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A Model of the Thermal-Spike Mechanism in Graphite/Epoxy Laminates

Michael J. Adamson

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      TEMPERATURE EFFECTS
MINS: / ADSORPTION/ CONCENTRATION (COMPOSITION)/ EPOXY COMPOUNDS/ EPOXY MATRIX
      COMPOSITES/ LAMINATES/ THERMAL EXPANSION
ABA:  Author
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A Model of the Thermal-Spike Mechanism in Graphite/Epoxy Laminates

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N83-12173#

A MODEL OF THE THERMAL-SPIKE MECHANISM IN GRAPHITE/EPOXY LAMINATES

Michael J. Adamson

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ABSTRACT: The influence of a thermal spike on a moisture-saturated graphite/epoxy composite was studied in detail. A single thermal spike from 25°C to 132°C was found to produce damage as evidenced by a significant increase in the level of moisture saturation in the composite. Approximately half of this increase remained after a vacuum-anneal at 150°C for 7 days, suggesting the presence of an irreversible damage component. Subsequent thermal spikes created less and less additional moisture absorption, with the cumulative effect being a maximum or limiting moisture capacity of the composite. These observations are explained in terms of a model previously developed to explain the reverse thermal effect of moisture absorption in epoxy and epoxy matrix composites. This model, based on the inverse temperature dependence of free volume, contributes an improved understanding of thermal-spike effects in graphite/epoxy composites.

Introduction

Absorbed moisture at elevated temperature is known to degrade both the physical *[1]* and mechanical *[2,3]* behavior of graphite/epoxy laminates when the moisture is present in significant quantities. Although this influence has been investigated extensively, the exact damage mechanisms have not been well defined. In general, it is assumed that these mechanisms take the form of (1) moisture-induced plasticization of the epoxy matrix, or (2) the introduction of microcracks in the matrix or at the fiber/matrix interface as a result of moisture-induced residual stresses, or (3) both. In any case, it is clear that the damage mechanisms can be relatively complex and that the severity of the property degradation of the laminate can be directly influenced by its thermal, as well as its mechanical service history. One important example of the influence of thermal history is thermal-spike damage.

Graphite/epoxy laminates containing significant absorbed moisture can be severely degraded as the result of a thermal spike, defined as an abrupt upward thermal excursion followed by a decrease in temperature [*1*]. Damage induced by a thermal spike can be far more severe than that caused by other components of the real-life environment and can have a lasting influence on the behavior of the laminate [*1*].

The purpose of the present work is to better understand the mechanism of thermal-spike damage by applying concepts developed to explain the "reverse thermal effect" (the inverse temperature dependence of moisture absorption near saturation) observed in epoxy and epoxy matrix composites [*4,5*]. First, we briefly review experimental observations associated with the reverse thermal effect. An applicable model is developed in terms of the temperature dependence of "free volume" in a glassy polymer, considering both the equilibrium and kinetic aspects of moisture sorption. Next, we present the experiments and results associated with thermal-spike damage in a laminate. Finally, we discuss a potentially feasible mechanism for thermal-spike damage based on the concepts developed to explain the reverse thermal effect of moisture sorption in epoxy.

The Reverse Thermal Effect of Moisture Sorption

Moisture content, as influenced by immersion time and temperature, was studied by Adamson [5] in the fully cured Hercules 3501-5 resin system and in the graphite/epoxy laminates of Hercules AS/3501-5 and Fiberite Corporation T300/934. The study was part of an investigation of the effects of accelerated environmental conditioning on graphite/epoxy composite specimens being prepared for mechanical testing. The temperature and humidity conditions used are unlikely to occur simultaneously but both are components of the real-life environment. Solar heating can increase an aircraft "skin" temperature to 74°C and rainfall, condensation, de-icing and washing are simulated by immersion. At moisture contents well below saturation, these materials were found to follow Fickian moisture absorption, with moisture content increasing with the square root of time, and the rate of absorption increasing with increasing temperature. When these materials contain moisture in concentrations near saturation, however, contrary behavior is observed: an immediate increase in the rate of moisture absorption occurs when the immersion temperature is reduced. This behavior, termed the reverse thermal effect [5], has also been reported by Apicella et al. [4].

An example of the effect is shown in Fig. 1, which is a plot of moisture pickup as a function of exposure time for the neat resin. For convenience of discussion, Fig. 1 has been divided into three regions. Region 1 reflects moisture absorption during the first 144 days of immersion in 74°C water--enough time for the resin to become nearly saturated with moisture. Next, the water temperature was changed to 25°C with the resulting sorption behavior shown in Region 2. As seen, the resin rapidly gained moisture at this lower temperature. After about 50 days, moisture content appears to be approaching a new level of saturation. Finally, Region 3 shows the very rapid desorption of moisture which was observed in the resin when the water temperature was returned to 74°C. Within 3 days, the moisture content had returned to a value very near to that which would have been expected from an extrapolation of the original 74°C absorption curve.

