**Thermal ageing degradation mechanisms on compressive behavior of 3-D
braided composites in experimental and numerical study**

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Abstract

This paper reports the thermal ageing degradation mechanisms on compressive damage behaviors of pure epoxy resin and 3-D carbon fiber/epoxy braided composites. The epoxy resin and the 3-D braided composite have been aged in air for 1, 2, 4, 8, and 16 days at 180°C. The compressive testing results and the scanning electron microscope (SEM) observations revealed that the epoxy resin degradation only occurred within regions close to surface layer. The inner parts of the epoxy resin were protected from the ageing oxidation by the outside oxidized layer. The compressive behaviors degradation of the braided composite is attributed to the epoxy resin degradation and interface debonding. The numerical study (Finite element analysis, FEA) illustrates the interface crack propagation. The interface degradation and crack propagation are the main mechanisms of the compressive behaviors degradation after the several-day's high temperature ageing.

Keywords Thermal degradation; epoxy resin; 3-D braided composites; degradation mechanisms; compression; finite element analysis (FEA).

1 Introduction

Three-dimensional (3-D) braided composite is widely used in aeronautical engineering due to its high damage tolerance and fatigue resistance [1]. The mechanical behaviors degradation of the 3-D braided composites under long time high temperature ageing is the key to aircraft design. There are many investigations on thermal degradations of polymer-matrix composites. Different methods have been used to investigate the effect of accelerated thermal degradation on polymer materials and composite materials [2-7]. Tsotsis [8-10] found that the weight loss during thermo-gravimetric test could not be used as criteria for material acceptance. The mechanical behaviors degradation should be characterized after thermal ageing. For the ageing test, it was found that the elevated pressure may be a good tool to reduce ageing time for the materials subjected to long exposures in oxygen-containing environments at elevated temperatures [11-13].

So far, the thermal ageing studies are mainly focused on laminated composite. As for 3-D braided composite, Song *et al* [14] studied the change of tensile properties of 3-D braided composites after different ageing times at different high temperatures. They found that the ageing time had a significant effect on tensile strength of the composites. Fan *et al* [15] investigated the influence of thermo-oxidative ageing on impact properties of braided composites and laminated plain woven fabric composites.

In this paper, we will concentrate on the compressive behaviors of 3-D carbon fiber/epoxy braided composites after thermal ageing. Because the braided preform is

embedded into the resin matrix, the compression behaviors are more dependent on the resin and more sensitive to thermal degradation. The compressive behaviors of the epoxy resin and the braided composite after thermal ageing will be presented. A finite element analysis (FEA) model was developed to reveal the failure mechanisms of the braided composite from the resin ageing and fiber/resin interface crack.

2 Experimental

2.1 Materials

The materials used in this study were T700S-12K carbon fiber supplied by Toray Inc. (Japan) and JA-02 epoxy resin supplied by Changshu Jiafa Chemical Inc. (China).

The 3-D braided preform (shown in Fig.1) with square cross section of 11×11 braiding yarn arrays, was manufactured with a four-step 1×1 braiding technique.

Epoxy resin was injected into the braided preform with vacuum assisted resin transfer molding (VARTM) technique. For comparison between the braided composite and the pure epoxy resin, an epoxy cube was prepared. Both the pure epoxy resin and the braided composite were consolidated with the same curing process: $90^{\circ}\text{C}/2\text{hrs} +$

$110^{\circ}\text{C}/1\text{hr} + 130^{\circ}\text{C}/4\text{hrs}$. As shown in Fig.2, the glass transition temperature (T_g) of the epoxy resin is 110°C based on our previous dynamic mechanical analyses (DMA) results [16]. The braided composite was cut into about 12.2mm along longitudinal direction which equals to the size of the braided cross section, i.e., the size of the braided composite cube is $12.2 \times 12.2 \times 12.2\text{mm}$. The epoxy cube has the same size

with the braided composite coupon. The surface braiding angle for the braided composite is 20°. The fiber volume fraction is 38%.

2.2 Thermal ageing

All testing coupons were pretreated in an oven for 1 hour at 80°C and then divided into six groups. One group (base group) was tested for comparing the ageing effect with other groups after several-day's ageing at 180°C.

