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Bistable Behaviour and Microstructure Characterization of Carbon Fiber/epoxy Resin Anti-symmetric Laminated Cylindrical Shell after Thermal Exposure

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Abstract: Bistable behaviour and microstructure characterization of carbon fiber/epoxy resin anti-symmetric cylindrical shells after thermal exposure are investigated by experimental method in this paper. The effect of thermal exposure temperature and duration on the curvatures, load-displacement curves and snap loads of bistable shells are discussed systematically. The results show that both the thermal exposure temperature and duration have a significant influence on the bistable behaviour of those antisymmetric laminated cylindrical shells. Some interesting phenomena after high-temperature exposure are found in snap-through and snap-back processes. The microstructure of the shell before and after thermal exposure is characterized using scanning electron microscope (SEM), which provide an insight into bistable deformation mechanism of those shells after being exposed to elevated temperature.

Keywords: Composite structure; bistable cylindrical shell; anti-symmetric layup; thermal exposure; microstructure characterization.
1. Introduction

Carbon fiber/epoxy bistable composite shells have potential applications in aeronautical and mechanical engineering due to their multifunctional advantages and superior mechanical properties [1]. Several studies have been done on bistable behaviour of laminated cylindrical shells in recent years [2-10]. The bistable characteristics in high-temperature environment depends largely on the mechanical properties of fiber reinforced polymer composites around or above the glass transition temperature ($T_g$) of the resin matrix [11-12]. In many engineering application, the bistable shells are adjacent to a thermal source, by which the shells are first are exposed to a high temperature then are cooled down to room temperature after the thermal source is removed [13-18]. The effect of high-temperature exposure on bistable laminated cylindrical shells should be investigated and well understood before they are used as load-bearing or actuating structures in thermal environments.

Mouritz et al. [19] reported a research progress of the structural modelling of laminates and sandwich composites in fire. The influences of temperature, decomposition, phase change, damage, mechanical properties, and failure on composite structures were discussed in the paper. Foster et al. [20] investigated the residual properties of externally-bonded FRP systems after high-temperature exposure through five different series of tests. Severe reductions in residual tensile strength and stiffness were observed at temperature exceeding the thermal decomposition temperature of the epoxy polymer matrix. Akay et al. [21] examined the effect of long-term exposure to high-temperature environment on the interlaminar-shear strength and impact performance of carbon fiber reinforced bismaleimide composite. The results showed that the degradation of matrix and fiber-matrix interface with ageing, accordingly the interlaminar-shear strength deteriorated progressively and the failure mode of the impact specimens changed from a brittle failure in the unaged state to a progressive delamination in the aged state. Liu et al. [22-26] studied the mechanical behaviours of carbon fiber composite sandwich structures with vertical lattice truss and pyramidal lattice truss cores at high and low temperatures by
experimental and theoretical methods. The out-of-plane compressive and shear behaviours of composite sandwich structures after high-temperature exposure with different temperatures and durations were investigated. Thermal and mechanical properties of different carbon fiber composites have been studied by various researchers[27-29]. To the best knowledge of the authors, no previous work has emphasized on the bistable behaviour and microstructure characterization of anti-symmetric laminated cylindrical shell after thermal exposure.

The present paper studies the effect of thermal exposure on bistable behaviour and microstructures of anti-symmetric laminated cylindrical shells. First, the bistable shells were exposed to different temperatures for different durations. After high-temperature exposure, the snap processes of specimens are tested at room temperature using assembled testing machine [7]. The effects of thermal exposure temperature and duration on the bistable behaviour are investigated. In addition, the surfaces and fiber-matrix interfaces of specimens are observed by a scanning electron microscope (SEM) to provide an insight into the effect of high-temperature exposure from micro scale.

2. Experimental methods

In order to study the effect of thermal exposure on the bistable characteristics, a testing machine (Reger3010) with the thermal chamber is used in the experiment. The adjustable temperature range is 20 °C~350 °C, the glass transition temperature $T_g$ of anti-symmetric laminated cylindrical shell is 85 °C. The environmental temperature is controlled by the thermal chamber and the whole snap-through and snap-back processes are captured by the testing machine [9]. The thermal chamber is heated and stabilized at a certain temperature. Some specimens are exposed to temperatures of 20 °C (room temperature), 40 °C, 80 °C, 120 °C and 200 °C respectively for a duration of 1 h to study the effect of thermal exposure temperature. Other specimens were exposed to a temperature of 200 °C for different durations of 1 h, 3 h and 6 h to study the effect of thermal exposure duration.