Thermal expansion and swelling behavior was also studied as a function of immersion time and temperature [5]. It was found that at a constant temperature, the cured epoxy resin swells initially by an amount significantly less than the total volume of absorbed water. Further, the swelling efficiency of the absorbed water entering the polymer varies with the total moisture content of the polymer. Approaching saturation, the polymer swells by an amount nearly equal to the volume of water being absorbed. Additionally, the thermal expansion of the cured resin, when saturated with moisture, is more than twice that of the dry resin. Based on these observations and others, the following model was developed for the reverse thermal effect (Fig. 1) in terms of a slightly modified concept of free volume in conjunction with the theory of polar molecule interactions.

For a dry resin (essentially free of moisture), the specific volume V is the sum of the volume occupied by the thermally vibrating polymer molecules (occupied volume V_o) and the unoccupied space between the cross-linked polymer molecules (free volume V_f). The free volume of a glassy polymer at the glass-transition temperature T_g is estimated to be 2.5% of the specific volume [*6,7*]. When a resin is cooled from a temperature T_B (below T_g) to a lower temperature T_A, the specific volume of the resin decreases, as shown schematically in Fig. 2. Thermal expansion in the glassy state is characterized by minor structural changes and by an inverse dependence of hydrogen bonding on temperature [*8*]. The basic assumption of the model is that the total volume contraction of the resin will be less than would be expected from the thermally induced contraction of the occupied volume. This remaining volume will be the nonequilibrium volume component of the free volume; it is associated with the difficulty with which the structure of the resin approaches equilibrium [*8*]. This nonequilibrium volume component will be added to the equilibrium free volume, making the total free volume greater at the lower temperature T_A than at the higher T_B, as shown in Fig. 2 [*5,9-11*].

Initial moisture pickup in a dry resin occurs very rapidly (first portion of Region 1, Fig. 1), as moisture both fills the free volume and swells the resin structure. Consistent with this, the specific volume of the resin increases far less than the volume of the moisture absorbed;

some of the moisture simply occupies the open space within the resin structure. Once this open space or free volume is filled, moisture absorption slows significantly as the moisture content approaches saturation (the end of Region 1, Fig. 1); as a result, the occupied volume slowly swells. In this stage of absorption, the resin swells at an efficiency nearly equal to the volume of moisture being absorbed.

Near saturation at a constant temperature, moisture fully occupies the free volume (V_f) in the resin structure. Moisture also combines with the occupied volume, to produce an equilibrium volume component owing to swelling, V_s. These volume components are shown schematically in the left half of Fig. 3, after long-time exposure to water at 74°C (Fig. 1). Upon rapidly reducing the immersion temperature to 25°C (the transition from Region 1 to Region 2, Fig. 1), the free volume in the resin rapidly increases, as discussed above and shown in Fig. 2. Moisture which once caused swelling can now fill the additional free volume created by the decrease in temperature, as shown schematically in Fig. 3. Because the swelling volume is reduced, the change in specific volume with temperature should be greater for the wet resin than the dry resin. The wet resin was found to have a volumetric thermal expansion coefficient of about $1.9\times10^{-4}/°C$ or about twice that of $8.0\times10^{-5}/°C$ observed for the dry resin [5]. If it is assumed that all of the non-equilibrium free-volume component created by a change in temperature is filled from the swelling volume, the nonequilibrium free-volume volumetric coefficient will be the difference between the wet and the dry volumetric coefficients with a negative sign to indicate the inverse relationship with temperature, or about $-1.1\times10^{-4}/°C$ over the temperature range investigated (74°C to 1°C) [*5*]. In effect, moisture has disappeared volumetrically, but remained visible gravimetrically. As moisture was allowed to be absorbed with time at 25°C (Region 2, Fig. 1), the specific volume of the resin was found to increase until, near saturation at 25°C (end of Region 2, Fig. 1), it approached that observed at 74°C, as shown schematically in the right half of Fig. 3. This suggests that the V_s component is greater at the lower temperature, which reflects an increased moisture content of the occupied volume in equilibrium with the external water. Apparently all the new hydrogen bonds created when the temperature was reduced [*8*] were disrupted by the newly absorbed moisture [*5*]. Other mechanisms, such as possible microcracking caused by high temperature absorption, are ruled out for several reasons. The change in moisture concentration does not occur in a random manner but reacts immediately to temperature change and is reversible and repeatable. Other work [*12*] reports that photomicrographs of 3501-5 resin and composites show no microcracking caused by similar high temperature, high humidity exposure for up to one year. Additionally, free volume in 3501-5 resin saturated at 40°C was found to be greater than that found at 74°C [*5*]. If the reverse thermal effect is to be attributed to microcracking, then the conclusion would necessarily be that microcracking is inversely proportional to exposure temperature between 1° and 74°C.

Finally, when the immersion temperature was raised from the lower temperature (25°C) to the higher temperature (74°C) (the transition from Region 2 to Region 3, Fig. 1), both the free volume and the swelling volume in the resin tended to contract and the resin was left in a highly supersaturated condition. (Shown schematically in the left half of Fig. 4.) The specific volume increase as the temperature at saturation is increased from 25°C to 74°C was found equal to the additional volume of moisture absorbed with time at 25°C, as shown schematically in the right half of Fig. 3. With time, the moisture is rapidly forced out of the free volume and swelling volume, and the moisture content of the resin is returned to its equilibrium value (the end of Region 3, Fig. 1); this is shown schematically in Fig. 4.

