A numerical approach to characterize the compression and relaxation behavior of uncured prepreg laminates in the process of hot press-forming

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Abstract

In the pre-forming process of uncured prepreg laminates with hot press, the compaction stress is an important parameter that affects the performance and quality of the final product. Therefore, it is necessary to explore the compaction response of the prepreg during manufacturing processes. In this study, the compression and relaxation viscoelastic behaviors of uncured prepreg laminates were investigated in different temperatures, displacements, velocities, and thicknesses. The results show that the power law model and the fractional viscoelastic model could accurately describe the compression and relaxation behaviors, respectively. The theoretical fitting curves match very well with experimental data at each condition and the R-squared value is greater than 0.95. The effect mechanism of compression and relaxation viscoelastic behaviors is analyzed and the obtained results could provide a reference for the process of hot press-forming and numerical simulation.

1. Introduction

Thermoset prepreg materials exhibit excellent specific strength and stiffness in the aerospace industry especially in manufacturing of commercial airliners [1]. Compared with spacecraft and military aircraft, commercial airliners have stricter requirements for manufacturing costs. Traditional methods of manufacturing composite parts such as manual layup are being phased out in the field of commercial aviation due to their low efficiency, high cost, and unstable quality. Some innovative technologies like automated layup and pre-forming methods are being used in manufacturing processes. The use of automated layup method is usually limited to produce parts with low to medium curvature [2]. The pre-forming method is widely used in the manufacturing process of large components [3]. The pre-forming of uncured prepreg laminates can be implemented in hot press-forming or hot diaphragm forming. The hot press-forming method has attracted the attention of aircraft manufacturers such as Boeing and Airbus, particularly in the manufacturing of composite parts with complex structures, such as stringers with omega-shaped cross-sections or curved profiles [4—6]. The typical process of hot press-forming is illustrated in figure 1. Firstly, the laminate is assembled by manual layup or automated equipment according to the predetermined ply design. Then, similar to the process of metal stamping, the prepreg laminate is used to convert the flat or curved board into specific shapes. Finally, the processed preform is solidified in the autoclave to form the final product [1].

In the process of hot press-forming, the uncured prepreg laminate is deformed by the complicated force. Defects are commonly observed in the product of hot press-forming such as wrinkles, high porosity, uneven distribution of resin, and fabric bridging. Defects are serious problems in the hot press-forming process, especially for products with complex configurations. Mukhopadhyay et al found that wrinkle significantly
reduces the compressive strength of the sample [7]. Baran et al reported that fiber misalignments severely reduce the stiffness of the laminate [8]. Hörmann et al studied the impact of defects on the service life of composite products. Their results indicate that fatigue life is reduced to approximately 50% compared to the non-defect material due to defect [9].

In recent years, many researchers have been investigating the mechanism of defect formation within uncured prepreg laminates. Erland et al have focused on the impact of shear between prepreg layers on defects [10]. They showed that the defects were caused by the hindrance of shear between the plies. The inter-ply slipping properties of prepreg laminates are affected by temperature, rate, and pressure. Farand et al analyzed the mechanism of defect formation such as wrinkle and fiber misalignment [11]. They observed that the reduction in friction of inter-ply can reduce defects during the process of drape forming. Many researchers have investigated inter-ply slipping behavior of prepreg laminates by the Stribeck Curve. On the Stribeck Curve, the coefficient of friction has a linear relationship with the Hersey number in the prepreg molding process and the Hersey number is associated with the normal stress. Peng et al proposed a three-stage model to investigate the slipping behavior of the inter-ply of prepreg laminate, which considers the effects of temperature, normal pressure and pulling rate [3]. Among these factors, the pressure effectively both drives and restricts the slippage of prepreg laminates. The slippage of the prepreg is closely related to the origination of defects. In order to avoid defects, it is necessary to explore the optimal process parameters in the hot press-forming process. Compared to the time and cost expensive ‘trial-by-error’ approaches, the numerical simulation facilitates the determination of suitable process parameters at low cost for the process of hot press-forming. The quality of the product is profoundly affected by the pressure and its distribution. Therefore, exploring the pressure response behavior of prepreg can provide the reference for numerical simulation.