The anti-symmetric laminated cylindrical shells made from 4-plies 0.48mm (0.12
mm thickness each ply) unidirectional T700/epoxy prepreg with the stacking sequence \([45^\circ/-45^\circ]_2\) are cured and cooled in a cylindrical steel mold before thermal exposure experiments. The entire experimental process for bistable behavior of T700/epoxy resin anti-symmetric laminated cylindrical shell under thermal exposure is shown in Fig.1. The specimens are exposed to temperatures of 20 °C (room temperature), 40 °C, 80 °C, 120 °C and 200 °C for duration of 1 h. All of the specimens are cooled down to room temperature, and the snap process of bistable shells are tested at room temperature. Each specimen is tested three times at every thermal exposure condition, and the average values of the experimental results are obtained as final results.

Using the testing method mentioned above, the load-displacement curves and snap loads are recorded by the computer using data acquisition software. The principal curvature \(C\) and twisting angle \(\theta\) of the bistable shells are measured by digital image processing technique [30]. Twisting deformation of bistable cylindrical shell after thermal exposure is observed, especially in the second stable state, as shown in Fig.2.
The curvature in the x direction $k_x$, the curvature in the y direction $k_y$, the twisting curvature in x-y plane $k_{xy}$ can be obtained using a Mohr’s circle [31,32] as below:

\[
\begin{align*}
    k_x &= \frac{C}{2}(1-\cos 2\theta) \\
    k_y &= \frac{C}{2}(1+\cos 2\theta) \\
    k_{xy} &= C\sin 2\theta
\end{align*}
\]

3. Results and discussion

The effect of different thermal exposure temperatures and durations on the bistable behaviour including the load-displacement curves and curvatures of bistable shells are discussed. The microstructure characterization of bistable shell after high-temperature exposure at 200 °C for different durations is also given in this section.

3.1. Effect of Thermal Exposure Temperature

3.1.1. Effect on the Curvature of Bi-stable Shell

The principal curvature radii $R$ ($R=1/C$) and twisting angles $\theta$ of bistable specimens after different exposure temperatures are listed in Table 1. The results show that the overall variation trends of principal curvature radii of two stable states increase steadily until thermal exposure temperature exceeding $T_g$ after which they decrease gradually as the thermal exposure temperature increases, which means the glass transition temperature $T_g$ is a critical influence factor for principal curvature radius. It is also noted that the twisting angles of both stable states increase as the thermal exposure temperature increases. The longitudinal and transverse thermal expansion coefficients $\alpha_{11}$ and $\alpha_{22}$ of the carbon-fiber epoxy resin composite have effects on the deformation of the shells. Generally, $\alpha_{22}$ always keep positive value while $\alpha_{11}$ changed from negative value to positive value when the temperature exceeds the glass transition temperature $T_g$. Moreover, the intrinsic reason is that the microstructure of the shells at the condition of exceeding $T_g$ will change according to its definition—the Glass Transition Temperature ($T_g$) is one of the most important
properties of any epoxy and is the temperature region where the polymer transitions from a hard, glassy material to a soft, rubbery material.

Table 1 Principal curvature radii and twisting angles of bistable specimens under different exposure temperatures.