The other five groups were aged for 1, 2, 4, 8, and 16 days at 180°C in air respectively. When the ageing time reached, the specimens were cooled down to ambient temperature and placed into tight plastic bags to prevent moisture absorption.

2.3 Microscopy observation

Scanning electron microscopy (SEM) photographs and optical microscopy photographs were used to observe the morphologies of all the coupons after thermal ageing. The microcracks and fiber surfaces were observed for illustrating the thermal ageing effect on composite coupons. The color change and the thickness of the oxidized layer in the pure epoxy resin were also observed.

2.4 Compression test

Compression tests were performed on MTS 810 material test system and the compressive speed was set as 2mm/min. The tests were at room temperature (20°C).

Each test was repeated three times and the average compressive behaviors were obtained. Fig.3 shows the out-of-plane compression.

During the compression tests, the compression load was obtained from the loading sensor of the MTS 810 tester and the compression deformation was assumed to equal to the stroke of the loading cell. Then the engineering stress of the composite coupons

was the quotient of the load and the cross-section area. The engineering strain is the quotient of the stroke of the loading cell and the coupon size along compression direction. Because the transverse strain was not measured, the strain gauge was not used to measure the local strain of the composite coupon. The engineering stress-strain curve was obtained to compare the compressive behaviors degradation after the composite ageing.

3 Modeling

3.1 Geometrical modeling

Based on the microstructure of 3D braided preform and the impregnating resin, the geometrical model was established as shown in Fig.4. It was assumed that all the fiber tows were completely impregnated by resin during the composite VARTM consolidation. In the finite element model, there were 279,282 solid elements for the entire microstructure model, where 252,141 elements for the epoxy resin and 23,045 elements for yarns and 4,096 elements for the clamp.

3.2 Material properties

There are gaps among fibers in a fiber tow. The gaps will be filled with resin during composite fabrication. It is more accurate to consider a fiber tow as a transversely isotropic unidirectional composite rather than a pure carbon fiber tow. The bridging model of Huang [17] was used for calculating the stiffness matrix of the fiber tow. It was assumed that the mechanical properties of the fiber tow remained unchanged because the carbon fibers were relatively stable during thermal ageing. The mechanical behaviors of the epoxy resin after ageing were introduced into the model

to calculate the compressive behaviors. Then the temperature-independent constitutive relationships could be avoided for the numerical study. Only the elastic-plastic constitutive relationships of the epoxy resins were used for FEA calculation.

3.3 Constitutive relationships

The compressive stress-strain curves of the epoxy resin and the braided composites both exhibit a yield region, where the strain increase is accompanied by a slow increase in stress. Before the stress reaches the maximum strength point, the total strain can be expressed as a sum of elastic (completely recoverable) and plastic (residual) strains:

$$\varepsilon^{\Sigma} = \varepsilon^e + \varepsilon^p \quad (1)$$

To define a work-hardening relationship, stress must be given as a function of the equivalent plastic strain. The equivalent plastic strain is zero before the stress reaches the yield strength, after which the equivalent plastic strain increases due to plastic deformation as the loading proceeds.

It is assumed that the matrix in this study is an elastic-plastic solid obeying the J2-isotropic hardening plasticity theory with an associated flow rule and a von Mises yield criterion [18].

Hill's anisotropic plasticity model is used to define the yield properties of fiber tow. Hill's stress function is a simple extension of the Mises function to allow anisotropic behavior. The function is

$$f(\sigma) = \sqrt{F(\sigma_y - \sigma_z)^2 + G(\sigma_z - \sigma_x)^2 + H(\sigma_x - \sigma_y)^2 + 2L\tau_{yz}^2 + 2M\tau_{zx}^2 + 2N\tau_{xy}^2} \quad (2)$$

in terms of rectangular Cartesian stress components, where F, G, H, L, M, N are

constants obtained by tests of the material in different directions. They are defined as

