Influence of $CaCO_3$ in pultruded glass fiber/unsaturated polyester resin composite on flexural creep behavior using conventional and time-temperature superposition principle methods

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Abstract: The effect of calcium carbonate on the creep phenomenon of glass fiber/unsaturated polyester resin composites (GFRP) (obtained by pultrusion) was investigated due to failure that happened during installation of one of the composite transmission tower. To assess long-term creep and predict the life of composites, a conventional bending method with 45-day creep and time-temperature superposition principle (TTSP) were used. In the conventional method, the composites (with and without calcium carbonate) underwent only slight deformation. It was found that their lifetime could be 25 years. However, based on the standard curve obtained by the TTSP method, significant differences were shown in the stability of calcium carbonate composite samples at 95°C (111 days) and 160°C (11 days). It was found that the addition of calcium carbonate extends the service life of the tested composites. Thus, the results obtained by the conventional method do not reflect the real behavior of the samples over time. On the other hand, the TTSP method allows better estimation of the long-term durability of composites.

Keywords: composite material, flexural test, unsaturated polyester resin, time-temperature superposition, dynamic mechanical analysis, master curve.

Ocena wpływu $CaCO_3$ na pełzanie przy zginaniu otrzymanego w procesie pultruzji kompozytu włókno szklane/nienasycona żywica poliestrowa za pomocą metody konwencjonalnej i metody superpozycji czasowo-temperaturowej

Streszczenie: Zbadano wpływ węglanu wapnia na zjawisko pełzania kompozytu włókno szklane/nienasycona żywica poliestrowa (GFRP) (otrzymanego metodą pultruzji). Do oceny długoterminowego pełzania i prognozowania czasu użytkowania kompozytów stosowano konwencjonalną metodę zginania z 45-dniowym pełzaniem oraz metodę superpozycji czasowo-temperaturowej (TTSP). W konwencjonalnej metodzie kompozyty (z węglanem wapnia i bez niego) uległy tylko niewielkim odkształceniom. Stwierdzono, że czas ich użytkowania może wynosić 25 lat. Natomiast na podstawie krzywej wzorcowej, otrzymanej metodą TTSP, wykazano istotne różnice trwałości próbek kompozytów z węglanem wapnia w temperaturze 95°C (111 dni) i 160°C (11 dni). Stwierdzono, że dodatek węglanu wapnia wydłuża czas użytkowania badanych kompozytów. Wyniki uzyskane konwencjonalną metodą nie odzwierciedlają więc rzeczywistego zachowania się próbek w czasie, natomiast metoda TTSP umożliwia lepsze oszacowanie długoterminowej trwałości kompozytów.

Słowa kluczowe: materiał kompozytowy, próba zginania, nienasycona żywica poliestrowa, superpozycja czasowo-temperaturowa, dynamiczna analiza mechaniczna, krzywa wzorcowa.

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An electrical transmission line is composed of electrical conductors carrying an electrical signal from the power generator to consumer. The cables are carried by transmission tower, which is made up of galvanized steel in the form of latticed steel and monopole steel tube. Commonly, the transmission tower is built up of peak, tower body, cage and cross-arm [1, 2]. The cross-arms are usually located near the top of the towers with a higher mean wind speed [3]. Visweswara [4] indicated that the height and width of the cross-arms is the general form of the tower carrying electrical conductors, which is a whole network of high voltage transmission line construction. In China, the composite cross-arms are produced and used in the corridor of the transmission line, and the weight is reduced to form the arm body rather than the conventional steel components. Usually, besides galvanized steel the cross arm in suspension tower is made up of hardwood material such as Chengal wood. However, a wooden material commonly experienced natural wood defects throughout their long service operation [5–7]. This issue has urged researchers to find a solution to substitute the current wooden cross arm with composite material [8, 9]. A composite is a material made from two or more different materials with different physical and chemical properties. When mixed, they are stronger than each of them separately [10–16]. Composites offer many advantages such as light weight, strength related to weight, corrosion resistance, design flexibility, part consolidation, dimensional stability, radar transparent, durability, high mechanical as well as thermal properties [17–21]. The composite material is widely used in many applications due to their excellent mechanical properties such as composite panel [22], electrical appliances [23], wind turbine [24], automotive components [25] and household product [26].

