수지 노화와 잔류응력 변화가 Avimid® K3B/IM7 복합재 적층에 미치는 영향

The effects of matrix aging and residual stress changes on Avimid® K3B/IM7 laminates

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ABSTRACT

In this paper, the effects of matrix hygrothermal aging and residual stress changes on Avimid® K3B/IM7 laminates in 80℃ water were studied. The factors causing the 80℃ water to degradation of the laminates could be the degradation of the matrix toughness, the change in residual stresses. After 500 hours fully saturated aging of the neat resin, the weight gain was 1.55% increase with the diffusion coefficient $7\times10^{-6}m^2/s$ and the fracture toughness was decreased about 41%. After 100 hours fully saturated aging of the $[+45/0/-45/90]_s$ K3B/IM7 laminates in 80℃ water, the weight gain was 0.41% increase with the diffusion coefficient $1\times10^{-6}m/s^2$ and the loss of the microcracking fracture toughness was 43.8% of the original toughness. To see whether the residual stress influenced the fracture toughtness, two ply [90°/0°] laminates were put in 80℃ water from 2 hours to 8 hours. The changes in residual stress in 8 hours are less than 3MPa. Because the 3MPa change is not sufficient to degrade the laminates, the main factor to degrade the microcracking fracture toughness was the degradation of the matrix fracture toughness.

Key words : Residual Stress, Matrix, Hygrothermal Aging, Microcracking Fracture Toughness, and diffusion

1. Introduction

This research was to study what was the main factor for aging of Avimid® K3B/IM7 laminates. Aging is defined as time-dependant changes in the composite. The changes can be due to physical aging or chemical aging. My interest is not chemical aging but physical aging. In the past physical aging was studied using tensile creep properties[1], in this paper using microcracking fracture toughness. The factors causing the 80℃ water to degradation of the laminates could be the degradation of the matrix toughness, the change in residual stresses.

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The times to saturation in 80℃ water for Avimid® K3B/IM7 laminates and the neat resin are 100 hours and 500 hours. Avimid® K3B neat resin samples were placed in 80℃ water for times ranging from 50 hours to 500 hours.

The first way to study hygrothermal aging is to study aging of neat resin. The neat resin samples were characterized by three-point bending tests under plane-strain conditions. The 80℃ water aging had an effect on the neat resin K3B. Therefore, matrix-dominated failure analysis for the laminates hygrothermal aging was approached. The laminates layup was [+45/0/-45/90]s. During the experiments to microcrack the 90° ply in the middle of the laminates, there were no microcracks in +45 or -45 plies. The microcracks in the 90° plies were found sufficient to yield good results. To see whether residual stress influenced the microcracking fracture toughness, two ply [90°/0°] laminates were put into 80℃ water from 2 hours to 8 hours and the curvature changes observed. Because of the thin thickness, the issue of plate effects was ignored. The changes in residual stress from 2 hours to 8 hours were less than 3 MPa. Because a 3MPa change was not sufficient to degrade the microcracking fracture toughness, the residual stress change may not be the main factor to change the laminates fracture toughness.

2. Experiment

2.1 The aging of Neat Resin Avimid® K3B in 80℃ Water

1) Materials and Methods

The neat resin samples that were 6.5mm thick, 12.6mm wide and 101.6mm long put in the container at 80℃ water for 50 hours through 500 hours. By three point bending test, a critical intensity factor K_{IC} was determined by means of Eq. (1).

$$K_{IC} = y\left(\frac{6}{BW^2} * \frac{PS}{4}\sqrt{a}\right) \tag{1}$$

where,

$y = 1.93 - 3.07r + 14.53r^2 + 25.11r^3 + 25.8r^4$,

S ; the length of the sample,

$S > 4W$,

P ; applied maximum load to fracture,

$r = \frac{a}{W}$

Next, the determined K value was plugged into the following plane-strain behavior ASTM requirements, and all the dimensions were checked to see whether the requirements were satisfied.

$$\begin{aligned} &a \text{ ; crack length } > 2.5\left(\frac{K_{IC}}{\sigma_y}\right)^2, \\ &B \text{ ; sample thickness } > 2.5\left(\frac{K_{IC}}{\sigma_y}\right)^2, \\ &W \text{ ; specimen width } > 6.27\left(\frac{K_{IC}}{\sigma_y}\right)^2 \end{aligned} \tag{2}$$

where K_{IC} is the plane-strain toughness and σ_y is the yield stress.