Material and Procedure

The material used in this study was Hercules AS/3501-5 prepreg tape (resin volume fraction about 40 percent) formed into a 16 ply unidirectional sheet. The fabrication procedure, which is described elsewhere [3], involved the standard 177°C cure process. The 0.1-in. (2.5-mm) thick composite sheet was cut into 2- by 2-in. (50- by 50-mm) square specimens. All specimens were given a vacuum-thermal treatment to remove the residual moisture. This treatment was specifically selected to reduce the possibility of damage to the specimens; it consisted of a vacuum dry at 100°C for at least 10 days, followed by a vacuum anneal at 150°C for 7 days. This drying treatment is more thorough than those used in most absorption studies. The result is an apparently higher than usual saturation moisture content since the calculated percent by weight moisture is dependent on the dry weight. No indication of damage was observed when the specimens were checked by the procedure to be described later in this section. All specimens were stored in a vacuum desiccator before testing.

The moisture sorption studies were performed by immersing the specimens in distilled water at the desired temperature. Water temperature was controlled to better than ±0.5°C, using a constant-temperature bath. Temperature changes were accomplished by transferring the specimens to an immersion bath at the different temperature.

Before weight-change measurements were made, the specimens were wiped dry, using a lint-free towel, and then air-dried at room temperature for 5 min. This procedure helped to eliminate possible errors caused by specimen heat upsetting the balance zero and the uncertainty of surface-water quantity. Specimen weight was determined using a Mettler analytical balance, which was accurate to ±0.05 mg. All calculations of percent weight increase were based on the dry weight just before moisture absorption. The scatter of all data was within ±0.01 wt percent.

For the purpose of the present study, the thermal-spike procedure consisted of an air-exposure in a preheated oven at 132°C for 20 min followed by removal to room temperature air. Damage was defined as an increase in moisture saturation concentration. Irreversible damage was defined as a concentration increase that could not be reversed by a vacuum-thermal treatment consisting of a vacuum dry at 60°C for 5 days followed by a vacuum anneal at 150°C for 7 days.

Results and Discussion

As has been observed in other resin/composite systems [5], moisture sorption in the Hercules AS/3501-5 graphite/epoxy composite was found to be similar to that in the Hercules 3501-5 resin itself. The first moisture absorption cycle for the composite immersed in 74°C water is shown in Fig. 5. As shown in Fig. 5, the form of the absorption curve is similar to that observed for the resin (Fig. 1) with the composite approaching a saturation level of about 2.3 wt percent moisture after 140 days of exposure. The composite specimens were then transferred to an immersion bath at 25°C. Like the resin (Fig. 1), the composite rapidly gained moisture at this lower temperature, exhibiting the reverse thermal effect. After 40 days, the composite approached a saturation level of about 2.6 wt percent moisture (Fig. 5).

If it is assumed that all the moisture absorbed in the composite enters the resin, the volume fraction of resin in the composite can be estimated based on the moisture absorption data. After an equal immersion time of 140 days at 74°C, the resin was found to absorb 6.8 wt percent moisture (Fig. 1); the composite absorbed 2.3 wt percent (Fig. 5). The ratio of the moisture content of the composite to that of the resin is about 34 percent. Likewise, the increased moisture absorption at 25°C compared with that at 74°C is about 0.81 wt percent for the resin and 0.26 wt percent for the composite. The ratio of the weight gain of the composite to that of the resin yields an estimated resin volume fraction of about 32 percent. These estimated volume fractions of resin in the composite agree well with the estimated volume fraction in the prepreg tape of 40 percent--resin volume fraction is reduced during the fabrication and curing processes. Additionally, this suggests that the reverse thermal effect is similar in both the resin and composite and is uninfluenced by the presence of carbon fibers.

A specimen, which had been saturated with moisture at 25°C, was subjected to an abrupt increase in temperature, or thermal spike, to 132°C for 20 min in air, air-cooled, and immediately returned to the 25°C water. The observed change in specimen weight as a result of this thermal spike is shown in Fig. 5. As can be seen, some loss in moisture was observed following the thermal spike. This is as would be expected from the contraction in free volume based on the model for the reverse thermal effect, as well as from thermally induced moisture desorption from the specimen surfaces. This moisture loss is seen in Fig. 5 to be replaced following immersion in 25°C water, with absorption continuing to a level well above the original level of moisture saturation at 25°C. The new level of moisture saturation after 60 days of immersion represents a 0.27 percent increase in weight of the composite (a 0.025 g increase in weight of the composite specimen) compared with the original level of saturation.