Composite cross-arm also has advantages such as excellent insulation properties, high mechanical strength, lightweight, corrosion resistance, ease of installation and much more. Composite cross-arm is also said to be revolutionary in composite material implementations in the transmission line through the use of the composite insulator [27]. Commercial thermosetting resins are commonly used with fillers, such as unsaturated polyester resins [28], as a neat [29] and also reinforced by fibers [30]. Murafa *et al*., [31] stated that this unsaturated polyester resin is typically used to be filled with similar and different materials such as iron, wood, plastics, non-metals or even ceramics. Reduction of surface size occurs at room temperature by curing process. The ability to polymerization via crosslinked polymer can be achieved when the resin has a good performance in curing rapidly, low viscosity and excellent compliance with mineral fillers and fiber reinforcement products.

The effect of fiber curing on the flexural stress-free conduct of fiber characteristics, as Hariharashayee *et al.* assesses [32] accurately on Sansevieria Cylindrical Fiber Reinforced Polymer Composite (SCFP) is presented as the upper and lower surfaces of the specimen under a three-point bending force, the bending stress (compression and strain) and the axisymmetric plane subjected explicitly to shear stress. Flexural testing was also carried out on the system in compliance with ASTM D2344-84, where 150 mm long and 20 mm wide samples were cut and mounted with a span-to-depth ratio of 16 : 1 on three-point bending [33]. Chaudhary *et al*., [34] specifies that flexural strength is essential in the development of composite material when the material is applied to lateral loads. Nearly equivalent to the flexural modulus used to calculate the rigidity of composite materials. This three-point bending test is com-

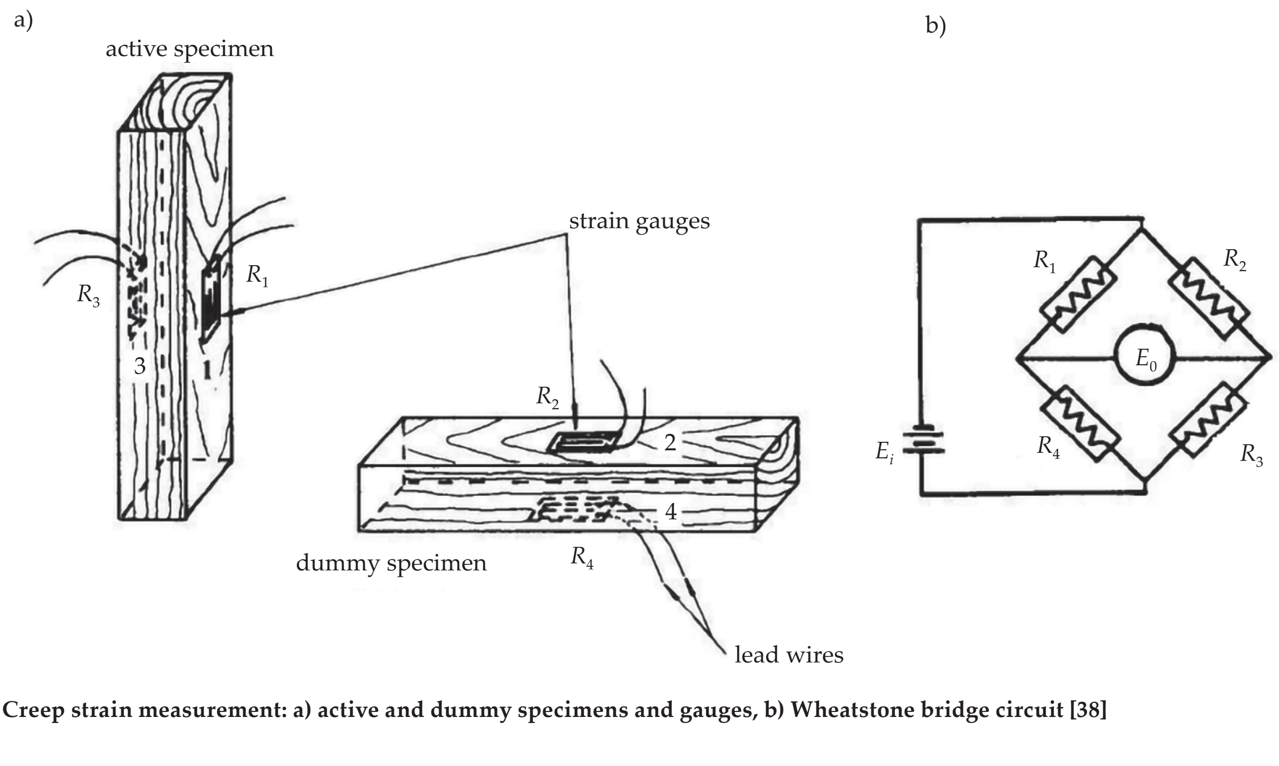


Fig. 1. Creep strain measurement: a) active and dummy specimens and gauges, b) Wheatstone bridge circuit [38]