<table>
<thead>
<tr>
<th>Thermal exposure temperature, $T/°C$</th>
<th>Principal curvature radius of first stable state, $R_1$/mm</th>
<th>Principal curvature radius of second stable state, $R_2$/mm</th>
<th>Twisting angle of first stable state, $\theta_1$/°</th>
<th>Twisting angle of second stable state, $\theta_2$/°</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>25.00</td>
<td>31.11</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>60</td>
<td>26.23</td>
<td>32.21</td>
<td>2.86</td>
<td>3.56</td>
</tr>
<tr>
<td>80</td>
<td>26.84</td>
<td>33.29</td>
<td>3.08</td>
<td>4.70</td>
</tr>
<tr>
<td>120</td>
<td>26.78</td>
<td>32.85</td>
<td>4.06</td>
<td>5.46</td>
</tr>
<tr>
<td>200</td>
<td>26.15</td>
<td>30.71</td>
<td>5.15</td>
<td>7.29</td>
</tr>
</tbody>
</table>

The influence of exposure temperature on the curvatures of bistable specimen is demonstrated in Fig.3. The principal curvature $k_{y1}$ and $k_{x2}$ have similar changing trend. The results show that the overall variation trends of principal curvature radii of two stable states increase steadily until thermal exposure temperature exceeding $T_g$ after which they decrease gradually as the thermal exposure temperature increases. As we can see from Fig.3, $T_g$ is the turning point of those curves, there are different curve gradients at the temperatures less than and beyond $T_g$ point. The twisting curvatures $k_{xy1}$ and $k_{xy2}$ increase with an increase in the thermal exposure temperature, indicating higher residual thermal stresses in bistable shells.

3.1.2. Effect on the Whole Snap Process

As mentioned above, the snap-through and snap-back processes are tested by using the assembled testing machine (Reger 3010), detailed testing method and boundary condition can be found in References [7] and [9]. Load-displacement curves of bistable specimen after different exposure temperatures are given in Fig.4. The exposure duration for all cases is 1 hour. As shown in Fig.4 (a), the curves for different exposure temperatures have similar changing trend. The snap-through load is around 20 N. It is interesting to note that when the thermal exposure temperature exceeds $T_g$, a saw-toothed undulation at the beginning of the loading stage and the test
of snap-back process is accompanied by some small acoustic emission. This might be due to the viscoelastic properties of the bistable structures caused by the higher temperature. Further studies on this phenomenon are still underway.

As shown in Fig. 4 (b), the snap-back process increases steadily with increasing displacement then followed by a steep decrease. The snap-back process has an approximately linear relationship with the displacement before reaching its peak value, but suddenly decreases until zero when the exposure temperature goes below \( T_g \). As the temperature increases, a retention stage is observed when the exposure temperatures are 120 °C and 200 °C, which corresponds to the observation in experiment, as shown in Fig.5. At the beginning stage of the loading, one straight edge gradually becomes a curve edge, while the other almost keeps straight. Subsequently the snap-back process decreases to zero, which indicates the snap-back process is completed.
Fig. 4. Load-displacement curves of bistable specimen after different exposure temperatures.

(a) Snap-through process

(b) Snap-back process

Retention stage
Fig. 5. Retention stage in snap-back process: (a) undeformed, (b) retention stage in front view, (c) left view and (d) right view, (e) second stable shape.

3.2. Effect of Thermal Exposure Duration

The effect of exposure duration on the bistable behaviour of shells is investigated in this section. Snap-through and snap-back processes are tested after the shells being exposed to a temperature of 200 °C for 0 h, 1 h, 3 h and 6 h, respectively.

Principal curvature radii $R$ and twisting angle $\theta$ of bistable specimens with different exposure durations are listed in Table 2. The principal curvature radii of two stable states decrease gradually as the thermal exposure time increases. It should also be noted that the twisting angle of first stable state $\theta_1$ decreases while the twisting angle of second stable state $\theta_2$ increases as the thermal exposure temperature increases. The influence of the exposure duration on the curvatures of bistable specimen is given in Fig. 6. The results show that $k_{y1}$ remains around 40 mm$^{-1}$ and $k_{x2}$ almost keeps at 33 mm$^{-1}$. $k_{xy1}$ decreases as the thermal exposure duration rises whereas $k_{xy2}$ increases with an increase in the duration.
Table 2 Principal curvature radii and twisting angles of bistable specimen at different thermal exposure time.