The increased saturation level observed at 25°C after a thermal spike (Fig. 5) can be explained in terms of the model for the reverse thermal effect. A sudden and rapid rise in temperature increases the thermal motion of the polymer molecules, thus increasing the occupied volume and reducing the free volume available in the resin structure. Moisture originally present in the free volume at the lower temperature is squeezed out of the free volume into the resin structure, creating what could be called an excess volume--the difference between the free volume at 25°C and that at 132°C. Rapid cooling of the composite back to 25°C freezes this excess volume in the resin structure. As indicated in Fig. 5, a thermal spike is similar to a rapid transition from Region 2 to Region 3 (Fig. 1), followed by a return to Region 2, all within 20 min. The free volume again increases at the lower temperature, creating additional volume for moisture absorption equivalent to the excess volume created during the thermal spike. This additional free volume will rapidly fill with moisture, resulting in a level of moisture saturation in the composite greater than that observed before the thermal spike.

The excess volume created by the thermal spike can be estimated from an extrapolation of the temperature dependence of the free-volume change, -1.1×10^{-4}/°C (established from the reverse-thermal-effect studies between 1°C and 74°C) and the volume of resin in the composite. Taking the volume fraction of resin in the composite at 33 percent (estimated above) and the actual volume of the composite specimen to be 5.617 cm^3, a temperature rise of 107°C will result in an excess volume of 0.022 cm^3. Assuming the density of water to be 1 g/cm^3, the increase in moisture absorption at 25°C, as the result of this excess volume, will be 0.022 g.

This calculated value of moisture uptake compares well with the observed value of 0.025 g (Fig. 5) and lends credence to the applicability of the model.

The creation of an excess volume during a thermal spike does not necessarily imply irreversible or permanent damage to the resin structure. To assess damage, the spiked and unspiked composite specimens of Fig. 5 were vacuum-dried and annealed to remove the moisture and allow structural equilibrium to be reestablished. All specimens were then immersed in 60°C water and moisture pickup was followed to saturation; this is shown in Fig. 6. As seen in the figure, the spiked composite specimen exhibited about the same initial rate of moisture uptake as the unspiked specimens. However, the level of saturation was significantly greater for the spiked specimen, suggesting the presence of irreversible damage. The difference between the saturation levels is about 0.15 wt percent (Fig. 6) compared with 0.27 wt percent observed before the vacuum-anneal treatment (Fig. 5). Apparently the effects of a thermal spike are partially reversible, with about half of the excess volume remaining as irreversible damage in the composite.

Finally, in an effort to qualitatively establish the cumulative effect of multiple thermal spikes, the spiked specimen of Fig. 6 was placed in 25°C water, allowed to equilibrate, and spiked three additional times at 132°C in a manner identical to the initial thermal spike. Moisture uptake was followed as a function of time; it is shown in Fig. 7. Interestingly, although each additional spike is seen to increase moisture uptake by the composite at 25°C, the magnitude of change induced by each spike is less than the previous spike, and the trend is toward a maximum or limiting moisture capacity. These effects are being investigated in greater detail.

Although the present work does little to identify the actual physical mechanism of thermal-spike damage in composites, some insights into the process may be gained. First, the irreversible component of the effect of a thermal spike is a permanent structural change in the composite--excess free volume or moisture absorption sites are permanently created. These sites within the polymer structure are probably voids, cavities, and microcracks created by the force of a contracting moisture-filled free volume. The apparent approach to a maximum or limiting value with consecutive spikes suggests that damage becomes more and more difficult to create. This is consistent with the microcrack mechanism of damage where initially, weak interfaces of nucleation sites are available and are eventually consumed, making damage more difficult to develop. Finally, the reversible component of the effect of a thermal spike may in itself represent physical damage--damage that can be reversed by annealing. One such mechanism may be increased plasticization of the epoxy induced by excess free volume caused by abnormal supersaturation of moisture. Studies are continuing in an attempt to better understand the kinetic as well as the mechanistic aspects of thermal-spike damage in composites.

Summary

A model developed to explain the reverse thermal effect of moisture absorption and associated volume changes in an epoxy resin (Hercules 3501-5) was reviewed in detail. The primary consequence of this model is that the total volume change of a resin as the temperature is decreased will be less than would be expected from the thermally induced contraction of the volume occupied by the polymer molecules. This additional volume is the nonequilibrium volume component of the free volume and is associated with the difficulty of the resin structure to approach equilibrium. This nonequilibrium free-volume component will be added to the equilibrium free volume, making the total free volume greater at a lower temperature than at a higher temperature.

The basic concept of the model developed to explain the reverse thermal effect in resins was successfully applied to better understand thermal-spike damage in a graphite/epoxy composite (Hercules AS/3501-5). In brief, the force of a contracting moisture-filled free volume during a thermal spike will create a quantitatively predictable excess volume in the composite. Approximately half of this excess volume was found to be irreversible and is thus presumed to indicate permanent damage in the composite. Finally, it was speculated that permanent thermal-spike damage is the result of the nucleation and growth of voids or microcracks within the resin matrix or at the fiber-matrix interface. Microscopic examination is planned for the future as part of this on-going project.

Acknowledgment

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Figure Captions

FIG. 1--The reverse thermal effect of moisture absorption in Hercules 3501-5 resin immersed in water.

