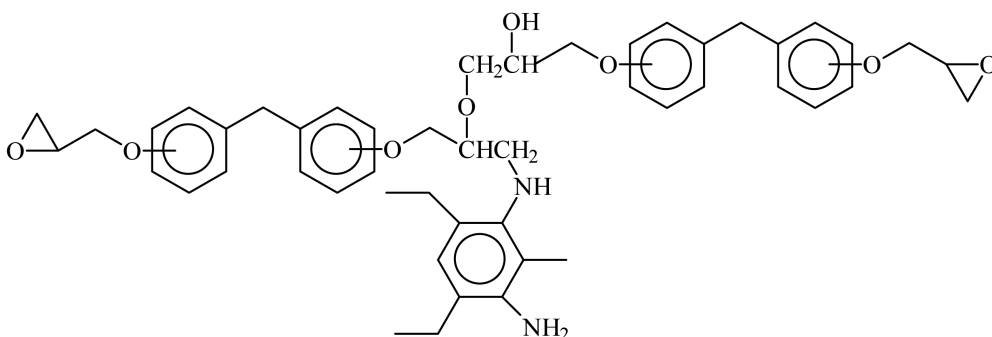
Figure 4.—Chemical structure representing functional groups present following the epoxy cure.
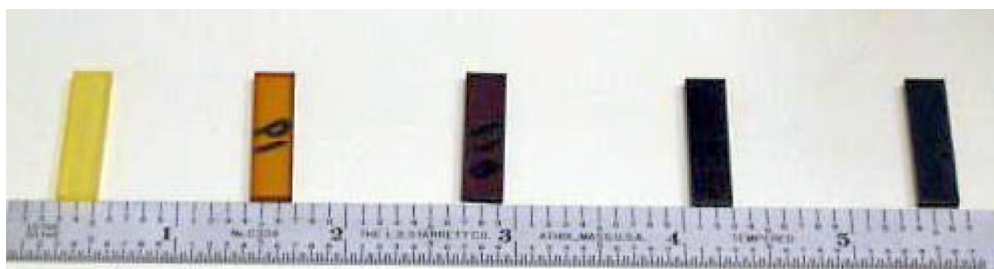
Figure 5.—Images from left to right show resin aged from: 0 cycles, 54 cycles (4 weeks), 217 cycles (16 weeks), 374 cycles (28 weeks), and 581 cycles (44 weeks).

FTIR

Two absorption bands were primarily used to monitor changes in chemical structure of the epoxy resin with aging. The first band absorbed at 907 cm^{-1}, and was attributed to stretching of the epoxy ring functionality. The second band at 1653 cm^{-1} was associated with amide formation resulting from oxidation of the crosslinked structure (Refs. 5 and 18).

The FTIR spectral overlay, Figure 6, shows a loss of the absorbance band at 907 cm^{-1} by 108 cycles. This suggests that from 0 through 108 cycles, the cure of the epoxy resin was advancing by crosslinking through the epoxide ring.

Beyond 108 cycles in the aging process, a second consistent change in the epoxy chemical structure by FTIR occurs. The spectra in Figure 7 demonstrate a continued growth of the bands at 1653 cm^{-1} and 1601 cm^{-1}. These were attributed to the formation of an amide functionality via degradation of the epoxy-amine crosslinked network (Refs. 3 and 19).