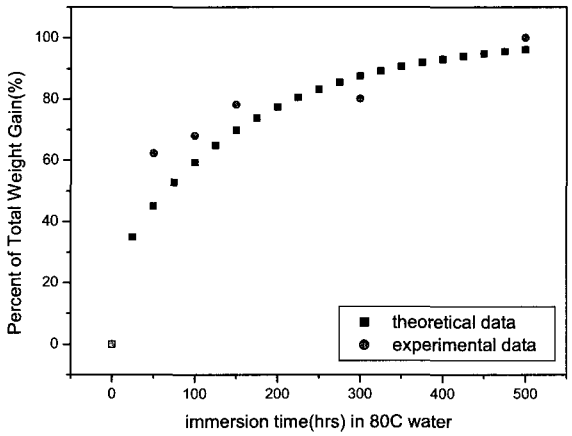
Under the plane-strain conditions, K_{IC} can be converted to the G_{IC}(E=3,760Mpa, ν=0.365).

2) Results and discussion

The average original weight for two samples was 16.78g. After 500 hours, the weight went up to 17.04g which was a 1.56% increase. The weight gain in 100 hours was a 1.06% increase. The detail data are in Table 1. In Fig. 1, the percent of total weight gain was estimated from each percent of weight gain divided percent of weight gain for 500 hours.

[Table 1] The experimental percent of weight gain for Avimid K3B neat resin

immersion time(hrs)	50	100	150	300	500
weight gain(%)	0.97	1.06	1.22	1.25	1.56



[Fig. 1] The percent of total weight gain as a function of immersion time at 80C water for neat resin K3B with diffusion coefficient $7.0\times10^{-6}m^2/s$

The percent of total weight gain for 500 hours is 100%. which means saturation. In only 50 hours, the percent of total weight gain increased to 62.20%, and then increase slowly. After 500 hours, the percent of total weight gain no longer increased or the sample was almost saturated. The theoretical data were drawn with the diffusion coefficient $7.0\times10^{-6}m^2/s$.

To understand the water diffusion into the neat resin K3B, the diffusion coefficient D, whose magnitude is indicative of the rate at which atoms diffuse, was introduced. Fick's second law are as follows:

$$\frac{\partial C}{\partial t}=D\frac{\partial^2 C}{\partial x^2} \tag{3}$$

where, C is concentration in terms of weight % or mass of diffusing species per unit volume (kg/m^3). D is called the diffusion coefficient whichh is expressed in square meters per second (m^2/s). t is time (sec).

Let us consider the model as the slab whose boundary conditions are :

for t=0, C=0 at $0\le x\le B$,

for t>0, $C=C_{sat}$ (saturated concent.) at x=0

$C=C_{sat}$ at x=B (thickness).

Applying these boundary conditions to Fick's second law yields the following solution (4) :

$$C(x,t)=C_{sat}+\frac{2}{B}\sum_{m=1}^{\infty}e^{-D\beta_m^2 t}\frac{1}{\beta_m}\sin\beta_m x \qquad (4)$$

$$(C_{sat}\cos m\pi - C_{sat})$$

where, $C(x,t)$ represents the concentration at length x after time t, and $\beta_m=\frac{m\pi}{B}$.

Taking a few terms in Eq. (4) and calculating the average value of the diffusion coefficients inside the body, the results were obtained (refer to Table 2).

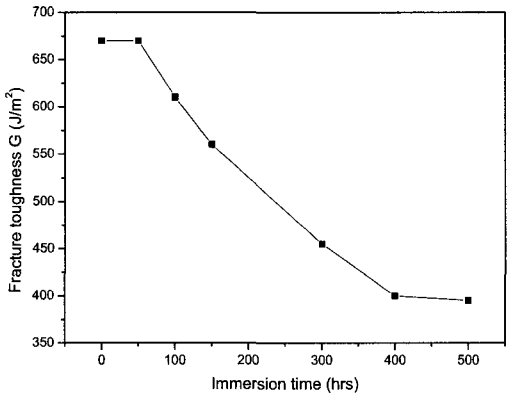
Figure 2 shows the changes in fracture toughness as a function of immersion time. The results present the average fracture toughness for two replicate experiments. The average toughness shows a gradual loss as the aging time increases. The unaged fracture toughtness was $670J/m^2$. The 100 hours aging changed the microcracking toughness to $610J/m^2$ which corresponded to a 9% decrease of the original toughness. In 500 hours, the loss of the microcracking toughness was 41% of the original toughness.