FIG. 2--Thermal expansion in a dry resin; volume components at two temperatures.

FIG. 3--Change in volume of a moisture-containing resin with a decrease in temperature; volume components at saturation at a high temperature and immediately following a decrease in temperature and after moisture saturation at the lower temperature.

FIG. 4--Change in volume of a moisture-containing resin with an increase in temperature; volume components at saturation at a low temperature and immediately following an increase in temperature and after moisture saturation at the lower temperature.

FIG. 5--Moisture absorption before and following a thermal spike applied to a moisture-saturated AS/3501-5 composite laminate.

● controls (data scatter within symbol); o spiked specimen.

FIG 6--Moisture absorption in spiked and unspiked (controls) AS/3501-5 laminate following moisture saturation, vacuum drying and annealing.

● controls (data scatter within symbol); o spiked specimen.

FIG 7--The cumulative effect of multiple thermal spikes on moisture absorption in a moisture-saturated AS/3501-5 laminate.

● controls (data scatter within symbol); o spiked specimen.

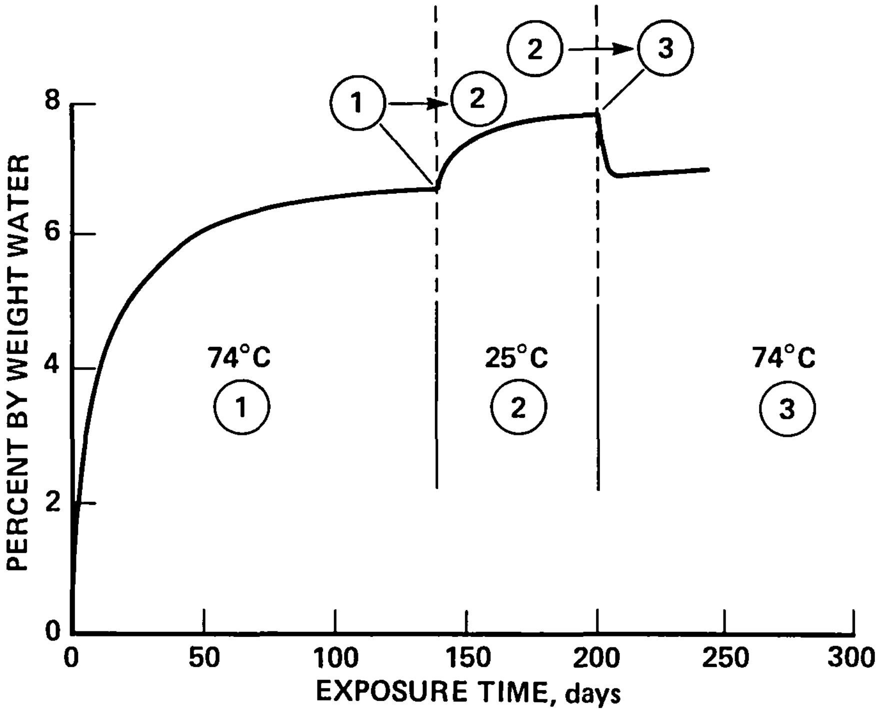

PERCENT BY WEIGHT WATER
0
2
4
6
8
0
50
100
150
200
250
300
EXPOSURE TIME, days
74°C
①
25°C
②
74°C
③
① → ②
② → ③

Fig. 1

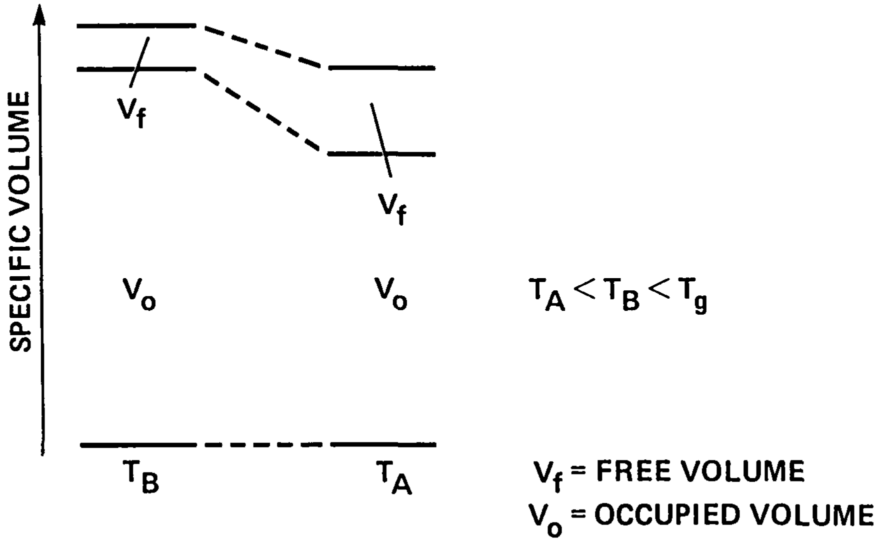
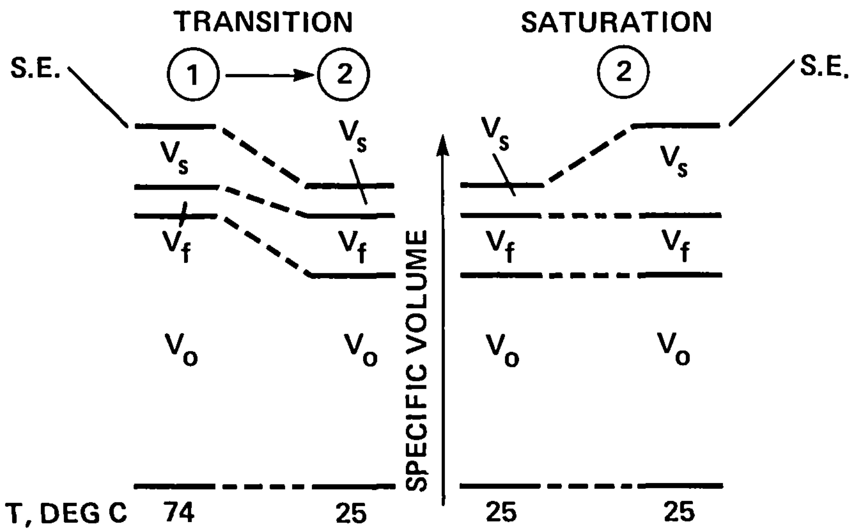