Bellenger and Verdu offer multiple reaction mechanisms which may be involved in amide formation following thermal or photo-oxidative degradation of a comparable diepoxide/diamine resin. Their report proposes the following mechanism, which proceeds through oxidation of the methylene carbon adjacent to the secondary amine; Figure 8.
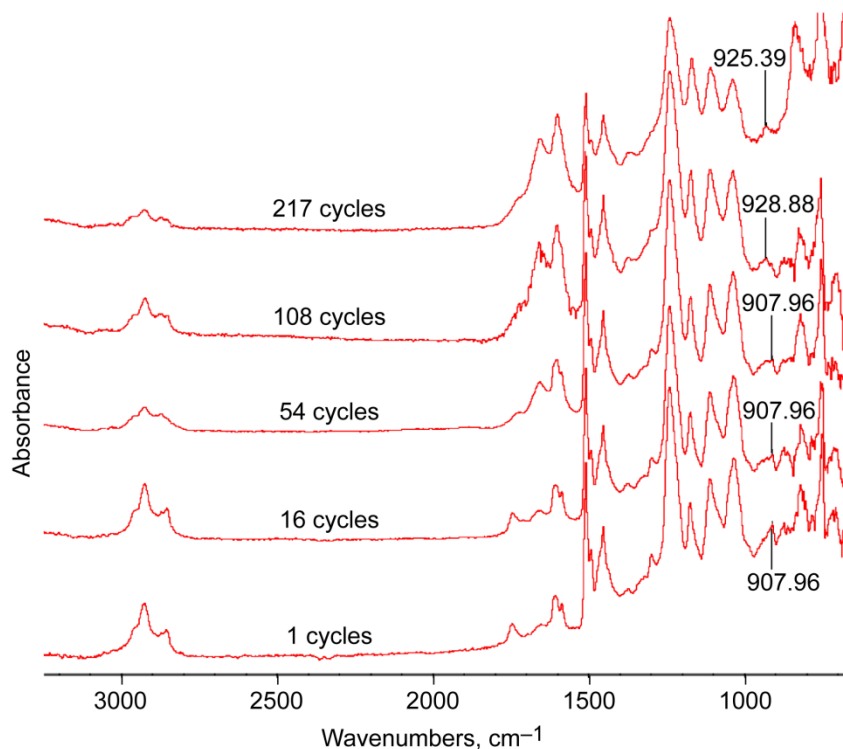

Figure 6.—FTIR spectra showing the loss of the band corresponding to the epoxy ring, 907 cm^{-1}.
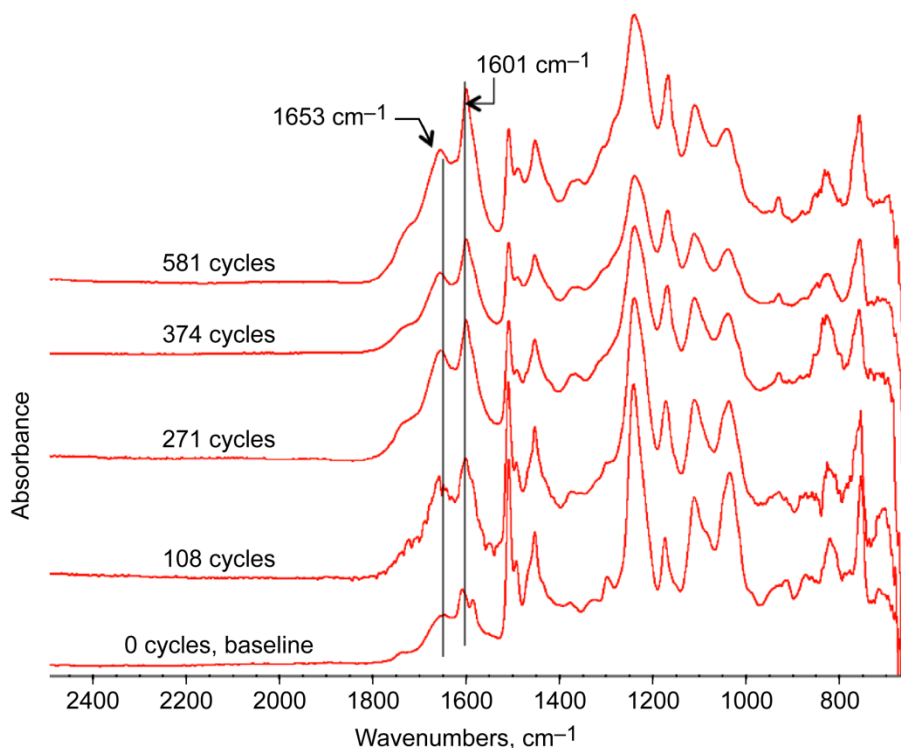
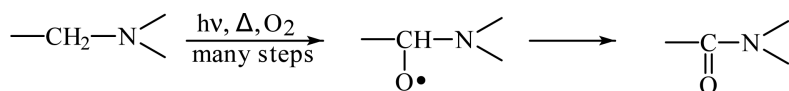Figure 7.—FTIR spectra of the epoxy resin aged from 271 to 581 cycles.

Figure 8.—Proposed mechanism of amide formation of aging.