[Table 2] The theoretical percent of weight gain for Avimid K3B neat resin as a function of immersion time in 80℃ water

immersion time(hrs)	50	100	150	300	500
weight gain(%)	0.70	0.93	1.09	1.36	1.50

[Table 3] The fracture toughness for Avimid K3B neat resin as a function of immersion time in 80℃ water

immersion time (hrs)	0	50	100	150	300	500
fracture toughness (J/m^2)	670	670	610	560	455	395



[Fig. 2] The fracture toughness as a function of aging time in 80C water for neat resin K3B.

2.2 The aging of Avimid® K3B/IM7 in 80℃ Water

1) Materials and Methods

The layup of Avimid® K3B/IM7 laminates was [+45/0/-45/90]s. The sample dimensions were 12.6 mm wide, 100mm long and had the thickness determined by the stacking sequence.

Before the samples were put in the water, the weight of the samples was measured. The samples then were placed in a container which contained water maintained at 80℃ for 10 hours through 100 hours. Before static tensile tests, the samples were dried out to the constant weight. The samples were then loaded on the MTS frame using a cross head rate of 0.01mm/sec until the samples were broken or the end-tabs failed.

2) Results and discussion

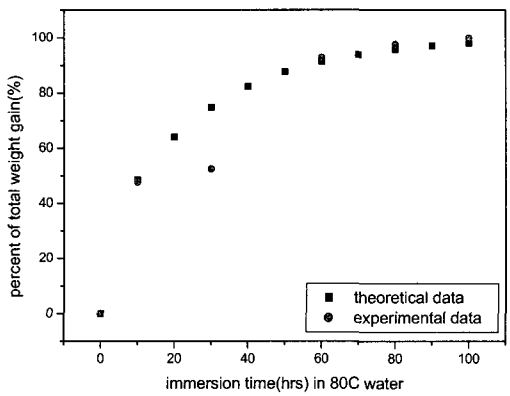
The average weight gain for two samples in 100 hours was 0.01g which corresponded to a 0.42% increase. The data are in Table 5.

Figure 3 describes the percent of total weight gain as a function of immersion time at 80℃ water for Avimid® K3B/IM7.

The percent of total weight gain was calculated from percent of weight gain divided by percent of weight gain of 100 hours. In 60 hours, the percent of total weight gain increased to about 95%, and then increased slowly. The red circle data are the experimental data and the black square data are the theoretical data with diffusion coefficient $1.0\times10^{-6}m^2/s$. Referring to Fig. 1 and

[Table 5] The percent of weight gain for Avimid K3B/IM7 as a function of immersion time in 80℃ water

immersion time(hrs)	10	30	60	80	100
the first sample weight gain(%)	0.18	0.20	0.40	0.43	0.43
the second sample weight gain(%)	0.22	0.24	0.38	0.40	0.41
average(%)	0.20	0.22	0.39	0.41	0.42



[Fig. 3] The percent of total weight gain for Avimid K3B/IM7 [+45/0/-45/90]s laminates as a function of immersion time at 80℃ water with the diffusion coefficient $1.0\times10^{-6}m^2/s$.

Fig. 3, the 80℃ water diffusion rate into the neat resin K3B was faster than diffusion rate into the Avimid® K3B/IM7.

Figure 4 shows in detail how the 80℃ water affected the aging of K3B/IM7 laminates as a function of immersion time. As the immersion time increased, the higher microcrack density was found at a lower stress. In fig. 4, the line was obtained the following variational mechanics analysis[2].

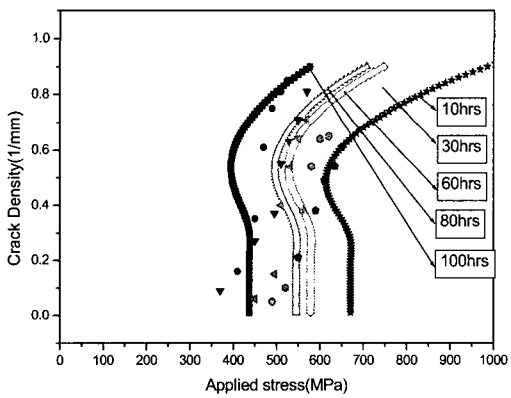
The variational mechanics analysis determines all components of the stress tensor in the x-z plane. In this paper, we only require the tensile stress in the 90° plies. The resul is[3]

$$\sigma_0 = \frac{1}{k_m^{(1)}}\left(\sqrt{\frac{G_{mc}}{G_3 t_1 Y(D)}} - k_{th}^{(1)} T\right) \tag{5}$$

