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Life Prediction of Carbon Fiber Reinforced Polymers using Time Temperature Shift Factor

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ABSTRACT

The properties of Carbon Fiber–Reinforced Polymers (CFRP) are greatly affected under extreme environmental conditions. This paper reports an experimental study to determine the response of IM7-carbon/977-2 cycom epoxy laminates under different humidity and temprature conditions. Short-term 3-point bending creep tests using Dynamic Mechanical Analysis (DMA) were used to test the dry and saturated samples at various temperature levels. The dry coupons were tested at the room temperature (RT) and at 60-120 °C with 20 °C increment and then at 130 °C, 150-180 °C with 10 °C increment for each next test. The saturated (wet) coupons were tested at RT, 40 - 120 °C with 10 °C increment in temperature for each next test and at 145 °C, 150 °C, and 160 °C. The time-temperature shift factor (TTSF) was applied and it is shown that the viscoelastic behavior of the invetigated IM7-carbon/977-2 epoxy laminates, is accurately predicted through the use of TTSF. It has also been shown that determining the viscoelastic behavior at elevated temperatures helps to predict temperature below the glass transition temperature using TTSF. The long-term life of the material is relatively easily predicted using TTSF by conducting traditional short-term laboratory tests.

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1. INTRODUCTION

Fiber Reinforced Polymers (FRPs) are light weight and possess high specific strength and stiffness. Therefore, their usage is continuously increasing in many primary and secondary load carrying structural applications such as aerospace, missiles, aircrafts, automobiles, etc. However, the properties of these materials are greatly affected when exposed to high temperature and moisture. Therefore, it is crucial to understand the behavior of these materials for their widespread usage. Hence, there is a need to develop such testing procedures and protocols to be used to evaluate life-times properties of materials in extreme service conditions. Therefore, many researchers have attempted to characterize FRPs under different testing conditions [1]. FRPs have long expected life in many applications. Therefore, it can be expected that the properties of these material in service may change over the long period of time. For example, it is expected that FRPs used in a highspeed commercial aircraft may last for 20 years with 2500 days (6.85 years) of the time at elevated temperature [2]. With services, lifetime measured for such a long time, it is not possible to conduct real time experimental tests under different testing conditions. Even if it becomes possible to do testing for this long period, considering advances in technology, this will be very impractical as the material under consideration for testing for couple of decades might have been totally changed that one might plan to initiate testing today. Due to these reasons, accelerated testing methodologies are getting more and more attention [2].

Stress-Number of cycle to failure (S-N) curve is very common among the researchers community for fatigue life prediction. In simple words, the curve assumes that fatigue life of the material is dependent on number of cycles. Simply increasing the loading frequency in the fatigue testing, the testing can be accelerated. A

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reduction in testing time by a factor of 120 through this has been reported in the literature [2]. However, there is limitation in this methodology that generally it does not consider the viscoelasticity or oxidation phenomenon in the fatigue response. Unlike metals, the polymers have strong viscoelastic behavior. Therefore S-N curve is not very helpful in accurately predicting the fatigue life of FRPs. As there is great dependency of viscoelastic materials on time, hence changing other parameters will not help either. As the accelerated testing of viscoelastic deformation is well known, therefore Time-Temperature Shift Factor (TTSF) is used for such materials [3]. The procedure of this principle is very simple. Time is effectively accelerated by elevating the temperature. In this way, this principle overcomes the difficulty faced by many accelerating methods to find the link between the accelerated test with the real-life operation.