It is important to note that the FTIR evidence of aging and crosslinking was limited to the sample surface. Polishing off the surface layer of an aged specimen yielded a FTIR spectrum identical to that of the unaged material. The FTIR data in Figure 9 compares the unaged (baseline) material with the surface and interior of a sample aged through 271 cycles. The surface of the aged specimen shows the disappearance of the epoxy ring band at 907 cm^{-1} and increased intensity of the bands at 1653 and 1600 cm^{-1}. By comparison, the spectrum of the interior of the aged sample is identical to that of the unaged resin.

The depth of the oxidized surface region was examined by polishing the sample surface and periodically examining the exposed material. The FTIR spectra stacked in Figure 10 are representative of the gradual removal of the exposed surfaces. The top spectrum is representative of the initial surface chemistry and the depth of each layer is indicated as a distance from the original sample thickness.

The surface of the sample exposed to 217 cycles shows the characteristic amide peak at 1653 cm^{-1}. The epoxy ring peak at 907 cm^{-1} is absent, suggesting full cure at the surface. Just 0.04 mm below that surface layer, there is evidence of the epoxide functionality. By removing 0.2 mm, the FTIR spectrum of the new surface is comparable to the unaged material. The peak identified by the asterisks is due to contamination of the FTIR instrument.
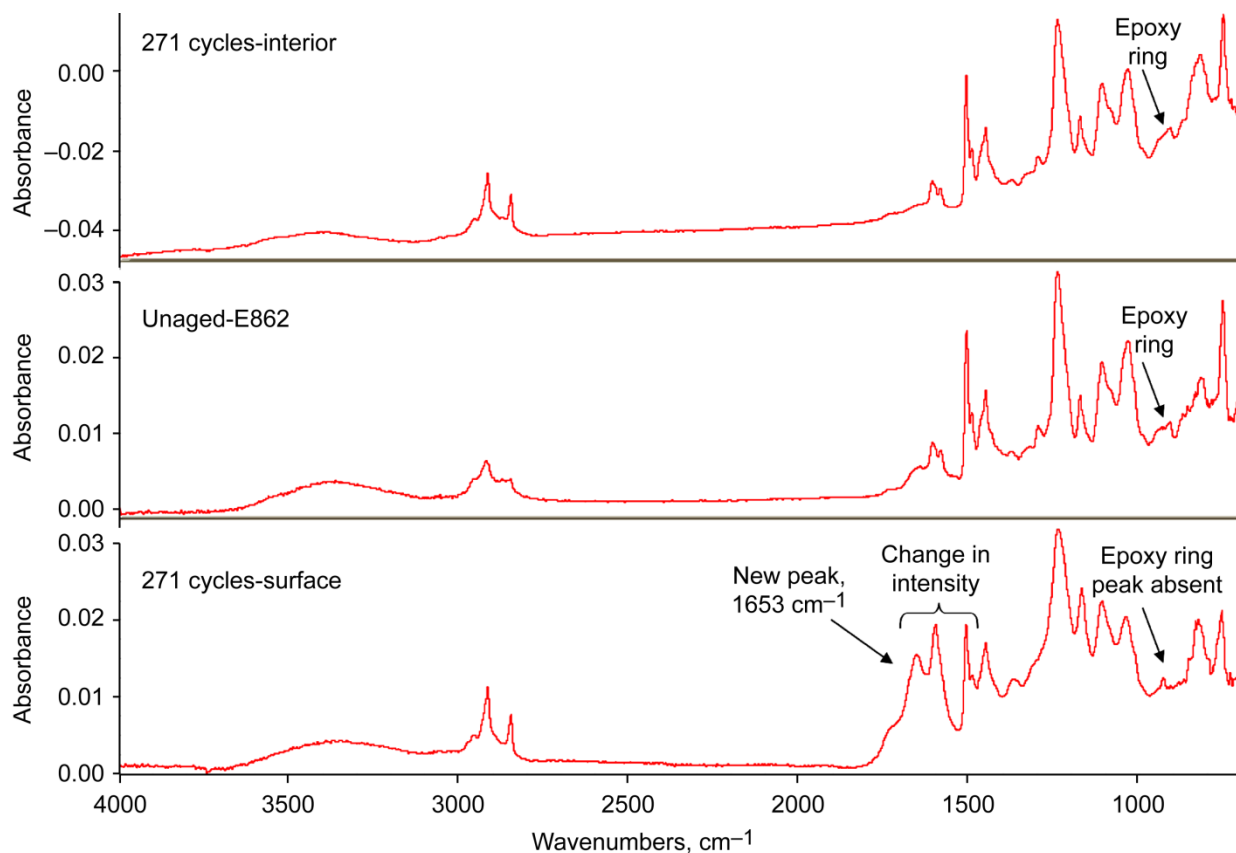
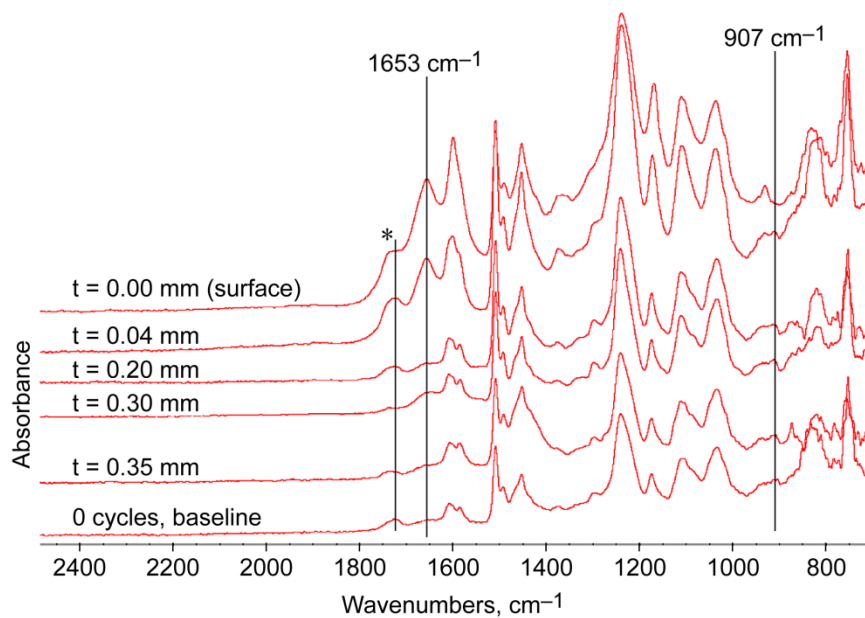Figure 9.—FTIR spectra of the baseline and aged material, and the interior of an aged material.