The compaction stress of prepreg in the process of hot press-forming is a complex behavior, which shows obvious viscoelastic characteristics, especially stress relaxation phenomenon. This viscoelastic behavior of the prepreg also affects the quality of product [11–13]. One of the widely used models to describe the viscoelastic behavior of materials is the Prony series. The model has been used to describe the viscoelastic behavior of materials under complex conditions like different temperatures, the hygrothermal environment and the curing process [14–17]. However, in order to accurately describe viscoelastic behavior, a large number of parameters are introduced into Prony series. In general, describing stress relaxation behavior requires at least a third-order Prony series, which consists of six different parameters like stiffness and relaxation time coefficient. There is uncertainty in the determination of the order of the Prony series, which is usually determined based on trial and error for a given material [18]. This makes it difficult to understand the physical meaning of Prony series. Besides, parameters of Prony series show different values in different mechanical tests [19]. To overcome the limitations of traditional methods, the fractional viscoelastic model can be applied for predicting the viscoelastic properties like relaxation and creep. The fractional viscoelastic model extends integration and differentiation operators to non-integer order, which can accurately reflect the experimental results with fewer parameters. Reza et al have used fractional to describe the viscoelastic behavior of materials, which included fewer
parameters to obtain more accurate results than the Prony series \[19\]. Paola et al have used Caputo fractional-derivative viscoelastic models to describe the viscoelastic behavior of polymers \[20\]. At the same time, many researchers have used the finite element method based on fractional constitutive model to simulate the viscoelastic behavior of materials \[21\].

In this paper, the compression behaviors of uncured prepreg laminates were investigated during the hot press-forming process. The compression and relaxation performance of uncured prepreg laminates in different temperatures, displacements, velocities, and thicknesses were studied as well. Then, the paper employed the power law model and the fractional viscoelastic model to describe the compression and relaxation behavior of uncured prepreg laminates. Finally, the effects of velocities and thicknesses on the compression and relaxation behavior were explored.

2. Experimental

The material selected in our work was the CYCOM® 970/T300 PW fabric prepreg. The resin content of the prepreg is 40% and the fiber areal weights are 193 gsm. The layer thickness of the prepreg is 0.21 mm.

The torsional rheometer (HR-2, TA Instruments) was used to measure the viscosity properties and time/temperature-dependent cure of the prepreg (figure 2(A)) \[22\]. The prepreg was cut into a circle with a diameter of 25 mm. Then processed prepreg was stacked into a wafer of five plies and adhered to the bottom fixture of the rheometer with cyanoacrylate. The linear viscoelastic region (LVE) of prepreg was measured by dynamic oscillatory test (strain sweep) with strain from 0.001% to 100% at a frequency of 1 Hz and a normal force of 5 N at ambient temperature to obtain the strain rate in the subsequent experiment. The viscosity-temperature test of prepreg was conducted at a singular frequency (1 Hz), strain rate (0.01%), and normal force of 5 N at a heating rate of 1 °C min\(^{-1}\). The viscosity-time test was performed at 1 Hz, 0.01 strain%, and a normal force of 5 N at 40 °C, 80 °C, and 120 °C, respectively.

The samples were 150 mm long and 100 mm wide. Samples were pressed under vacuum (Pressure > 75 kPa, 1 h, 23 °C) before the test. The length, width, thickness, and quality of samples were measured separately before and after the experiment. As shown in figure 2(B), the testing device was similar to that used by Hubert and installed on a tensile testing machine (ETM204C, Wance Testing Machine CO. LTD) \[23\]. The device was placed in an environmental chamber and two thermocouples were installed on the upper and lower plates respectively to monitor the temperature of the device. The sample was pre-heated for 10 min before each of the experiments.
to achieve a uniform temperature. Before the experiment, the tapered ruler was used to measure the gaps at the four vertices between the upper and lower plates. And the device was adjusted to maintain consistent upper-lower gaps and ensure the parallelism, to ensure a uniform compaction stress force applied to the sample.