$$\begin{aligned}
 F &= \frac{\sigma_0^2}{2} \left(\frac{1}{\bar{\sigma}_{22}^2} + \frac{1}{\bar{\sigma}_{33}^2} - \frac{1}{\bar{\sigma}_{11}^2} \right), \\
 G &= \frac{\sigma_0^2}{2} \left(\frac{1}{\bar{\sigma}_{33}^2} + \frac{1}{\bar{\sigma}_{11}^2} - \frac{1}{\bar{\sigma}_{22}^2} \right), \\
 H &= \frac{\sigma_0^2}{2} \left(\frac{1}{\bar{\sigma}_{11}^2} + \frac{1}{\bar{\sigma}_{22}^2} - \frac{1}{\bar{\sigma}_{33}^2} \right), \\
 L &= \frac{3}{2} \left(\frac{\tau_0}{\bar{\tau}_{23}} \right)^2, \\
 M &= \frac{3}{2} \left(\frac{\tau_0}{\bar{\tau}_{13}} \right)^2, \\
 N &= \frac{3}{2} \left(\frac{\tau_0}{\bar{\tau}_{12}} \right)^2,
 \end{aligned} \tag{3}$$

where $\sigma_0, \bar{\sigma}_{11}, \bar{\sigma}_{22}, \bar{\sigma}_{33}, \bar{\tau}_{12}, \bar{\tau}_{23}, \bar{\tau}_{13}$ are specified and $\tau_0 = \sigma_0 / \sqrt{3}$. $\bar{\sigma}$ and $\bar{\tau}$ are the

values of stress that make the potential equal to σ_0 if only one stress component is nonzero.

3.4 Definitions of materials' contact

During thermal ageing process, the mechanical properties' degradation occurred not only in the epoxy resin, but also in the fiber/resin interface. In the FEA model, the 'surface to surface contact' was chosen for contact definition. The fiber tows' surface was defined as the master surface, while the resin surface was regarded as the slave surface.

3.4.1 Linear elastic traction-separation behavior

Surface-based cohesive behavior was used to describe the materials contact and to

model the debonding at interfaces directly in terms of traction versus separation. The traction-separation model assumes initially linear elastic behavior followed by the initiation and evolution of damage. The elastic behavior is established in terms of an elastic constitutive matrix that relates the normal and shear stresses to the normal and shear separations across the interface.

$$\mathbf{t} = \begin{Bmatrix} t_n \\ t_s \\ t_t \end{Bmatrix} = \begin{bmatrix} K_{nn} & K_{ns} & K_{nt} \\ K_{ns} & K_{ss} & K_{st} \\ K_{nt} & K_{st} & K_{tt} \end{bmatrix} \begin{Bmatrix} \delta_n \\ \delta_s \\ \delta_t \end{Bmatrix} = \mathbf{K} \boldsymbol{\delta} \quad (4)$$

where $\mathbf{t}$ represents the nominal traction stress vector, the three components: t_n, t_s , and

t_t represent the normal (along the local 3-direction) and the two shear tractions (along

the local 1- and 2-directions) respectively. δ_n, δ_s , and δ_t are the corresponding separations.

3.4.2 Damage initiation

Damage initiation in the interface was based on maximum stress criterion. Damage is assumed to initiate when the maximum contact stress ratio (as defined in the expression below) reaches a value of one. This criterion can be represented as:

$$\max \left\{ \frac{\langle t_n \rangle}{t_n^0}, \frac{t_s}{t_s^0}, \frac{t_t}{t_t^0} \right\} = 1 \quad (5)$$

where t_n^0, t_s^0 , and t_t^0 represent the peak values of the contact stress when the separation

is either purely normal to the interface or purely in the first or the second shear

direction, respectively. The symbol $\langle \rangle$ represents the Macaulay bracket with the usual interpretation. The Macaulay brackets are used to signify that a purely compressive displacement or a purely compressive stress state does not initiate damage.

3.4.3 Damage evolution

The damage evolution law describes the rate at which the cohesive stiffness is degraded once the corresponding initiation criterion is reached. This process can be defined based on the fracture energy, which is equal to the area under the traction-separation curve. The Benzeggagh-Kenane fracture criterion [19] was used herein to govern the damage evolution process. It is given by

$$G_n^C + (G_s^C - G_n^C) \left\{ \frac{G_s}{G_T} \right\}^\eta = G^C, \quad (6)$$

where $G_s = G_s + G_t$, $G_T = G_n + G_s$, and η is a cohesive property parameter.