The working function of TTSF is that measurements of material compliance are taken, at several different temperatures for a small time period. These values are then plotted against the log of time. TTSF is utilized to shift the values for compliance measurements on the log time scale. Shifting the values on the log time scale generates a curve which is named as master curve. The compliance of the material for any time range can then be predicted using the master curve. The acceleration in the testing due to elevated temperatures is established by compliance values overlapping curves at different temperatures [2]. To draw the master curve using TTSF, one curve is taken as reference and hence does not move along the log time scale. Whereas rests of the curves obtained during specific testing regime are shifted to match with the fixed curve. Therefore, it should be kept in mind that master curve predicts the time-temperature deformation for the temperature associated with the first curve. By noting the magnitude of the shifts required to form the master curve, a relation between the horizontal/vertical shifts and temperature/modulus is developed. Therefore, the entire master curve is moved to other temperatures and moduli to predict the life of the viscoelastic materials. This curve is also useful to predict creep compliance by shifting it to appropriate temperatures. Although the application of TTSF was initially for non-destructive characterization of material properties over the time, it has now been extended to characterize the polymeric matrix composites deformation properties [4-8]. Models have been developed to predict the shift factor with change in temperature.

Miyano et al. [9] measured the effect of different constant strain-rates and temperatures on the compressive strength of resin impregnated carbon fibre strands. Using the procedure for the tensile strength reported in literature [10], they obtained master curve for compressive strength. They predicted the tensile and compressive strengths of unidirectional Carbon Fiber–Reinforced Polymers (CFRP) materials along the longitudinal direction under constant strain-stress loadings by making use of Rosen's strength formula [11-12] for elastic matrix composites.

Despite the many attempts, the durability of materials is generally not understood comprehensively under the exact nature of environmental attacks such as moisture and higher temperature. It is well understood now that higher temperature not only causes degradation but also changes significantly the performance and failure mechanisms of CFRP [13-15].

The aim of this study is therefore to conduct a comprehensive experimental study to consider the effect of different significant parameters such as moisture uptake and high temperature to predict the life performance of aerospace graded IM7-carbon/977-2 epoxy laminates using Time Temperature Shift Factor (TTSF).

Following the introduction section, the structure of the manuscript is designed as follows: In section 2, the theoretical background of TTSF is detailed along with supporting mathematical expressions. In section 3, sample preparation and procedure are detailed. Results and discussions are presented in section 4 while in section 5 concluding remarks are highlighted, followed by references.

2. THEORATICAL BACKGROUND

Viscoelastic materials deform slowly when exposed to an external force but return to their original configuration when the external force is removed. Wiechert model is very helpful to understand the overall response of such materials with viscoelastic properties. The model helps to grasp the phenomenon of distribution of relaxation time. The model is used to calculate the relaxation modulus as given below:

$$C(\mathrm{t}) = E_e + \sum_i E_i \, exp\left(\frac{-t}{\tau_i}\right)$$

where the notations used have their usual meanings. i.e C(t), Ee, Ei and τi are the viscoelastic relaxation modulus, the equilibrium modulus, elastic modulus and relaxation time, respectively.

The expression can then be used to derive expression for E' and E''.

$$E' = E_e + \sum_i E_i \frac{\omega^2 \tau_i^2}{1+\omega^2 \tau_i^2}$$

$$E'' = \sum_i E_i \frac{\omega \tau_i}{1+\omega^2 \tau_i^2}$$

where E', E'' and ω are the storage, loss modulus and applied circular frequency, respectively. Moduli are derived in terms of the relaxation distribution.

It is worth nothing from expression for E'' that when the applied circular frequency is infinitely small then in turn E''will be very small. Hence the loss modulus is neglected mostly. Therefore, in this case, resulting stress and strain have no lag phase. If the case remains valid under negligible or very small frequency, then it can be

assumed that E', $\approx$ C (t). If the expression remains valid then Miyano et al. [13] verified that the relaxation modulus can be used to obtain creep compliance S(t). It must however be noted that the creep response takes longer to get to its equilibrium than relaxation response. It is worth noting that increasing the temperature reduces the viscoelastic relaxation time for viscoelastic materials. Therefore, increase in temperature accelerates the process effectively for such materials. For thermorheologically simple materials, the relaxation modes of such materials can be calculated in terms of temperature by defining time-temperature stated below:

$$\tau_i(T) = a_T \tau_i(T_0)$$

where T_0 and a_T are reference temperature and temperature-dependent horizontal shift factor, respectively. Therefore, the viscoelastic behavior of the material at T_0 is helpful to calculate the storage modulus at temperature T as:

$$E'(\omega, T) = E_e(T_0) + \sum_i E_i(T_0) \frac{(a_T\omega)^2 \tau_i^2(T_0)}{1+(a_T\omega)^2 \tau_i^2(T_0)}$$