For the relaxation experiment time, we have investigated the relaxation behavior of prepregs in 6000 s. The results related to the relaxation behavior of prepreg are depicted in figure S1 and table S1 (available online at stacks.iop.org/MRX/9/055102/mmedia). As can be seen from the figure S1, the decrease ratio of stress first increases rapidly and then levels off. Different from some polymer materials [24, 25], a significant drop was shown in prepreg stress within 3600 s and the decrease ratio was 54.12% (table R1). After 3600 s, the magnitude of the stress drops of the prepreg diminished. The decrease ratio was 55.38% at 6000 s, which had only increased by 1.26% compared to 3600 s. And the hot press-forming process generally does not exceed one hour in the process of production. Therefore, the relaxation experiment time was selected as 3600 s. Samples were tested at different temperatures, velocities, thicknesses, and compression distances. The experiment was repeated three times under the same conditions and the average was taken.

3. Model approach

As mentioned above, the experiment can be divided into two stages (figure 3): compression ($0 \leq t < t_0$) and relaxation ($t > t_0$), and these two stages are explored separately.

In the stage of compression, the behaviors of prepreg can be divided into three regimes: an initial linear regime, a final linear regime, and a nonlinear regime in between [26, 27]. In the first two regimes, the large voids are been filled because of the flow of resin and the deform of bundles in prepreg. In the final linear regime, the resin and fiber in prepreg are subject to large deformation, which leads to a rapid rise in stress. The material’s strain hardening behavior can be described by a simple power law as follows:

$$\sigma = \frac{A}{1000} \left( \frac{v t}{h_0} \right)^B$$

where $\sigma$ is the stress of prepreg (kPa), $A$ is the strain hardening coefficient and $B$ is the stiffening factor. $v$ is the compression velocity. $h_0$ is the average thickness of the sample.

The relaxation behavior of prepreg laminates can be described using the fractional viscoelastic model. This model can be expressed as [28]:

$$\sigma(t) = c_0 \frac{d^\beta \varepsilon(t)}{dt^\beta}$$

where $c_0$ is a parameters depending of the material, and $\beta$ is the order of the fractional derivative. The fractional viscoelastic model behaves as a linear elastic model when $\beta = 0$, and as an ideal viscosity model when $\beta = 1$ figure 4.
The $\frac{d^\beta \varepsilon(t)}{dt^\beta}$ is defined by the Caputo fractional derivative, we can obtain:

$$\sigma(t) = \varepsilon_\beta \frac{1}{\Gamma(1 - \beta)} \int_0^t \frac{\varepsilon'(\tau)}{(t - \tau)^\beta} d\tau$$

(3)

where $\Gamma(\cdot)$ is the Euler gamma function. In fact, the fractional viscoelastic model shows long-term memory and affected by the initial loading history [29]. Paola et al had shown that the best fitting performed by taking or not into account the loading history gives very different parameters [20]. Due to the distance of compaction is uniform loading, the real deformation history during the test can be divided into two parts (figure 3):

$$\varepsilon(t) = \begin{cases} 
0 & t = 0 \\
\frac{t}{t_0} & 0 < t < t_0 \\
\varepsilon_0 & t > t_0 
\end{cases}$$

(4)

In the stage of relaxation, substituting equation (4) into equation (3) and rewrite the unit of stress as kPa, we can obtain:

$$\sigma(t) = \frac{\varepsilon_3 \varepsilon_0}{1000\Gamma(2 - \beta) t_0} [t^{1 - \beta} - (t - t_0)^{1 - \beta}]$$

(5)