Figure 10.—Stacked FTIR curves of aged resin surface and interior following gradual polishing into the bulk; (t = distance from surface, into the bulk resin). Resin was aged for 217 cycles.

DMA and DSC

The initial cure reaction of an epoxy system oftentimes leaves unreacted epoxy. Consequently, resins are typically postcured to further advance the cure and increase T_g. While the materials in this study were not postcured, an increase in T_g was observed following a drying step at 104 °C. In effect, the drying step acted as a postcure. Postcure can result in release of absorbed moisture or low molecular weight species as well as possibly continued cure. All of these processes would lead to the increase in T_g which was observed throughout the early aging cycles.

DMA and DSC experiments showed an increase in the glass transition temperature during the early stages of aging, 0 to 217 cycles; followed by a leveling off of the T_g. The data plotted in Figure 11 shows considerable scatter, however the overall trend demonstrates nearly constant T_g on continued aging.

The overlaid DSC plots in Figure 12 show the rise in T_g following the initial aging cycle. This has been attributed to a postcure effect, as described earlier. The stability of T_g during aging is reasonable considering that the T_g is a bulk material effect and the aging processes observed have been limited to the resin surface. Therefore, while FTIR evidence points to continued crosslinking through the epoxy ring, that reaction is limited to the surface and there is no net effect to the resin T_g.

In addition to T_g, the DSC plots show an endotherm (negative peak) at a temperature slightly above the glass transition temperature. This peak initially becomes larger as the number of aging cycles increases then maintains a constant area beyond 200 cycles, Figure 12. This peak is attributed to volume relaxation (densification) from a quenched state toward an equilibrium volume state at a temperature below T_g, i.e., physical aging. Figure 13(a) plots the weight change in the sample with aging and Figure 13(b) plots the volume loss with aging. The loss of volume with constant weight is characteristic of the densification that results from physical aging. The volume loss within the sample is significant during the early number of cycles, then levels off beyond 200 cycles; corresponding well to the DSC data described above. The effects of physical aging (including the appearance of the endothermic peak) can be reversed by reheating the material to a temperature above T_g followed by quenching to a temperature below T_g. Appearance of this endotherm on aging and disappearance of the peak after heating above T_g has been reported in the literature for epoxy resins (Ref. 3).