In the storage modulus equation, frequency ω is replaced with time as ω=1/t. Therefore, time-temperature superimposed master curve is obtained by shifting the response curves at different temperatures along the logarithmic time axis as shown in Figure 1 [15].

Scaling factors are temperature dependent; in logarithmic axis, time scaling represents horizontal shift, a_{T0} while modulus scaling represents a vertical shift, b_{T0}.

$$\log a_{T0}(T) = \log t - \log t'$$

$$\log b_{T0}(T) = \log D_c(t, T) - \log D_c(t', T_0)$$

where Dc is the creep compliance measured from the deflection of the specimen's center.

Hence, the life of composite materials is relatively easily estimated through the application of TTSF by conducting traditional short-term laboratory tests.

3. EXPERIMENTAL METHODLOGY

3. 1. Material and Sample Preparation IM7-carbon/977-2 epoxy Composite system was selected for the current research work. 977-2 is an epoxy blended with a thermoplastic polymer for toughening purposes. This material is extensively employed in primary aeronautical structures, such as fuselages and wings.

Unidirectional IM7-carbon/977-2 epoxy prepreg of 450 mm length and 200 mm width were cut and then five layers of the prepreg were laid down manually. To avoid the air entrap between the plies, vacuum was applied after every layer. After laying down the plies, the laminate was then vacuum bagged. The manufacturer recommended curing data cycle was strictly followed for curing the material in autoclave. After autoclave curing, laminates were cut in desired rectangular strips of 50 x 15 x 1 as shown in Figure 2, using diamond saw cutter. The

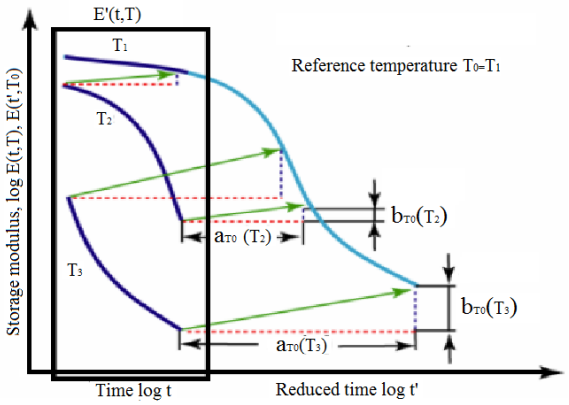

Figure 1. Shifting of storage modulus using TTSF

samples were divided into two (minimum 40 test samples in each half). Half of the coupons were stored in desiccator until just before the time of testing. The remaining half of the coupons were used for moisture uptake. Water bath was used for this purpose. The water bath temperature was kept at 25 ˚C. As it was noted that moisture uptake rate was very slow hence the temperature was increased to 80 ˚C. This accelerated the moisture uptake as expected. The moisture uptake in samples in the water bath were monitored regularly and were kept in the bath until all the samples had moisture content of about 1%.

3. 2. Dynamic Mechanical Analysis (DMA) For all the tests reported in this manuscript, Dynamic Mechanical Analysis (DMA) was applied using 3-point bending to investigate the viscoelastic effect on CFRPs samples. The testing reporting in this manuscript was performed according to ASTM standard. DMA is used to deform the coupon mechanically and then the sample response is measured. DMA facilitates to monitor the deformation response of the sample using time or temperature as a function.