4. Results and discussion

As shown in figure 5(A), the prepreg viscosity shows a slowly rising trend with the temperature increasing from 40 °C to 200 °C. As shown in figure 5(A), the viscosity of prepreg decreases slowly at 40 °C–80 °C. When the temperature reaches from 130 °C to 160 °C, the viscosity drops rapidly as the temperature increases. When the
The viscosity of the prepreg rises rapidly due to the curing of the resin. High temperature produces a faster curing reaction speed and shorter operational time. In order to prevent the influence of curing behavior at high temperature, 120°C is chosen as the maximum temperature in the further experiment. The compression and relaxation behaviors of uncured prepreg laminates is be investigated at 40°C, 80°C, and 120°C, respectively. The relationship between viscosity versus time of the prepreg in different experimental temperatures were studied. Within one hour, the viscosity of the prepreg remains unchanged at 40°C and 80°C. Figure 5(B) shows the viscosity versus time curve of the prepreg at 120°C. It can be found that the viscosity of the prepreg has shown a rising trend above the 1310 s mark because of the curing reaction of resin.

Figure 6(A) shows the compressed response of the uncured prepreg laminates at different temperatures. It can be found that the prepreg exhibits similar compression behavior at 80°C and 120°C due to similar viscosities. The compressed behavior of the prepreg at 40°C shows a steep curve compared to other experimental conditions. The molecule chain of resin in prepreg become more inactive at the lower temperature, which leads to the higher maximum stress in the stage of compaction [30, 31]. The results of the relaxation stage are shown in figure 6(B). In the early stage of stress relaxation, the stress of prepreg decreases rapidly at different temperatures, which shows similar stress relaxation behavior. However, the stress of the prepreg rises after 1000 s due to the curing reaction. This is also confirmed by the relationship of time versus viscosity. In general, the viscosity of prepreg should be as small as possible during the process of hot press-forming to ensure minimal resin resistance. In the process of hot press-forming, the curing of prepreg should be avoided as far as possible. Because the curing behavior impedes interlayer sliding, which leads to defects like winkle. In order to prevent the curing reaction of the prepreg, the 80°C was been chosen as the experimental temperature in the subsequent study.

Figure 6. (A) Compression curves of samples at different temperature, with a velocity of 1 mm min⁻¹, a compression distance of 0.4 mm and ten layers; (B) Relaxation curves of samples at different temperature. The shaded region is standard error.

Figure 7. (A) Compression curves of samples at different compression distances, with a velocity of 1 mm min⁻¹, a temperature of 80°C and ten layers; (B) Relaxation curves of samples at different compression distances. The shaded region is standard error.
The experimental results of the uncured prepreg laminates under different compression distances are shown in figure 7. The prepreg laminates under different compression distances show similar compression properties. However, the prepreg laminates exhibit different stress relaxation behaviors under different compression distances. The quality of prepreg laminates remains unchanged after compression, the resin in the prepreg is not extruded during the compression stage, and therefore exhibit a similar compression behavior. The prepregs under different compression distances show different relaxation properties, which could be attributed to the different states of the prepregs.

Figure 8 shows the stress responses over time of compression and relaxation experiments at different compression velocities. Evidently, the stress of prepreg laminates increases rapidly with time. It can be seen from figure 8(A) that the stress of the prepreg laminates under the same strain increases with increasing the compression velocity. The inspection of the compression process indicates that the prepreg has a significant velocity-dependent compression property. It is due to the viscoelastic behavior of the resin and fiber bed in the prepreg laminates, which include the entanglement of resin’s molecular chains and the deformation and flow resistance of the fiber bed [32, 33]. The results related to the relaxation behavior of prepreg are depicted in figure 8(B). As the relaxation time increases, the stress of the prepreg laminates decreases rapidly at first and subsequently the rate of decrease tends to slow. It could be found that the stress relaxation of the prepreg...
laminates within the first 500 s accounts for 70%–90% of the total relaxation stage of the prepreg (3600 s). This means that the stress relaxation behavior of the prepreg laminates could not be ignored in the manufacturing process of composite materials.

As can be seen in Figure 9, the curves of the theoretical fitting match well with the experiment results. The R-squared value of each condition is larger than 0.99 and increases with increasing velocity. It shows the power law fitting for the experimentally measured stress in the relaxation stage for different velocities. (A) 0.1 mm min⁻¹; (B) 0.5 mm min⁻¹; (C) 1 mm min⁻¹; (D) 5 mm min⁻¹; (E) 10 mm min⁻¹.