The reversibility of physical aging for a specimen exposed to 427 thermal cycles is demonstrated in Figure 14 by the absence of the endotherm in the DSC curve of the aged resin following a quench from above T_g. The initial temperature ramp, the quench to –90 °C, and second ramp were performed consecutively in the DSC.

While physical aging is reversible, it contributes to material embrittlement, and can lead to reduced mechanical properties (Ref. 3).
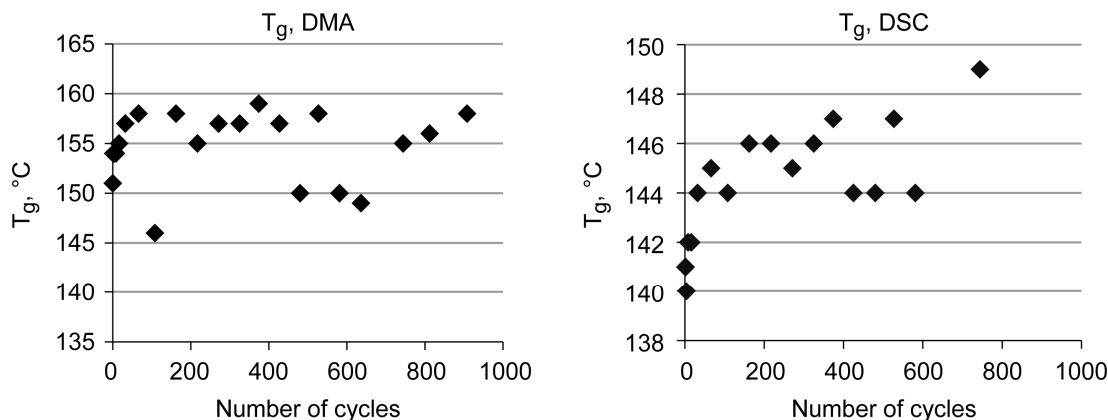

Figure 11.—T_g values of unaged and aged samples as determined by DMA and DSC.
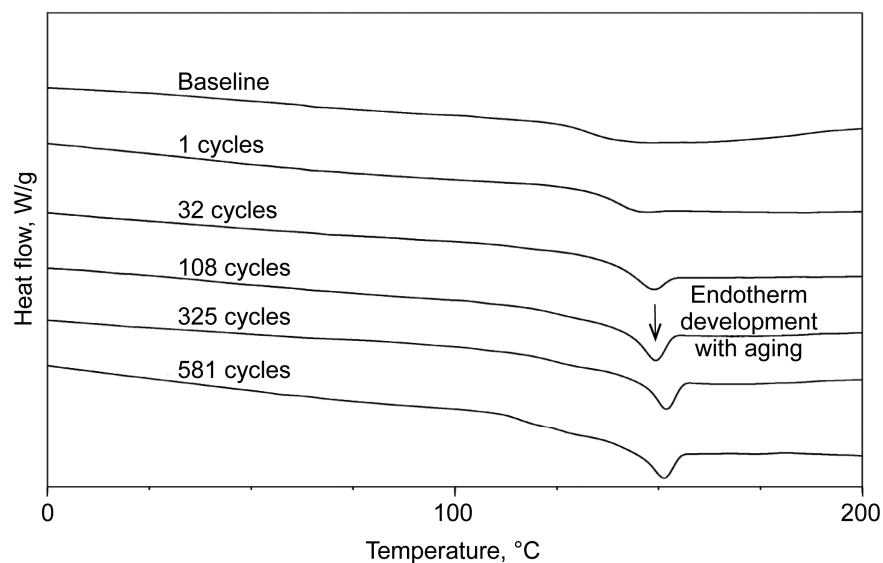
Figure 12.—DSC plots illustrating the growth of an endothermic peak above T_g during aging.
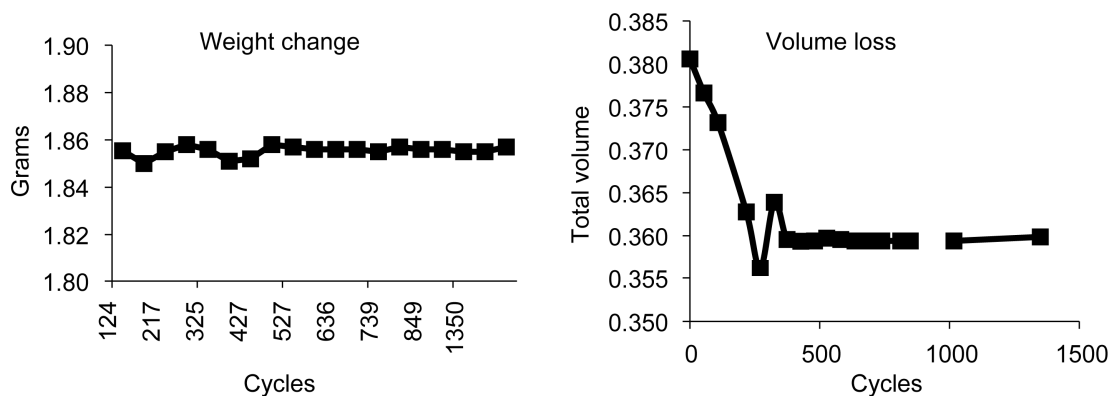