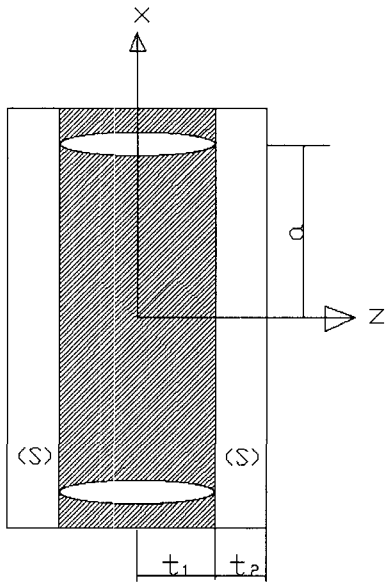
Where, the terms $k_m^{(1)}$ and $k_{th}^{(1)}$ are the effective mechanical and thermal stiffnesses of the 90° plies.

$$k_m^{(1)} = \frac{E_x^{(1)}}{E_c^0} \text{ and } k_{th}^{(1)} = -\frac{\Delta\alpha}{C_1} \tag{6}$$

Here E_c^0 is the x-direction modulus of the



[Fig. 4] The microcrack density as a function of the applied load in Avimid K3B/IM7 [+45/0/-45/90]s laminates



[Fig. 5] Edge view of microcracks in the 90° plies of $[(S)/90_n]_s$ laminates. ((s) is sublaminates)

laminate, $E_Z^{(1)}$ is the x-direction modulus of the 90° plies, $\Delta\alpha = \alpha_x^{(1)} - \alpha_x^{(2)}$ is the difference between the x-direction thermal expansion coefficients of the 90° plies and the (S) sublaminate, and C_1, C_3 are the constants defined in the Ref (3). $D = \frac{N}{L}$ is the average crack density, N is the number of cracks and L is the sample length and Y is a function defined in Ref. (3). In eq. (5), there are two unknowns G_{mc} (microcracking fracture toughness), T(the temperature difference that determines the level of residual stresses). T can be measured by various means. Therefore if we have an unknown G_{mc}, we can predict the experimental results (stress(σ_0) vs microcrack density(D)). The unknown G_{mc} can be obtained from the comparison the experimental data with the theoretical line drawn from eq. (5) using a single value of G_{mc} in Table 5 from the mechanical properties in Table 6. In fig. 6, the solid lines were obtained like this method.

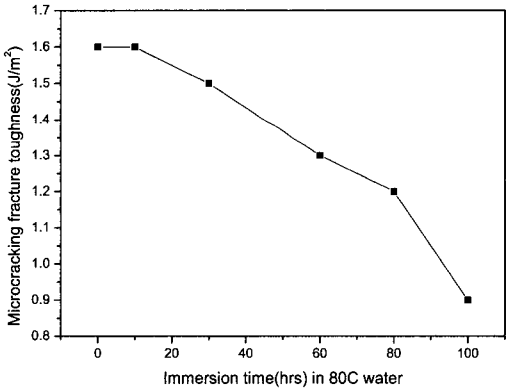
Figure 6 shows how the 80℃ water has

[Table 5] Microcracking fracture toughness for Avimid K3B/IM7 as a function of immersion time in 80℃ water

immersion time(hrs)	0	10	30	60	80	100
Microcracking Fracture Toughness(J/m^2)	1.6	1.6	1.5	1.3	1.2	0.9

[Table 6] The mechanical properties for Avimid K3B/IM7 unidirectional laminate

E_A (GPa)	E_T (GPa)	G_A (GPa)	G_T (GPa)	ν_A	ν_T	α_A (ppm /℃)	α_T (ppm /℃)	T_{eff}
134	9.8	5.5	3.6	0.3	0.5	-0.09	28.8	-225



[Fig. 6] The microcracking fracture toughness as a function of immersion time in Avimid K3B/IM7 [+45/0/-45/90]s laminates in 80℃ water

changed the microcracking toughness of K3B/IM7 laminates as a function of aging time. The unaged microcracking toughtness was $1.6J/m^2$. The 30 hours aging changed the microcracking toughness to $1.5J/m^2$ which corresponded to a 6.3% decrease of the original toughness.