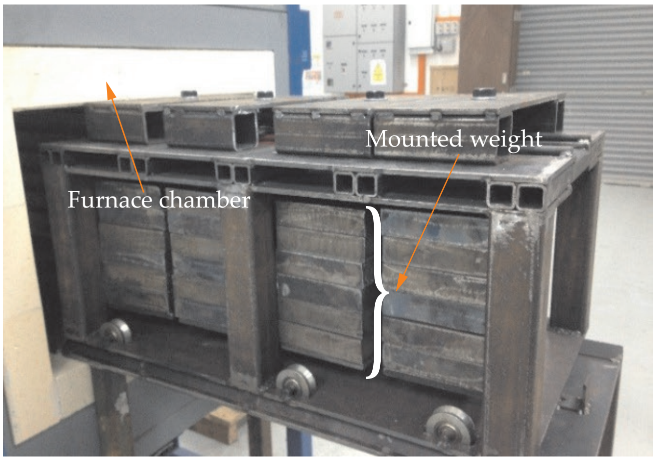


Fig. 2. Short-term accelerated test apparatus [38]

monly used to determine flexural strength and flexural modulus. That has been addressed further by Acharya & Soma, [29], where they studied the flexural characteristics of woven jute glass hybrid composite reinforced with natural fiber. They found that they could enhance the flexural properties of jute E-glass epoxy and its composites by using glass fiber as extreme fiber.

Anand *et al.* [35] mentioned that creep is characterized as plastic deformation of the material at sustained stress-temperature over a prolonged time. The creep effect changes the size and shape of material when exposed to long-term loading. To be more specific, the creep study is based on creep strain versus time to explain the mechanical phenomenon further. These two variables are dependent on each other as the creep strain is based on Hooke's law [36]. National Instrument (NI) has become commonly one of the data loggers used to measure strain and to record material breakdown for duration of time. As mentioned by Stochioiu *et al.* [37], the implementation of National Instruments NI PXI 4220 data acquisition module was used to assess stress in quasi-static testing via MTSC-45 tensile testing machine (TTM) fitted with a load cell of 100 kN and deformation with GOM Aramis measuring device.

Strain gauges were glued to two opposite sides of each active and dummy specimens used in the accelerated test to measure the compressive strain and linked to a Wheatstone bridge circuit as seen in Fig. 1 and Fig. 2 [38].

The pressure gauges used in the experiment were self-temperature, which compensated for constant alloy foil strain gauges attached to the plastic backing. The gauges were glued to the wood surface using an epoxy adhesive, AE-10, developed by the manufacturer of strain gauges, and covered by a moisture-protective coating. The gauge arrangement shown in Fig. 1 was designed to calculate pure compressive pressure and remove bending and temperature effects [39]. The creep strain was analyzed and read on a mobile IBM-PC with a data acquisition system.

According to Ghosh, *et al.* [40], creep can be described as the tendency of a material to buckle permanently under the effect of mechanical stresses due to long term contact to high levels of stress. For a completely elastic material (both linear and nonlinear), strain (ε) is a time-dependent property and is directly related to the applied stress (σ). Polymers such as epoxy demonstrate viscoelastic properties. Elastic energy is being restored within them when distorted, and the energy is utilized to return to their original form after the deforming stress is removed. Above a certain temperature, they act as liquids and display viscosity. $S(t)$ is called the creep compliance, which can be defined as the inverse of stiffness. The equation of the creep compliance is as follows:

$$S(t) = \frac{\varepsilon(t)}{\sigma} \tag{1}$$

A general polymeric material takes around tens of thousands years to deform and therefore it is impractical to be determined through long-term creep testing until the completion of the designated lifetime of the specimen. Thus, an accelerated creep testing on materials at an elevated temperature over a shorter period is conducted, and the results are extrapolated to ascertain the behavior of materials at room temperature for an elongated time. As mentioned by Guimaraes [41], most of the stress-rupture models have been based on tests executed at room temperature and at high-stress levels, when creep failures can be predictable in days.