Figure 13(a).—Sample weight change as a function of cycles and Figure 13(b) Sample volume loss as a function of cycle number.
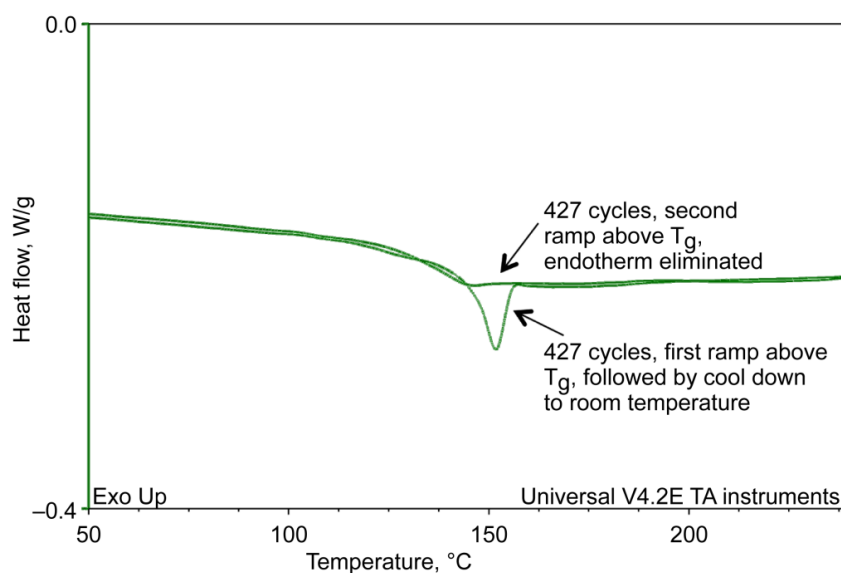

Figure 14.—DSC plots showing the reversibility of the endotherm which follows T_g.