Figure 10. The theoretical fitting for the experimentally measured stress in the relaxation stage for different velocities. (A) 0.1 mm min⁻¹; (B) 0.5 mm min⁻¹; (C) 1 mm min⁻¹; (D) 5 mm min⁻¹; (E) 10 mm min⁻¹.

Figure 11. (A) Compression curves of samples for the different number of prepreg layers, with a compression distance of 0.4 mm, a temperature of 80 °C and a velocity of 1 mm min⁻¹; (B) Relaxation curves of samples for the different number of prepreg layers. The shaded region is standard error.

Table 1. The parameters of power law model for different velocities, with a compression distance of 0.4 mm, a temperature of 80 °C and ten layers.

| Velocity/mm-min⁻¹ | 0.1 | 0.5 | 1   | 5   | 10  |
|-------------------|-----|-----|-----|-----|-----|
| h₀/mm             | 2.41| 2.33| 2.42| 2.33| 2.31|
| A                 | 406606 | 948801 | 1428889 | 1615884 | 2024976 |
| B                 | 2.234 | 2.562 | 2.452 | 2.500 | 2.514 |

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Figure 12. The theoretical fitting for the experimentally measured stress in the compression stage for the different number of prepreg layers. (A) Five layers; (B) Seven layers; (C) Ten layers; (D) Twelve layers; (E) Fifteen layers.

Figure 13. The theoretical fitting for the experimentally measured stress in the relaxation stage for the different number of prepreg layers. (A) Five layers; (B) Seven layers; (C) Ten layers; (D) Twelve layers; (E) Fifteen layers.

Table 2. The parameters of fractional viscoelastic model for different velocities, with a compression distance of 0.4 mm, a temperature of 80°C and ten layers.

| Velocity/mm-min⁻¹ | 0.1 | 0.5 | 1 | 5 | 10 |
|-------------------|-----|-----|---|---|----|
| $c_0/\text{Pa} \cdot \text{s}^{-\beta}$ | 74233.95 | 87715.19 | 118429.87 | 120826.21 | 134492.61 |
| $\beta$ | 0.12 | 0.12 | 0.11 | 0.11 | 0.10 |
| $t_0/\text{s}$ | 240.00 | 47.93 | 24.00 | 4.76 | 2.40 |
| $\varepsilon_0$ | 0.167 | 0.172 | 0.165 | 0.172 | 0.175 |
| $R^2$ | 0.979 | 0.970 | 0.995 | 0.956 | 0.960 |
law model can effectively describe the behaviors of prepreg laminates in the compression stage. There is a certain deviation between the fitted curve and the experimental results in the low-velocity starting area. At the beginning of compression, the slipping behaviors of bundles and fibers within the bundle occur inside the prepreg laminates. This behavior creates additional friction and causes some errors in fitting results. The parameter of the power law model is listed in table 1. It can be observed that the stiffening factor B is similar in each velocity condition. This shows that the increased tendency of stress is similar at different experimental velocities. The strain hardening coefficient A increases with the increase in velocity. This indicates that the magnitude of the stress increase is proportional to the velocity of compression. The explanation is that the fibers in the prepreg laminates have less time to rearrange and relax at higher velocities, resulting in more load to compress [34].

Figure 10 presents the stress versus time data in the stage of relaxation with different velocities. It can be seen that the fractional viscoelastic model is consistent with the experimental results, and the R-squared value is more than 0.95. Table 2 display the parameters of the fractional viscoelastic model for the different tests. The unit of parameter $c_0$ is $Pa \cdot s^{-\beta}$ due to the consistency of dimensional. Some researchers believe that this parameter is the product of stiffness values ($E/Pa$) and relaxation time ($\tau/s$) like same parameters in Prony series. But it is difficult to design experiments to separately measure the parameters $E$ and $\tau$. Usually, the parameter $c_0$ is interpreted as the ‘firmness’ of a material [20]. $\beta$ is the order of the fractional derivative, which is considered to be a parameter characterizing the viscosity and elasticity of materials. It can be observed that the parameters $c_0$ and $\beta$ do not change as the number of prepreg layers increases at a near-linear trend.