The time-temperature superposition is broadly used in the linear viscoelastic region (or infinitesimal strain) when illustrating mechanical relaxation in polymers science. With any experimental set up close to T_g in terms of strain rate, $\dot{\varepsilon}_1$ and temperature, T_0, such principle results in the fact that storage and loss moduli should be equal to storage and loss moduli for all other conditions close to T_g, ($\dot{\varepsilon}_2$, T_2), using a shift factor, a_t Ferry [42] that depends only on T_0 and T_2 such as:

$$\dot{\varepsilon} = \frac{a_{T_2}}{T_0\dot{\varepsilon}_1} \tag{2}$$

At 140°C and above, the material (PMMA) is in quasi-liquid, making the tensile tests highly challenging. Using the TTSP method, the obstacle can be overcome by selecting conditions with a lower temperature but lesser strain rate for verifying low equivalent strain rate. Pooler and Smith [43], justified where master curves have come from creep curves that were formed by a short-term creep curve based on temperature-time superposition principle, and not a full-scale long-term creep test. Koemer *et al.*, [44] had come out with a common form of the equation of Arrhenius. It is based on "rate-process theory", which defines the relation between the rate of reaction and temperature for many physical and chemical reactions. The equation as follows:

$$k = k_0 e^{-\left(\frac{E}{RT}\right)} \tag{3}$$

where k – kinetic reaction rate, k_0 – rate constant, E – activation energy, R – universal constant, T – absolute temperature.

For temperature-time cases, the rate of reaction corresponds to the creep strain rate $\dot{\varepsilon}$. Even though the Arrhenius equation designates the rate of reaction, time is not incorporated as a variable in Eq. (3) can be set by contrasting the ratio of strain rate, $\dot{\varepsilon}_1$, at temperature T_1, to strain rate, $\dot{\varepsilon}_2$, at temperature T_2 as follows:

$$\ln\left(\frac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2}\right) = \frac{E}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \tag{4}$$

Equation (4) can be applied to foretell the creep strain rate at a reference (room) temperature from the creep strain rate quantified at an elevated temperature. The Arrhenius equation guesses that the viscoelastic mechanism (*e.g.*, creep) remains unaffected at elevated temperature.

EXPERIMENTAL PART

Materials

In this experimental investigation on the test coupon of unsaturated-polyester composite glass fiber (GFRP), samples were obtained by pultrusion. The process itself was prepared by Electrius Sdn. Bhd. a joint venture company with Tenaga Nasional Berhad, TNB. The coupons are used for further analysis of the characterization and behavior of the materials which undergo short term analysis procedure.

The composite samples in Fig. 3 are prepared with 2 groups of materials. One sample is prepared from the material of unsaturated polyester resin, fiberglass with addition of calcium carbonate while another sample without calcium carbonate. Both samples with depth of 8 mm and 7 mm, respectively, and length of 176 mm. Four layers of glass fiber sheets are applied.

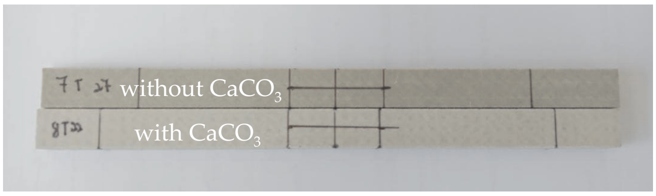


Fig. 3. Coupon test bar sample prepared with and without calcium carbonate

Methodology

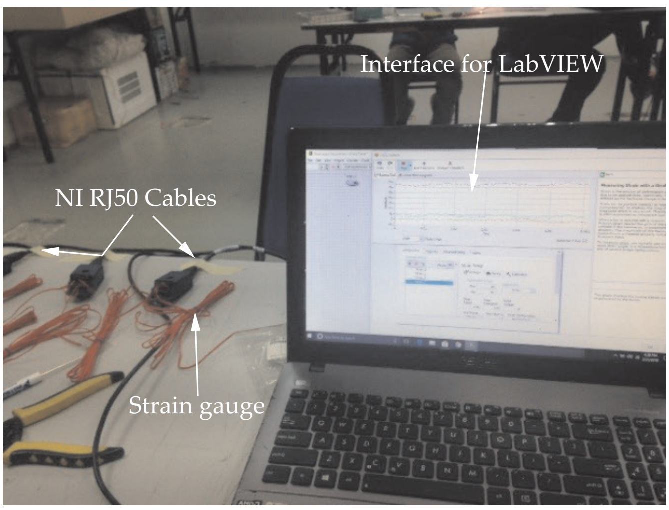
Preparation of experimental (conventional method) set up on long term creep testing

The preparation of the conventional method for testing the sample bars is carried out and shown in Fig. 4. The samples set up first the National Instrument (NI 9237, Data Logger) must be calibrated for the next process of collecting the data for the creep deformation on the samples of fabricated cross-arm. 4 NI RJ50 Cables are used. Two of them are dummy cables while the other two cables are implemented to record the data.