Tensile Tests

The tensile stress-strain curves for material exposed to 0, 54, and 108 cycles are shown in Figure 15. Figure 15 shows the true stress (which uses the instantaneous cross-sectional area rather than the initial cross-sectional area to calculate stress) as a function of percent strain. An increase in the plateau stress occurs after a small number of cycles. This is a result of the postcure effect discussed earlier. Postcure involves both reversible and non-reversible changes to the resin. Non-reversible effects include changes to the chemical structure resulting from continued cure reactions and loss of residual low molecular weight chemical species that were produced by the cure reactions. A reversible effect is the volume relaxation (physical aging) discussed earlier. Exposing the resin to a rejuvenation thermal cycle (heating to a temperature above T_g followed by a quench to room temperature) should reduce the plateau stress by reversing the physical aging component of postcure. The plateau stress is not expected to be fully reduced to the level of the unaged resin because the effects of additional cure and release of volatiles is not reversed during the rejuvenation cycle. Preliminary data on the effect of a rejuvenation cycle is discussed below. In addition to the effect on plateau stress, the data in Figure 15 indicate that hygrothermal aging causes a large decrease in strain to failure. Determining the potential effects of aging and rejuvenation on strain to failure is complicated by the large scatter typically observed for failure strain data. For example, the strain to failure ranges from about 25 to 40 percent for the three unaged resin curves in Figure 15. Failure can initiate at surface flaws, at interior flaws, or by cavitation of the resin in the interior (Refs. 5 and 20). The source of the scatter in the failure strain data is local variability in material properties and in the flaw size distribution that controls failure initiation. A large number of tests and statistical analysis of the test data would be needed to fully account for the effects of material variability and flaw distribution on strain to failure for each aged condition. Additional testing would then be needed to determine the effect of a thermal rejuvenation cycle. Although the data is limited, Figure 15 does indicate that hygrothermal aging causes a large reduction in strain to failure. This embrittlement becomes so severe after 108 cycles that it is difficult to test the specimens without failure of the resin within the gripping fixtures. The reduced strain is a result of the combined effects of chemical aging, physical aging, and desorption of volatiles during the hygrothermal cycling. Since physical aging is reversible, it should be possible to reduce the amount of embrittlement and increase the strain to failure for an aged specimen by exposing the aged specimen to a thermal rejuvenation cycle. Initial results showing the effects of a rejuvenation cycle on plateau stress and strain to failure are shown in Figure 16.
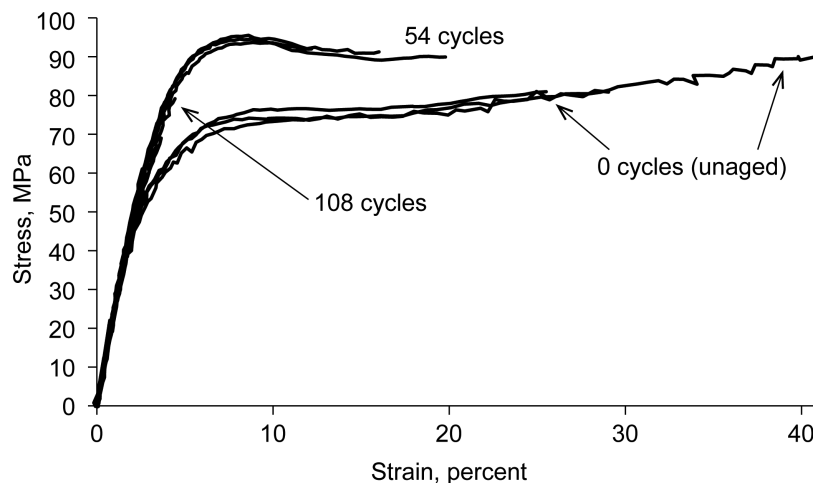

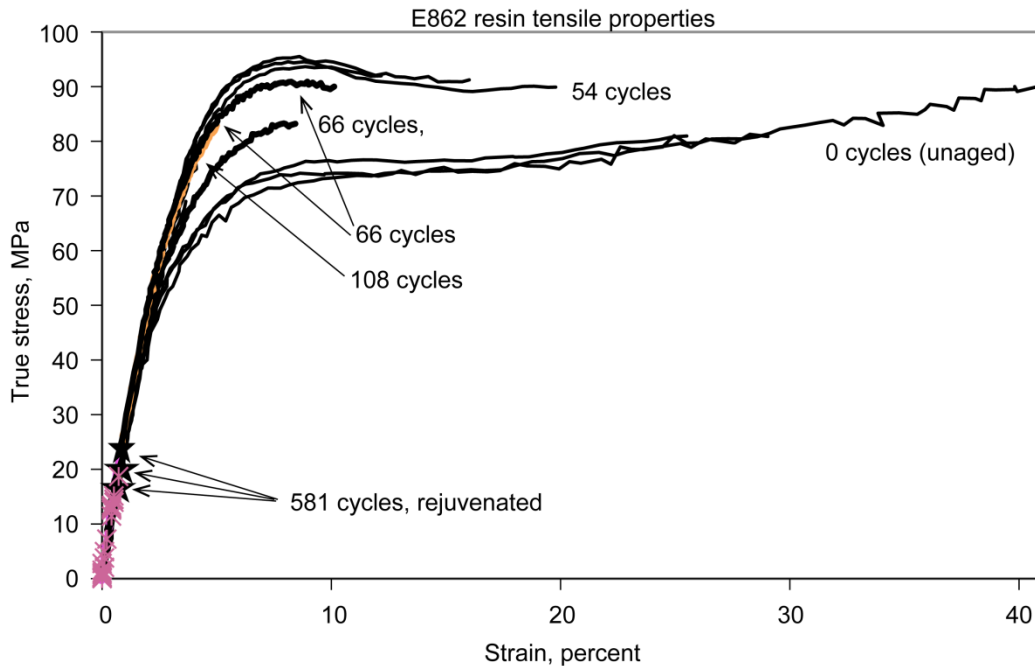Figure 15.—Stress-strain curves of baseline and aged resin.