In 100 hours, the loss of the microcracking toughness was 43.8% of the original toughness.

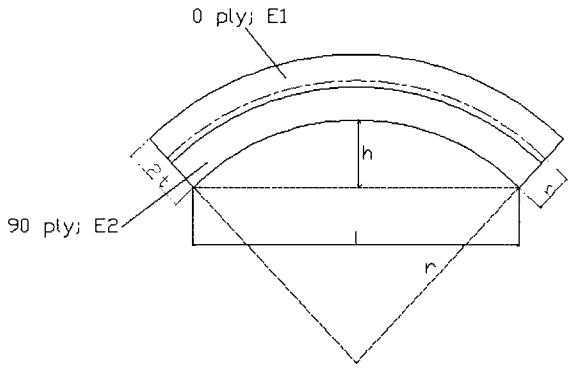
Therefore, the microcracking toughness of the saturated laminates in 80℃ water was degraded almost half.

The factors causing the 80℃ water to degrade the microcracking toughness of the laminates could be the degradation of the matrix fracture toughness, a change in residual stresses. Referring to Fig. 2, the 80℃ water full developed aging whichh is 160℃ lower than the glass transition temperature changed 41% of the original toughness of the neat resin K3B. Therefore, the main factor to degrade the microcracking toughness of the laminates was the degradation of the matrix fracture toughness.

2.3 The residual stress of Avimid® K3B/IM7 in 80℃ Water

1) Materials and Methods

To see whether the residual stress influenced the microcracking toughness of the laminates, two ply [90°/0°] laminates put into 80℃ water from 2 hours to 8 hours. Because the big difference of hygrothermal expansion coefficients between 90° and 0° ply, [90°/0°] laminates were chosen to measure the residual stress(refer to Fig. 7). The laminates dimensions were 0.25mm thick, 12mm wide, 100mm long. After measuring l and h with a traveling microscope, the radius of curvature could be calculated using trigonometric equation.



[Fig. 7] Two ply [90°/0°] laminates of AvimidR K3B/IM7. n is the neutral positon

2) Results and Discussion

The neutral position n and the radius of curvature r are as follows. Here, E_1 is the longitudinal modulus and E_2 is the transverse modulus of the unidirectional laminate and t is the thickness of a ply. Because of the thin thickness, the issue of plate effect is probably ignored. From the relation between stress and strain, residual stresses at the bottom surface of the 90° ply were calculated in Table 8.

$$n=\frac{t(E_2+3E_1)}{2(E_1+E_2)},\ r=\frac{(\frac{l}{2})^2+h^2}{2h} \quad (7)$$

[Table 7] The changes of the residual stress for the two ply [90°/0°] laminate as a function of immersion time

		before aging	after aging	change (MPa)
1st test	2 hours	60.6	62.3	1.7
	6 hours	60.5	62.5	2.0
	8 hours	64.3	67.1	2.8
2nd test	2 hours	57.1	58.8	1.7
	6 hours	57.7	59.3	1.6
	8 hours	59.1	61.0	1.9

3. Summary

In the present work, the effects of matrix hygrothermal aging and residual stress changes were studied to research the hygrothermal aging on Avimid® K3B/IM7 laminates in 80℃ water. The factors causing the 80℃ water to degradation of the laminates could be the degradation of the matrix toughness, the change in residual stresses. The times to saturation in 80℃ water for the laminates and the neat resin were 100 hours and 500 hours. After 500 hours aging of the neat resin, the weight gain was 1.55% increase with the diffusion coefficient $7\times10^{-6}m/s^2$. The fracture toughness was decreased about 41% by 3-point bending test.

After 100 hours fully saturated aging of the $[+45/0/-45/90]_s$ K3B/IM7 laminates in 80℃ water, the weight gain was 0.41% increase with the diffusion coefficient $1\times10^{-6}m/s^2$. The 80℃ water diffusion rate into the neat resin was faster than into the laminates. In 100 hours, the loss of the fracture toughness of the laminates was 43.8% of the original toughness by the microcracking fracture toughness criterion. To see whether the residual stress influenced the fracture toughness, two ply [90°/0°] laminates were put in 80℃ water from 2 hours to 8 hours. The changes in residual stress in 8 hours are less than 3MPa. Because a 3MPa change is not sufficient to degrade the laminates, the main factor to degrade the microcracking fracture toughness was the degradation of the matrix fracture toughness.

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