Dimensions of the dry and saturated coupons were kept same. The length, width and thickness for the tested samples were kept as 50, 15 and 1 mm respectively. The schematic of the test samples and the test setup, showing

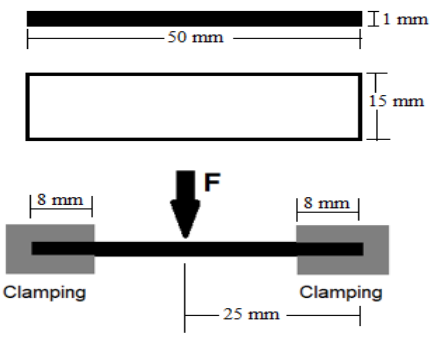

Figure 2. Schematic of the testing and set-up

the width, length and thickness dimensions is shown in Figure 2. The testing time for dry as well as saturated coupons were chosen to be 2000 seconds (33.33 minutes). However, to estimate the full trend of the samples under creep, it was decided to run the dry sample test at 130 °C for 20000 seconds (5.5 hours). It was noted that for the sample, load started to drop below zero after just running for 3836 seconds (63.9 minutes) as is obvious from Figure 3.

Dry coupons were tested at RT, and at 60 - 120 °C with 20 °C increment in temperature and then at 130°C, 150°C - 180 °C with 10 °C increment in temperature as plotted in Figure 3. Saturated (wet) coupons were tested at RTRT, 40 - 120 °C with 10 °C increment in temperature for each next test and at 145 °C, 150 °C, and 160 °C as plotted in Figure 4. It is clear that the wet/saturated samples have shown lower storage modulus compared to storage modulus for the dry coupons for almost all of the temperatures tested except for 60 °C but even in this particular test there is a slight difference between the storage moduli of the coupons.

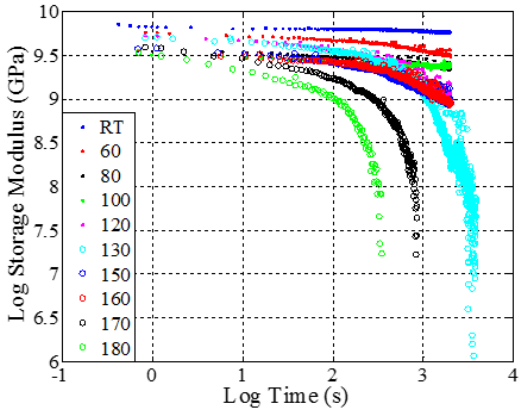

Figure 3. Storage modulus from relaxation tests for different temperature ranges for the dry CFRP coupons

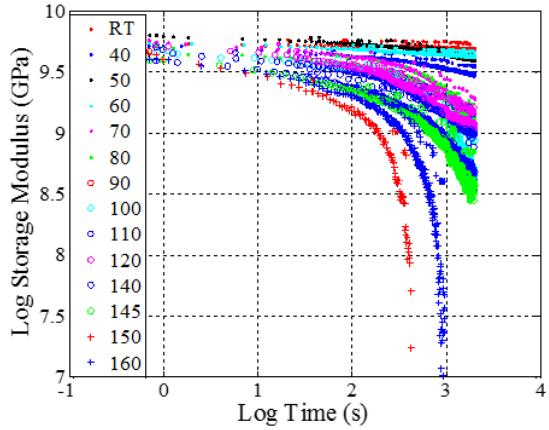

Figure 4. Storage modulus from relaxation tests for different temperature ranges for the wet CFRP coupons

4. TEST RESULTS AND DISCUSSIONS

Following major steps were applied to obtain the master curve for the dry and wet coupon tests performed under 3-point bending test using DMA.