Figure 16.—Stress-strain curves of baseline and aged resin, included the thermally annealed 66 cycle sample.

Figure 16 shows the stress-strain curves from Figure 15 along with additional data for specimens aged 66 cycles (before and after thermal rejuvenation) and additional data for specimens aged 581 cycles (rejuvenated data only since non-rejuvenated specimens were too brittle to test). Only three specimens aged for 66 cycles were available for this preliminary investigation of the effects of rejuvenation. Two specimens were tested after aging for 66 cycles. One specimen was tested after aging for 66 cycles followed by rejuvenation. Rejuvenation resulted in a reduction in the plateau stress compared to the two non-rejuvenated specimens. Rejuvenation does not reduce the plateau stress to the level observed for the unaged (0 cycles) specimen. This was expected since rejuvenation reverses only the physical aging portion of the postcure effect. Chemical changes that occurred during postcure were not reversible. Rejuvenation does not appear to significantly increase the strain to failure for the single specimen tested after aging for 66 cycles followed by rejuvenation. However, since strain to failure typically shows a large variation, testing of more specimens is needed to determine the effect of rejuvenation on strain to failure. There is some evidence supporting a possible increase in strain to failure following rejuvenation. Specimens aged for 581 cycles were too brittle to test without breaking in the test fixtures prior to applying load. After rejuvenation it was possible to test these specimens up to a strain of about 1 percent. Results are shown in Figure 16. These preliminary results will be used to guide a more detailed investigation of the effects of rejuvenation on tensile properties in future work when more aged specimens become available.

Conclusions

Epon 862/W resin was aged in hygrothermal conditions for 908 cycles. The initial cycles acted as a post cure causing an increase in T_g and an increase in the plateau stress in the tensile stress-strain curve. Other chemical aging processes, including cure and oxidation, were limited to the surface of the resin. This was demonstrated by polishing the surface of the sample and examining the FTIR spectra of the bulk material; which remained unchanged from the baseline epoxy resin. Confinement of chemical aging to the surface after the initial postcure effect resulted in little change in T_g of the bulk resin throughout the subsequent aging cycles. Hygrothermal aging caused a continuous reduction in tensile strain to failure

with increasing aging cycles and an increase in the plateau stress after the initial aging cycles. The increase in the plateau stress was partially reversible by exposing the aged specimen to a thermal rejuvenation cycle. There is some evidence that rejuvenation can increase the strain to failure, but testing of a larger number of specimens is needed to confirm this effect. This work focused on the effects of hygrothermal cycling on the epoxy matrix material. A separate investigation is being performed to determine the effects of hygrothermal cycling on composite mechanical properties and ballistic impact resistance. Preliminary results from these studies indicate that resin embrittlement described in this paper does not translate into reduced quasi-static or impact strength. Further investigation of aging effects in matrix materials and composite materials is ongoing.

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14. ABSTRACT
The hygrothermal aging characteristics of an epoxy resin were characterized over 1 year, which included 908 temperature and humidity cycles. The epoxy resin quickly showed evidence of aging through color change and increased brittleness. The influence of aging on the material's glass transition temperature (Tg) was evaluated by Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA). The Tg remained relatively constant throughout the year long cyclic aging profile. The chemical composition was monitored by Fourier Transform Infrared Spectroscopy (FTIR) where evidence of chemical aging and advancement of cure was noted. The tensile strength of the resin was tested as it aged. This property was severely affected by the aging process in the form of reduced ductility and embrittlement. Detailed chemical evaluation suggests many aging mechanisms are taking place during exposure to hygrothermal conditions. This paper details the influence of processes such as: advancement of cure, chemical degradation, and physical aging on the chemical and physical properties of the epoxy resin.

15. SUBJECT TERMS
Composites; Epoxy; Aging; Hygrothermal aging; Differential Scanning Calorimetry (DSC); Fourier Transform Infrared Spectroscopy (FTIR)

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