<table>
<thead>
<tr>
<th>Thermal exposure time, ( t/h )</th>
<th>Principal curvature radius of first stable state, ( R_1/\text{mm} )</th>
<th>Principal curvature radius of second stable state, ( R_2/\text{mm} )</th>
<th>Twisting angle of first stable state, ( \theta_1/\degree )</th>
<th>Twisting angle of second stable state, ( \theta_2/\degree )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25.15</td>
<td>30.46</td>
<td>8.28</td>
<td>11.55</td>
</tr>
<tr>
<td>3</td>
<td>24.79</td>
<td>29.30</td>
<td>7.57</td>
<td>13.05</td>
</tr>
<tr>
<td>6</td>
<td>24.73</td>
<td>28.86</td>
<td>6.34</td>
<td>16.28</td>
</tr>
</tbody>
</table>

Fig. 6. Exposure duration influence on the curvatures of the bistable shell.

Load-displacement curves of bistable shell after different exposure durations are depicted in Fig. 7. The same phenomena like the elevated temperature is found that with increased thermal exposure duration, the saw-toothed undulation and small acoustic emission are observed due to the long term high-temperature exposure. The displacements of snap-through for all cases are around 25 mm. As shown in Fig. 7(b), the snap-back load becomes lower as the thermal exposure time increases. The snap-back load is 10.81 N for 0 h and 9 N for 6 h, respectively. The retention stage in snap-back process test are also observed and highlighted in Fig. 7(b).
Fig. 7. Load-displacement curves of bistable specimen after different exposure time.

(a) Snap-through process

(b) Snap-back process

Retention stage
3.3. Microstructure Characterization

Scanning electron microscope (SEM, type: HITACHI S-4700) is used to observe the surfaces and fiber-matrix interfaces of the bistable shell after thermal exposure of 200 °C for 0 h, 1 h, 3 h and 6 h, respectively. SEM micrographs of the surfaces are given in Fig.8. It can be found that the cylindrical shell surfaces have many irregular solid particles. The resin substrate particles are densely adhered to the surfaces of the cylindrical shell after high-temperature curing, and the particles are relatively large at the scale of 50 µm. As the thermal exposure duration increases, the number of irregular solid particles attached to the shell’s surfaces decreases rapidly because of the degradation of epoxy resin. After 6 hours of thermal exposure, solid particles attached to cylindrical shell surfaces get less and have been decomposed into smaller particles.

![SEM micrographs of shell’s surface after thermal exposure of 200 °C for different time. (a) 1 hour; (b) 3 hour; (c) 6 hour.](image)

SEM micrographs of shell’s fiber-matrix interfaces at the scale of 20 µm after different exposure durations are given in Fig.9, in which the thermal temperature is
Using Jmicro Vision software (Version 1.2.7), the diameters of carbon fibers are measured. Without a thermal exposure, the arranged carbon fibers have a diameter of 6.86 µm. With an increased thermal exposure duration, epoxy resin properties degraded severely due to the significant thermal decomposition of the polymer matrix, which makes the ability that resins protected and unified the fibers become lower. When the exposure duration reaches 6 hours, the number of resins between the fibers decreases significantly, the gap between the fibers can be found and the measured diameter of the carbon fiber is 7.27 µm. By comparing SEM micrographs at different thermal exposure durations, it is observed that the structure change under microscale of the carbon fiber is negligible, whereas the change of the epoxy resin is quite noticeable. It is seen that the thermal exposure has a significant influence on microstructures of the anti-symmetric laminated shells, which further demonstrated the effect of thermal exposure on the bistable behaviour of those shells.

Fig. 9. SEM micrographs of shell’s fiber-matrix interface after thermal exposure of 200 °C for different time. (a) 0 hour; (b) 1 hour; (c) 3 hour; (d) 6 hour.
4. Conclusions

The bistable behaviour of anti-symmetric laminated cylindrical shells after thermal exposure is investigated in this paper. Some experiments have been carried out to determine the effects of thermal temperature and duration on the bistable curvatures and snap loads. The microstructures of bistable shell’s surfaces and fiber-matrix interfaces after high-temperature exposure with different durations have been observed by SEM. Experimental results from macroscale to microscale demonstrates that the thermal exposure temperature and duration have significant effects on the bistable behaviour of anti-symmetric laminated cylindrical shells. The current research is expected to provide very valuable instructions for the engineering application of those bistable shells in thermal environments.

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