SPECIFIC VOLUME
V_f
V_f
V_o
V_o
$T_A < T_B < T_g$
T_B
T_A
V_f = FREE VOLUME
V_o = OCCUPIED VOLUME

Fig. 2



V_s = SWELLING VOLUME
V_f = FREE VOLUME
V_o = OCCUPIED VOLUME
S.E. = STRUCTURAL EQUILIBRIUM

Fig. 3

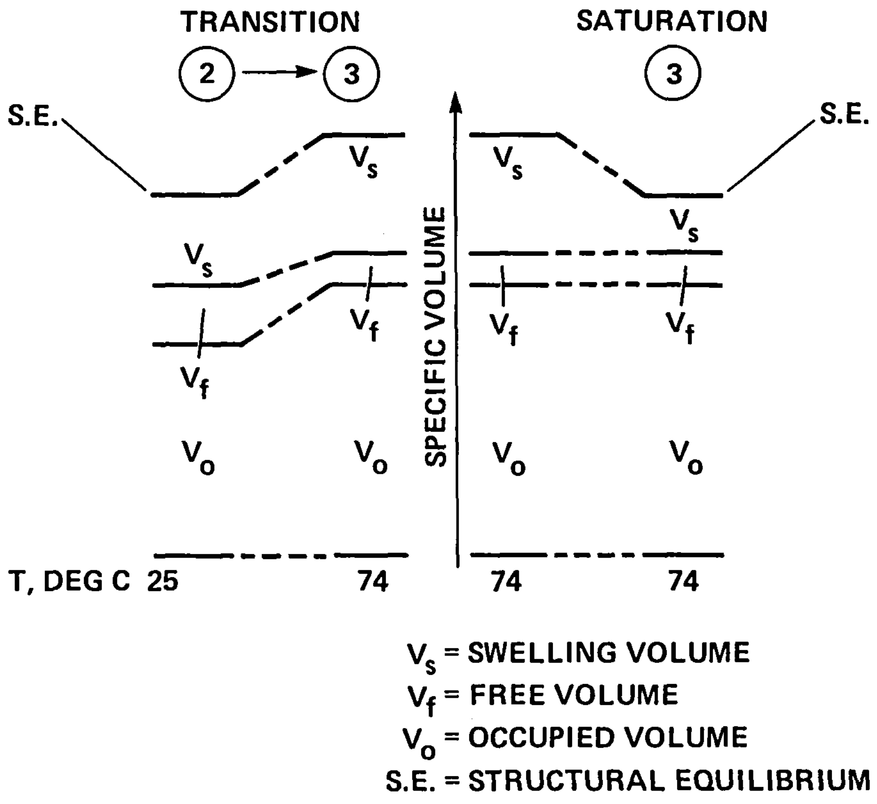

TRANSITION
2
3
SATURATION
3
S.E.
S.E.
V_s
V_s
V_s
V_s
V_f
V_f
V_f
V_f
V_o
V_o
V_o
V_o
SPECIFIC VOLUME
T, DEG C 25
74
74
74
V_s = SWELLING VOLUME
V_f = FREE VOLUME
V_o = OCCUPIED VOLUME
S.E. = STRUCTURAL EQUILIBRIUM