A scalar damage variable, D , represents the overall damage at the contact point. It initially has a value of 0. If damage evolution is modeled, D monotonically evolves from 0 to 1 upon further loading after the initiation of damage. The contact stress components are affected by the damage according to

$$\begin{aligned} t_n &= \begin{cases} (1-D) \bar{t}_n, & \bar{t}_n \geq 0 \\ \bar{t}_n, & \text{otherwise (no damage to compressive stiffness)} \end{cases} \\ t_s &= (1-D) \bar{t}_s, \\ t_t &= (1-D) \bar{t}_t, \end{aligned} \quad (7)$$

where $\bar{t}_n$, $\bar{t}_s$, and $\bar{t}_t$ are the contact stress components predicted by the elastic traction-separation behavior for the current separations without damage.

A linear softening behavior was chosen to specify the damaged response, and the damage variable, D , could be expressed as follows:

$$D = \frac{\delta_m^f (\delta_m^{\max} - \delta_m^0)}{\delta_m^{\max} (\delta_m^f - \delta_m^0)}, \quad (8)$$

where $\delta_m^f = 2G^C / T_{eff}^0$ with T_{eff}^0 as the effective traction at damage initiation. $\delta_m^{\max}$ refers

to the maximum value of the effective separation attained during the loading history.

Due to the degradation of the interface property, the data specified for damage initiation and damage evolution would be lowered in the later stage of the ageing.

Once the damage initiation criterion is satisfied, the damage state is calculated and updated based on the Equations (6) and (8). When $D = 1$, the interface will debond.

4 Results and discussions

4.1 Optical changes

It can be observed from Fig.5 that color changes appeared both in the pure epoxy resin and the braided composites as ageing time increased. The pure epoxy resin (Fig.5 (a)) turned black when exposure time reached 16 days. The surface resin of the braided composite, by contrast, gave a brownish tinge to the specimens (Fig.5 (b)).

The color change indicates that there are chemical reactions that occurred in the epoxy resin due to thermal degradation. These observations are in accordance with previous work of Tsotsis et al [10]. It could be concluded that the epoxy resin oxidation is detrimental to the compressive behaviors of the braided composite.

4.2 Microscopic morphology

Figs.6-8 show the morphologies of specimens as the ageing time increased. Fig.6 (a)

is the surface photograph of the unaged composite. There is no evident crack between the carbon fiber tows and the epoxy resins. As shown in Fig.6 (b) and Fig.6 (c), the cracks could be obviously shown as the ageing-day increases. The cracks between fiber tows and resins shown in Fig.6 (b) and Fig.6 (c) are due to the different thermal stress in fibers and resins during the high temperature ageing. The coefficients of thermal expansion (CTE) of carbon fiber are -1.0×10^{-6} and $12.0 \times 10^{-6}/^{\circ}\text{C}$ in the longitudinal and transverse directions, respectively [20]. The CTE is $43.62 \times 10^{-6}/^{\circ}\text{C}$ for fully cured epoxy resin and $87.02 \times 10^{-6}/^{\circ}\text{C}$ for partially cured epoxy resin above the glass transition temperature (T_g) [21]. For the un-aged composite coupon at room temperature, the residual stress at the fiber/resin interface is not greater than the interface bonding strength. There are not visible cracks in the composite coupon surface. While at high temperature of 180°C , the mismatch of the thermal stress between the carbon fiber and the epoxy resin will be the main reason for the interface crack development. For example, if the room temperature is 20°C , the axial modulus of the carbon fiber is 230GPa, then the axial thermal stress in the carbon fiber approximates to $230 * (-1.0 \times 10^{-6}) * (180-20) = -36.8\text{MPa}$. The thermal stress for the epoxy resin is $3.4 * (43.62 \times 10^{-6}) * (180-20) = 23.7\text{MPa}$. The thermal stress difference approximates to 60.5MPa. As the ageing time continues, the interface crack will be propagated gradually with the epoxy resin deformation and damage as shown in Fig.7 (the axial tensile strength of epoxy resin at room temperature is about to 72MPa [22]). Fig.8 shows the epoxy resin coupons isothermally aged in air at 180°C at the different days. The oxidized layer appears darker than the specimen core. The region between

the outside oxidized layer and inner un-oxidized core is a transition region. This region almost disappears after 16 days' exposure. The thickness of the oxidized layer is seen to approach a plateau value of 0.8mm as the oxidation growth rate reduces considerably for longer ageing time periods. That is to say, the oxidized layer may reach a limit thickness. The oxidized layer protects the inner epoxy from further oxidation. The previous works [23-25] also confirmed this phenomenon.