1. Perform creep relaxation tests in a prescribed temperature range

2. Fix a reference temperature and obtain the constants for the reference temperature test(s) via a nonlinear regression on

$$Log[E_s(t,T_{RT})] = \beta_1^{RT}\left[\frac{1}{2} - \frac{1}{\pi}\tan^{-1}[\beta_2^{RT}(Logt - \beta_3^{RT})]\right]$$

where Es (t,T_{RT}) is the room temperature (RT) storage modulus after time t and β_i ^{RT} are coefficients that are estimated from a least square regression performed on the experimental data; T_{RT} is the reference temperature. The resulting fit for dry and saturated coupons is also shown in Figures 5 and 6.

3. Estimate the horizontal and vertical shift factors at the various temperatures via minimising the quadratic error w.r.t. to the "shifted" equation

$$Log[E_s(t,T)] = Log[b_{RT}(T,H)] + \beta_1^{RT}\left[\frac{1}{2} - \frac{1}{\pi}\tan^{-1}[\beta_2^{RT}(Logt - Log[a_{RT}(T,H)] - \beta_3^{RT})]\right]$$

The time-temperature shift factor implies that there exists a rescaled time t', also known as "reduced" time, given by

$$t' = \frac{t}{a_{RT}(T)}$$

where $a_{RT}(T)$ is the "horizontal" shift corresponding to the temperature T. The storage modulus at time t' and reference temperature T_{RT} is related as in the equation

$$E_s(t,T) = b_{RT}(T)E_s(t',T_{RT})$$

where $b_{RT}(T)$ is the "vertical" shift factor. Hence, the storage modulus is easily found at any time and temperature from a reference curve, if the corresponding shift factors are known. Horizental and verticle shift factors are determined via the regression. The verticle shift value was close to unity. Therefore, the application of TTSF to Cycom 977-2 does not require the introduction of a vertical shift, since the regression yields $b_{RT}(T)$ values very close to unity for the whole temperature range considered. However, this was not the case with horizental shift factor, $a_{RT}(T)$ as the values for $a_{RT}(T)$ varied significantly. Therefore, Arrhenius relation was applied with two different activation energy levels.

4. Verify that the "shifted" data collapse on the master curve

The "wet" coupons considered in this work had moisture content of about 1%. Using the data plotted in Figure 4 for wet coupons, the regression yields again a unit value for the vertical shift factor, $b_{RT}(T)$. However,

the horizontal shift factor, $a_{RT}(T)$ is highly affected by the moisture content, as shown in Figure 7. On average, the horizontal shift factor is halved by moisture content of 1% for temperatures exceeding 60°C. Below this temperature, the difference becomes even larger, approaching one order of magnitude. Hence, humidity accelerates the material creep, as expected. In Figure 7, it is also worth observing that the glass transition temperature, corresponding to a sudden drop of the horizontal shift factor, decreases with the moisture content. Therefore, humidity plays an important role in the material creep accelaration.

In Figures 5 and 6, Matlab routine was programmed for plotting the master curve making use of "nlinfit" command which estimates the coefficients of a nonlinear regression function, using least squares estimation. The master curve for dry test case was used as "reference" and the values for $ß_1^{RT}$, $ß_2^{RT}$ and $ß_3^{RT}$ as used in the expression were found to be 10.072, 3.51 and 5.991, respectively. This is very clear from the Figures 5 and 6 that the data fit very well to the master curve for the dry CFRP samples.

It is worth noting that the effect of humidity is accounted for in the activation energies and characteristic temperatures in the expression of the horizontal shift factor. It is clear that the storage modulus of coupons which were kept in water and absorbed water (i.e. wet samples) decreased at higher rates than those samples which were not kept in water (i.e. dry samples) as expected, with temperature approaching glass transition region. The temperature at which 30–50 carbon chains start to move. At the glass transition temperature, the amorphous regions experience transition from rigid state to more flexible state making the temperature at the border of the solid state to rubbery state. This can be concluded that the plasiticization effect of the absorbed water has led to a higher rate decrease in storage modulus for the wet samples.