Fig. 4

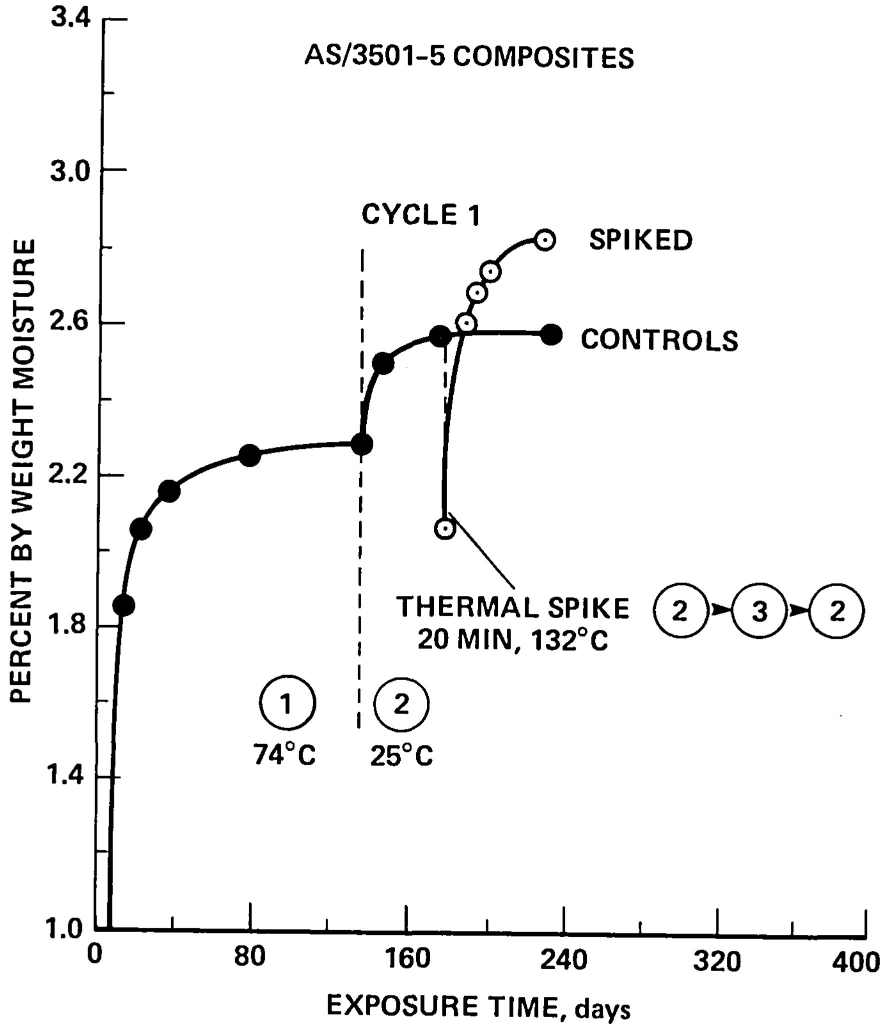

AS/3501-5 COMPOSITES
PERCENT BY WEIGHT MOISTURE
3.4
3.0
2.6
2.2
1.8
1.4
1.0
0
80
160
240
320
400
EXPOSURE TIME, days
CYCLE 1
SPIKED
CONTROLS
THERMAL SPIKE
20 MIN, 132°C
② ➤ ③ ➤ ②
①
74°C
②
25°C