4.3 Compression behaviors

4.3.1 Comparisons between the epoxy resin and the braided composites

Fig.9 and Fig.10 show the compression behaviors of the epoxy resin and the braided composite after ageing for different days. The compressive behaviors of the epoxy resin changes significantly after 1-day ageing. While after the 1-day ageing, the compressive stress strain curves become stable. This is probably attributed to the outside ageing layer prevent the further oxidization of the inner resin. For the braided composite, the stress strain curves decreases continuously as the ageing time. The reason is probably the crack development at the interface during the long time ageing. Fig.11 (a) and (b) illustrate the different degradation mechanisms of the two materials. No distinct crack was observed in pure resin at the first. The outside oxidation layer protects the inner cores from further degradation. While for the braided composite, the fiber/resin interface crack will provide pathways for the further oxidation in the inner cores. This leads to the further crack formation and oxygen penetration (see Fig.11 (b)). Both the outer layer epoxy resin degradation and inner interface oxidation lead to the continuous reduction of the compressive behaviors of the braided composite as

shown in Fig.10

4.3.2 Validation of the FEA model

Fig. 12 compares the stress strain curves between FEA and experimental for 1-day ageing. In Fig. 12(a), there are evident discrepancies in the stress-strain curves between experimental and FEM. The experimental curve shows a tiny slow-growth region at the initial stage. This initial nonlinear region in experimental results was also widely observed in previous studies [26-30]. The marked compressive nonlinearity at the start of the test is possibly from the contact between the compressive fixtures and the specimen, and also from the inner voids of the specimen. The first, the surface of the specimen is not perfect smooth. The contact between the specimen and the compressive fixture at the start of the test occurred at the peak regions of the specimen. Then the compressive load will increase gradually. This will leads to the initial slow-growth stage in the compressive stress-strain curve in experimental. Furthermore, some defects such as micro-voids formed during the processing of composites may also contribute to this phenomenon. And also, the viscoelasticity of the epoxy resin is also a possible reason for the initial slow-growth of the stress strain curve.

For the finite element model with an ideal geometrical structure and no micro-voids inside, in contrast, possesses a perfect contact and thus increases linearly at the start of the stress strain curve. If the initial slow-growth stage of the experimental stress strain curve was removed and then parallel moved to the left, as shown in Fig.12 (b), the stress strain curves between the FEA and the experimental agree well. This suggests

the FEA model could simulate the compressive stress strain curves well except the initial stage. If the viscoelasticity of the epoxy resin was introduced in the finite element model, the agreement of the stress strain curve between the FEA and the experimental would be better.

The FEA model was also used to calculate the compressive stress strain curves with different ageing days as shown in Fig.12 (c). Both the compressive stiffness and strength were well predicted.

4.3.3 Stress distribution and interfacial damage

Figs. 13-17 show the stress distributions evolution in the braided composite, the resin matrix and the braided preform after exposure at 180°C for different days. Four representative moments were chosen to describe the stress transfer process (see Fig.13). The braided preform carries most of the compressive load, as shown in Fig.14. The stress carried by matrix distinctly reduces due to its stiffness degradation as shown in Fig.15. Although the carbon fiber tows are assumed as non-degradable materials, the stress carried by the braided preform also decreases (Fig.16). Fig.17 illustrates that the stress transfer is heavily dependent on the reinforcement architecture. For both the resin and the braided preform, the corners and interior regions bear the majority of the applied load. Moreover, stress concentration occurs at the buckling segments in fiber tows. It can be explained that the stress is more difficult to transfer through the buckling segments than in the straight parts. Fig.18 shows the interfacial damage evolution after ageing for different days. The grayscale represents the damage variable D in Section 3.4.3. Fig.19 reveals that most

of the cracks are from the fiber/resin interface debonding. It indicates that the interface failure is the main failure mode under the out-of-plane compression. Both the damage morphologies from SEM photographs and FEA simulations show that interfacial damage becomes more serious as the ageing time increases. Fig.20 suggests that the surface exposed to air is seriously damaged, while the inner location suffers less damage. This phenomenon may lead to the conclusion that interface damage occurs in the surface first and then expand to the interior regions. Outer interface debonding leads to further oxygen penetration and finally induce the oxidation of the inner parts.