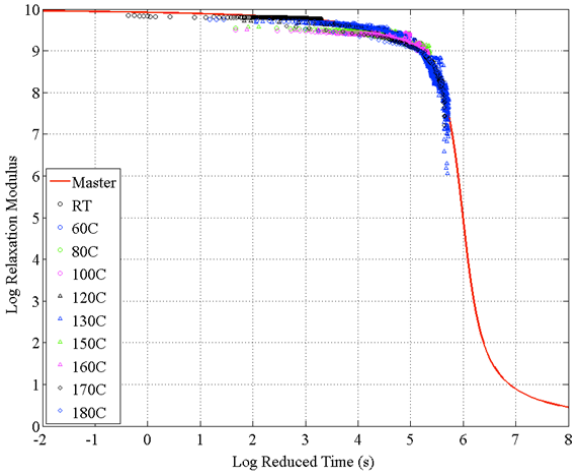

Figure 5. Master curve of creep relaxation modulus (dry samples)

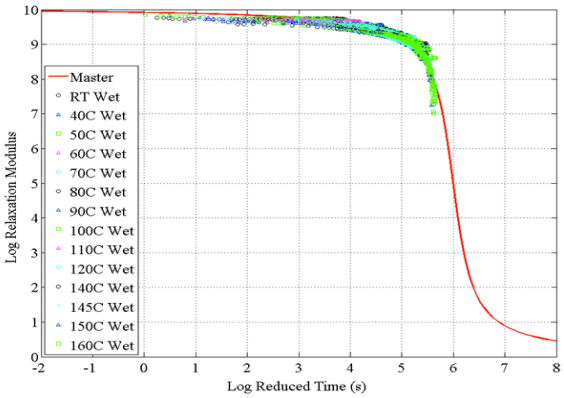

Figure 6. Master curve of storage modulus (wet samples)

When the experimental data for 1% H_2O were plotted; Arrehnius type regression with two different slopes was obtained as given in the expressions below and shown in Figure 7.

$$a_{RT}(T,H) = \frac{\Delta H_1}{R}\left[\frac{1}{T} - \frac{1}{T_0}\right] \quad T \le T_1$$

$$a_{RT}(T,H) = \frac{\Delta H_2}{R}\left[\frac{1}{T} - \frac{1}{T_2}\right] \quad T \ge T_1$$

A sharp drop of the horizontal temperature shift is noticed close to the glass transition temperature. This has to be attributed to the fact that the material comes into a "rubbery" like state due to this temperature transition and because of the materials being in rubbery state it will further enhance creep effect creep. It is also worth observing that the glass transition temperature, corresponding to a sudden drop of the horizontal shift factor, decreases more with the moisture content.

It is not clear why a "kink" like apprearance has observed in the horizontal shift factor for $T = T_1$. This might be attributed to the fact that the transition temperature T_1 corresponds to a phase transition of the blend as 977-2 is an epoxy blended with a thermoplastic polymer for toughening purposes.

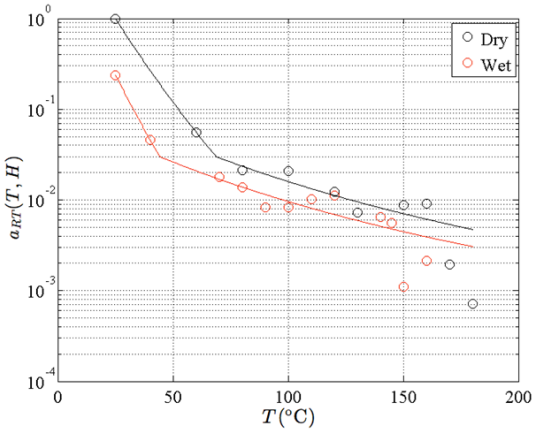

Figure 7 Horizontal time-temperature shift factor for experimental data of the dry and wet IM7/977-2

The master curves obtained for the storage modulus allow predicting the strength of the material in the full range of environmental conditions considered. The key outcome of this research is that the visco-elastic response of the material has a massive impact on the strength and that environmental effects strongly influence the visco-elastic response. It has also been observed that the shift factor associated with temperature and humidity can be suitably described by characteristic activation energies, which are inherent material properties.The residual strength properties of the fully saturated material have been found to be extremely low, particularly in terms of transverse tension. This poses significant challenges for the design of composite structures and highlights the importance of predicting the actual moisture content in service, as well as inhibiting the moisture ingress in composite materials via suitable surface protections.