As can be seen in figure 13, the fitting curves of the fractional viscoelastic model match well with the experimental data and the R-squared value is greater than 0.95. Table 4 present the parameter of the fractional viscoelastic model for samples with different number of prepreg layers. The values of $\beta$ in table 4 do not change significantly with the increase of prepreg thickness, which fluctuates from 0.09 to 0.12. The values of $\beta$ are considered to be a parameter characterizing the viscosity and elasticity of materials. When the temperature remains constant, the viscoelasticity of the material also remains the same. Therefore, the value of $\beta$ does not change significantly with the increase of prepreg thickness, which fluctuates from 0.09 to 0.12. The values of $\beta$ are considered to be a parameter characterizing the viscosity and elasticity of materials. When the temperature remains constant, the viscoelasticity of the material also remains the same.

### Table 3. The parameters of power law model for different number of prepreg layers, with a compression distance of 0.4 mm, a temperature of 80°C and a velocity of 1 mm min$^{-1}$.

| Number of layers | 5    | 7    | 10   | 12   | 15   |
|------------------|------|------|------|------|------|
| $h_0$/mm         | 1.31 | 1.78 | 2.42 | 2.92 | 3.50 |
| A                | 3710737 | 3133119 | 1428589 | 352769 | 111309 |
| B                | 3.72 | 3.15 | 2.452 | 1.74 | 1.19 |

### Table 4. The parameters of fractional viscoelastic model for different number of layers, with a compression distance of 0.4 mm, a temperature of 80°C and a velocity of 1 mm min$^{-1}$.

| Number of layers | 5    | 7    | 10   | 12   | 15   |
|------------------|------|------|------|------|------|
| $c_0/PA \cdot s^{-\beta}$ | 179103.05 | 143752.45 | 118429.87 | 110406.70 | 1033842.48 |
| $\beta$          | 0.09 | 0.09 | 0.11 | 0.12 | 0.10 |
| $t_0/s$          | 23.93 | 24.00 | 23.93 | 23.93 | 24.00 |
| $\varepsilon_0$ | 0.305 | 0.225 | 0.165 | 0.137 | 0.114 |
| $R^2$            | 0.978 | 0.974 | 0.995 | 0.973 | 0.969 |
change much either. According to the fitting parameters of the model, the parameter $c_3$ decrease when increasing the thickness of the sample. This result indicates that the energy released in the relaxation process of the compression stage is significantly different for different thickness samples under the same compression distance. There are two reasons for this: first, the samples of different thicknesses have different strains; more energy is stored in the sample with larger strain during compression, which in turn releases more energy in the relaxation stage. Secondly, the increase in the number of prepreg layers introduces more interfaces, which increases the number of pores in the prepreg laminates. This also makes thick samples store less energy during the stage of compression. Therefore, this causes the parameters in the fractional viscoelastic model to decrease as the thickness increases.

5. Conclusions

In this work, the pre-forming experiments of the CYCOM® 970/T300 uncured prepreg laminates in different temperatures, displacements, velocities, and thicknesses were systematically carried out to explore the behavior of compression and relaxation in the process of hot press-forming. The viscoelastic behavior of the uncured prepreg laminates in the stage of compression and relaxation using the power law model and fractional viscoelastic model was described. The theoretical fitting curves matched well with the experimental data and the R-squared value is greater than 0.95. The trend of compression performance at different velocities was similar and the magnitude of the stress increase is proportional to the velocity of compression. Additionally, the degree of linearity of compressive stress was proportional to the thickness of the sample under the same compression distance. For the stage of relaxation, the parameter of fractional viscoelastic model increased on the strain velocity. And the same parameter decreased as increasing the thickness of the sample under the same compression distance because of strain and porosity.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflict of interest

The authors declared that they have no conflicts of interest to this work.

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