5 Conclusions

The thermal ageing effect on the compressive behaviors of pure epoxy resins and 3-D carbon fiber / epoxy braided composites were investigated and numerically illustrated. The composite coupons have been exposed to the 180°C air for 1, 2, 4, 8, and 16 days. The compressive behaviors of the pure epoxy resins and the braided composite were tested for finding the ageing effect for several days. The surface cracks were analyzed with scanning electron microscopy (SEM) photographs for explaining the thermal ageing of the pure epoxy resins and the braided composites. The FEA model was established at microstructure level to simulate the stress distribution after ageing. For the pure epoxy resin, the degradation only occurred within a surface layer. The inner cores were protected from oxidation. For the braided composite, the interface crack provided pathways for oxygen diffusion and thus led to further degradation. A finite element model was established to investigate the stress transfer and distribution of the

3D braided composites with different ageing time. The resin degradation and the fiber/resin interface crack were the main reasons for thermal degradation of the 3-D braided composites. The resin modification and interface strengthening may effectively improve composites' heat resistance and thermal degradation.

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References

Fig.1 Three-dimensional carbon fiber rectangular braided perform in 11×11 array.

Fig.2 DMA temperature sweep results of epoxy resin [16]

Fig.3 Compression tests

(a) Compressive test on MTS 810 material test system; (b) out-of-plane compression.

Fig.4. Geometrical model and mesh schemes
(a) geometrical model; (b) clamp; (c) preform; (d) resin.

Fig.5 Specimens after thermal ageing (a) epoxy resin aged for 1, 2, 4, 8, 16 days and the un-aged one; (b) composite samples aged for 1, 2, 4, 8, 16 days and the un-aged one.

Fig.6 SEM images of the composite specimens (a) cross section of un-aged specimen;
 (b) cross section of specimen aged for 8 days; (c) cross section of specimen aged for
 16 days.

Fig.7 SEM images of a crack on the cross section of composite specimen aged for 16

days.

Fig.8 Photomicrographs of epoxy resin showing the formation of oxidized region.

Fig.9 Accelerated thermal ageing effect on compression behaviors of epoxy resin

Fig.10 Accelerated thermal ageing effect on compression behaviors of 3-D braided composites

Fig.11 Sketch diagrams for degradation mechanisms (a) degradation mechanism of pure epoxy resin: the oxidized layer reaches a self-limit thickness and protects cubic resin from further degradation; (b) degradation mechanism of 3D braided composite system: interfacial debonding provides pathways for oxidation and further

degradation.

(a)

(b)

Fig.12 Stress-strain curves of finite element model: (a) Comparison between FEM and experimental results of specimen aged for 1 day; (b) Comparison between FEM and the shifted experimental curve of specimen aged for 1 day; (c) Stress-strain curves of finite element model with different ageing times.

Fig.13 Four representative points in stress-strain curve of FEA model: A- the starting

point of the curve; B- a point at elastic response phase; C- a point in the yield zone;
D- the end point of the curve.

Fig.14 Stress distribution of the 3D braided composites (a) stress distribution of the unaged composite; (b) stress distribution of composite aged for 8 days at 180°C; (c)

stress distribution of composite aged for 16 days at 180°C.

Fig.15 Stress distribution of the resin aged after different days at 180 °C (a) the unaged one; (b) aged for 8 days; (c) aged for 16 days.

Fig.16 Stress distribution of braided preform aged after different days at 180 °C, D* represents the semi-section view of moment D: (a) the unaged one; (b) aged for 8 days; (c) aged for 16 days.

Fig.17 Stress distribution of the unaged resin (a) the surface contact with press head;
(b) the side surface.

Fig.18 Damage evolution of the fiber/resin interface aged after different days at 180

°C: (a) the unaged one; (b) aged for 8 days; (c) aged for 16 days.

Fig.19 Interface damage morphologies in SEM images and finite element model.

Fig.20 Interfacial damage morphologies on 1/4, 1/2, 3/4 and entire cut section view perpendicular to Z direction of the unaged material