5. CONCLUISON

In this study, time-temperature shift factor (TTSF) has been applied to study of the temprature and water absorption effect on the long-term viscoelastic response of IM7-carbon/977-2 epoxy composite materials.

The 3-point bending tests on dry as well as wet coupons using DMA show that the water uptake by the samples have affected the storage modulus, *E*s, of the IM7-carbon/977-2 epoxy composite at temperatures below Tg. It has been shown that the storage modulus of the wet samples were affected more severely and decreased at higher rates than the dry samples, with temperature approaching the glass transition region. This higher rate decrease in storage modulus for the wet coupons can be associated with plasiticization effect. However, no significant vertical shift factor is obtained from the investigated samples analysis. When the data were plotted for horizontal shift factor then Arrehnius type regression with two different slopes was obtained. All the experimental data for the dry as well as wet coupons fit very well by using the principle of TTSF and it is concluded that the properties of these composites can be relatively easily predicted through the application of TTSF by conducting traditional short-term laboratory tests. Future work for assessing the real hazard posed by environmental factors on the durability of composites in service should be focussed on the characterisation/prediction of the effects of cyclic temperature and moisture. This study has proven the viability of the timetemperature-humidity shift principles for steady environmental conditions.

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Persian Abstract

چکیده

خواص پلیمرهای تقویت شده با فیبر کربن (CFRP) تا حد زیادی در شرایط شدید محیطی تحت تأثیر قرار می گیرند. در این مقاله مطالعه تجربی به منظور تعیین پاسخ لمینتهای اپوکسی اکسید کربن IM7-karbon / 977-2 در شرایط مرطوب و شرایط مختلف ارائه شده است. از آزمون خمش ۳ نقطه ای کوتاه مدت با استفاده از آزمون خزش دینامیکی مکانیکی (DMA) برای آزمایش نمونه های خشک و اشباع شده در سطوح مختلف دما استفاده گردید. کوپنهای خشک در دمای اتاق RT و در دمای ۶۰- ۱۲۰ درجه سانتیگراد با افزایش ۲۰ درجه سانتیگراد و سپس در دمای ۱۳۰ درجه سانتیگراد ، ۱۵۰ تا ۱۸۰ درجه سانتیگراد با افزایش ۱۰ درجه سانتیگراد برای آزمایش بعدی مورد استفاده قرار گرفت. کوپنهای اشباع شده (مرطوب) در RT، ۴۰ C ° 120 - با ۱۰ درجه سانتیگراد افزایش دما برای هر آزمایش بعدی و در ۱۴۵ درجه سانتیگراد ، ۱۵۰ درجه سانتیگراد و ۱۶۰ درجه سانتیگراد مورد آزمایش قرار گرفتند. ضریب تغییر زمان دما (TTSF) استفاده شد و نشان داده شده است که رفتار ویسکوالاستیک لمینت های اپوکسی داخلی IM7-کربن / ۹۷۷-۲ درونشویه ، با استفاده از TTSF با دقت پیش بینی می شود. همچنین نشان داده شده است که تعیین رفتار ویسکوالاستیک در دماهای بالا به پیش بینی دما در زیر دمای انتقال شیشه با استفاده از TTSF کمک می کند. عمر طولانی مدت مواد با استفاده از TTSF با انجام آزمایشات سنتی آزمایشگاهی کوتاه مدت پیش بینی می شود.