Figure 5 shows the set up for long-term the 3-point bending flexural (ASTM D790) creep data under a consistent room temperature of 30°C. The experiment has been carried for 1000 hours which was equivalent to 42 days under a constant load of 60 kg per sample. Again, KYOWA strain gauges have been mounted to each sample similar to short-term creep test set up, and the best data has been documented. All obtained data has been formulated into a line equation for further analysis. The line equation for all lines has been plotted to predict the service life using the conventional method (v). Using the formula used by Somaiah *et al.*, [46]

$$Flexural\ strength = \frac{3Fl}{2wd^2}\left(\frac{\text{N}}{\text{mm}^2}\right)$$

a)



b)

Fig. 4. a) Calibration of the coupon test set up, b) National Instrument NI RJ50 cable

a) b)

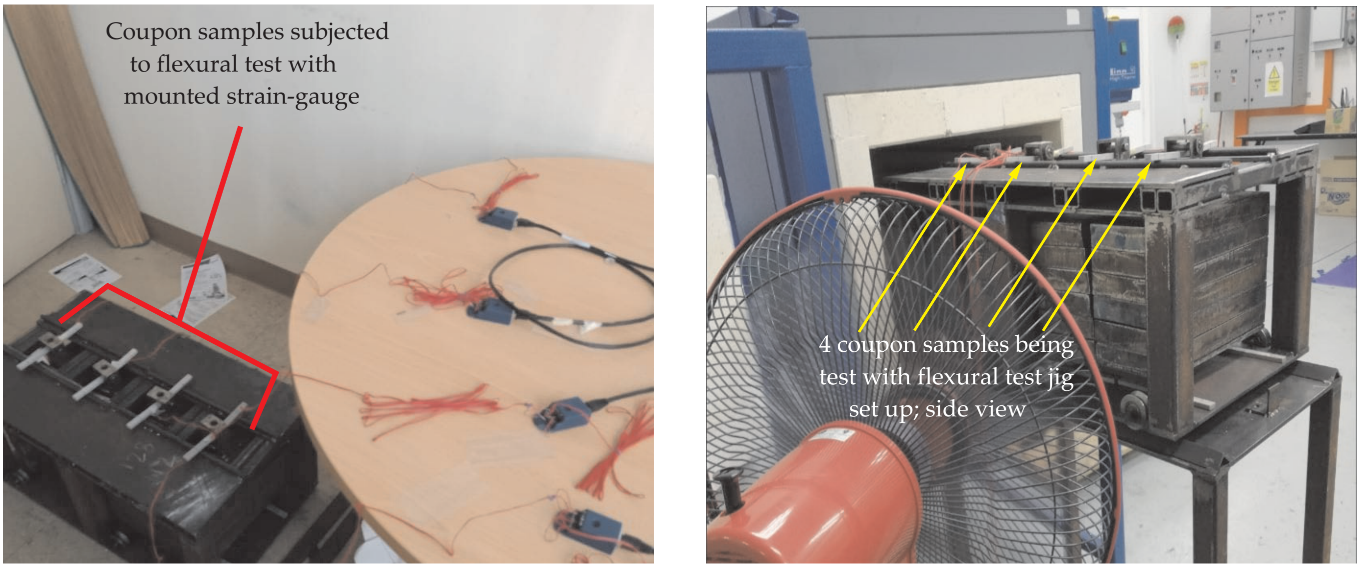


Fig. 5. Calibration of a set of test instruments for short term creep test at room temperature: a) top view b) side view

Table 1. Flexural strength value for the samples with and without $CaCO_3$

Material	Load *F* N	*L* mm	*d* mm	*w* mm	Flexural strength N/mm^2
GFRP with $CaCO_3$	588.6	176	8	12	202
GFRP without $CaCO_3$	588.6	176	7	12	264

the flexural strength of the specimen can be calculated, and Table 1 is formed.

From the Table 1 it is clear that flexural strength of GFRP sample without $CaCO_3$ is higher than of the one with $CaCO_3$. But, this is just an initial result of comparison and further analyzed during creep testing process where high temperature with a constant load are applied.