Fig. 5

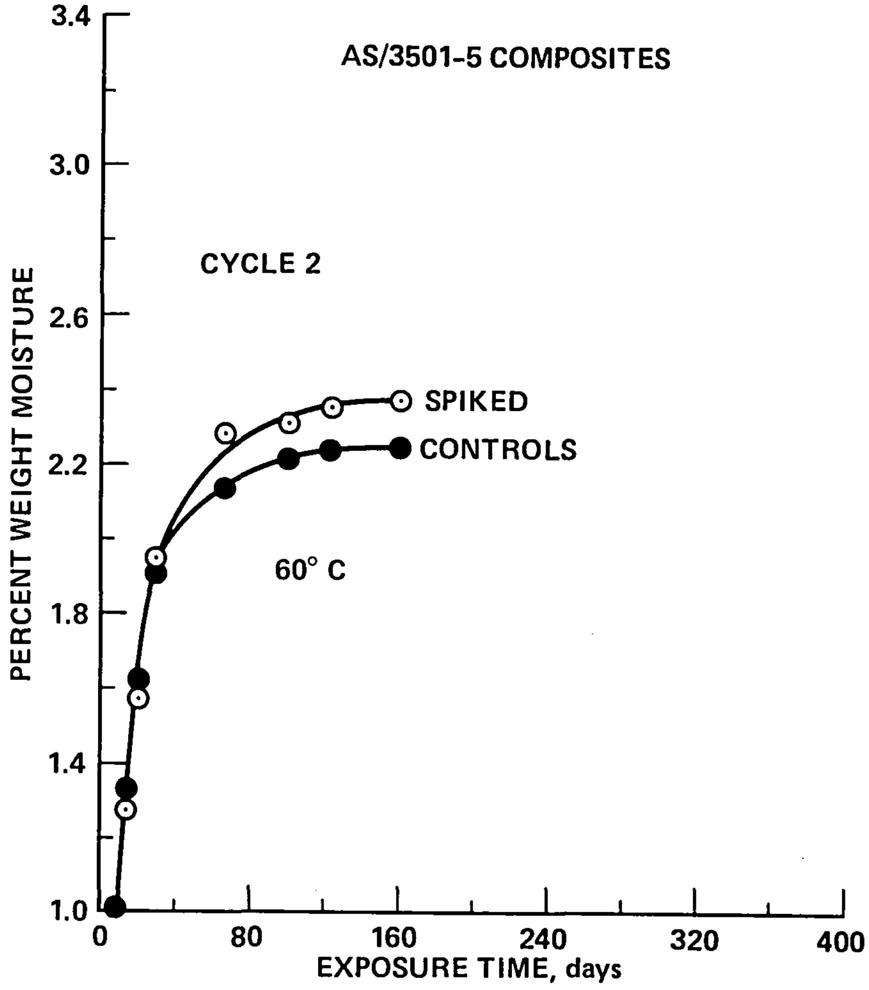
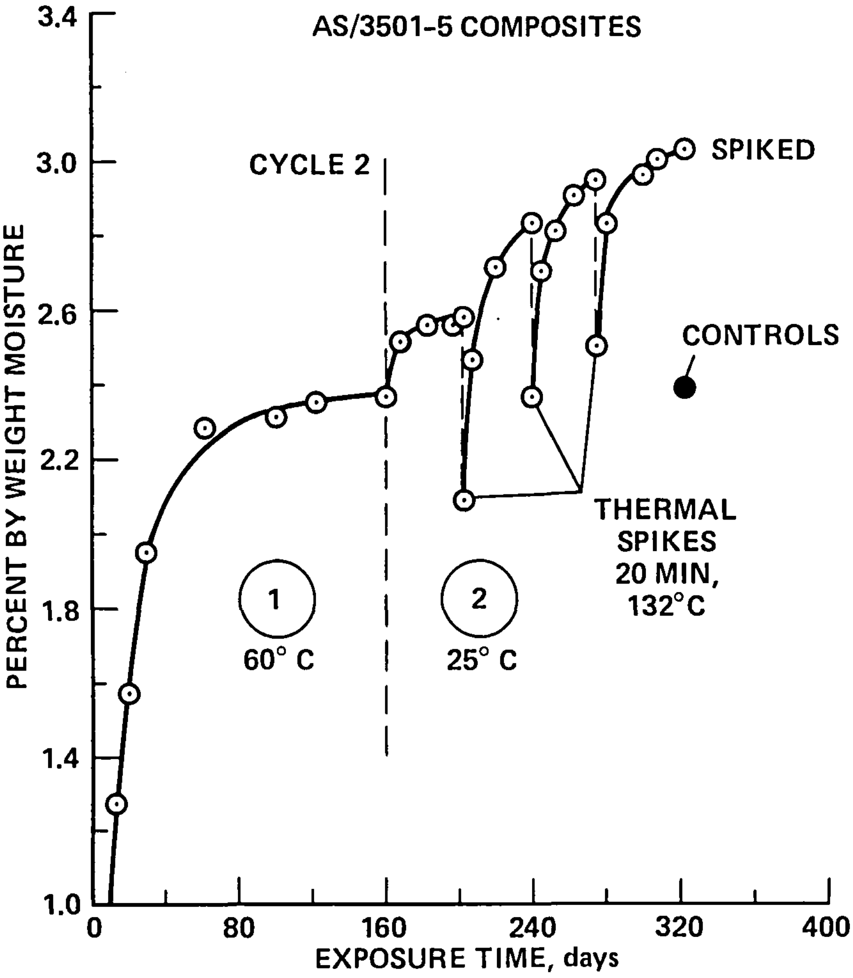

AS/3501-5 COMPOSITES
CYCLE 2
60° C
SPIKED
CONTROLS
PERCENT WEIGHT MOISTURE
3.4
3.0
2.6
2.2
1.8
1.4
1.0
0
80
160
240
320
400
EXPOSURE TIME, days

Fig. 6



AS/3501-5 COMPOSITES
PERCENT BY WEIGHT MOISTURE
3.4
3.0
2.6
2.2
1.8
1.4
1.0
0
80
160
240
320
400
EXPOSURE TIME, days
CYCLE 2
SPIKED
CONTROLS
THERMAL SPIKES 20 MIN, 132°C
1
60° C
2
25° C

Fig. 7

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16. Abstract

The influence of a thermal spike on a moisture-saturated graphite/epoxy composite was studied in detail. A single thermal spike from 25°C to 132°C was found to produce damage as evidenced by a significant increase in the level of moisture saturation in the composite. Approximately half of this increase remained after a vacuum-anneal at 150°C for 7 days, suggesting the presence of an irreversible damage component. Subsequent thermal spikes created less and less additional moisture absorption, with the cumulative effect being a maximum or limiting moisture capacity of the composite. These observations are explained in terms of a model previously developed to explain the reverse thermal effect of moisture absorption in epoxy and epoxy matrix composites. This model, based on the inverse temperature dependence of free volume, contributes an improved understanding of thermal-spike effects in graphite/epoxy composites.

17. Key Words (Suggested by Author(s))
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Swelling
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Graphite/epoxy composites
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