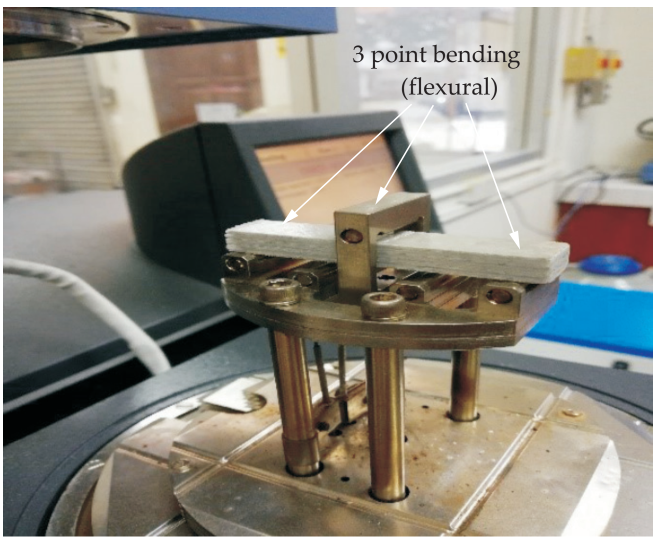


Fig. 6. 3-point bending test using TA instruments for DMA testing

Preparation of Dynamic Mechanical Analysis (DMA) set up for time-temperature superposition (TTSP) data analysis

Figure 6 shows the process of 3-point bending flexural under the TA Instruments and heat has been applied as a mode of frequency sweep until the glass transition temperature 160°C. This process is done and predicted to fail around temperature 160°C as the T_g defines the maximum glass transition temperature at break before, therefore comparison can be made easily during the analysis of the set up. The dimension of the prepared sample for the test is 58 mm length × 12 mm width × 3 mm depth according to the standard set up by Nan and DeVallance [47].

RESULTS AND DISCUSSION

Analysis for long term test

Figure 7 shows the curve of strain *vs.* time for 2 sets of the dummy and actual samples. Those two lines: W/O $CaCO_3$ T (actual sample) without and W/O $CaCO_3$ D (dummy sample) and W $CaCO_3$ T (actual sample) with $CaCO_3$ and W $CaCO_3$ D (dummy sample) which are moving through consistent negative creep deformation indicates the same value of strain of 0.003E+00 mm/mm for all samples (line position is adjusted to differentiate the pattern among all lines) carried out for 1000 hours which was equivalent to 3 600 000 s of the experimental setup. The line equation for all lines has been plotted in order to predict the service life using the conventional method (y = mx + c).

For line 1: Actual sample for a sample without calcium carbonate:

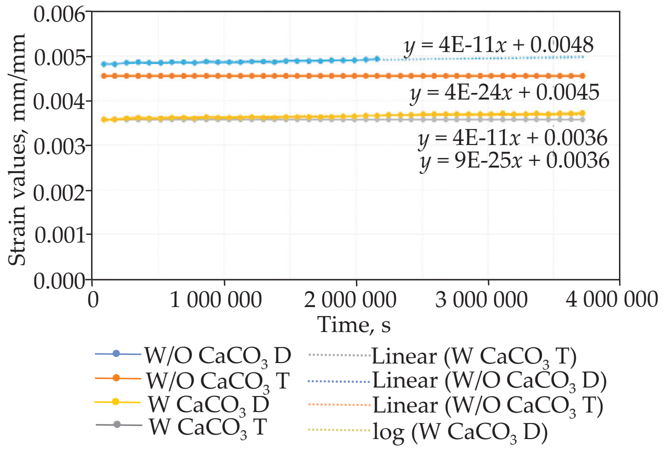


Fig. 7. Strain *vs.* time (s) of 2 sets of sample (2 dummy samples [W $CaCO_3$ D & W/O $CaCO_3$ D], 2 actual samples [W $CaCO_3$ T & W/O $CaCO_3$ T]) coupon test at constant temperature (30°C); note: W – with, W/O – without, D – dummy, T – tested

$$\frac{0.03076765 - 0.0048}{4 \cdot 10^{-11}} \simeq 769191250 \text{ s} = 24 \text{ years}$$

For line 3: Actual sample for a sample with calcium carbonate

$$\frac{0.03076765 - 0.0036}{4 \cdot 10^{-11}} \simeq 769191250 \text{ s} = 24 \text{ years}$$

Therefore, it can be concluded that by extrapolating the graph into the conventional method which was using the conventional line equation $y = mx + c$, and by doing creep testing under constant room temperature (30°C), both lines (both sample with and without $CaCO_3$) shown a better prediction of service life which was recorded to be usable up for 769191250 seconds which is until 24 years of services.

Analysis using TRIOS software time-temperature superposition principle analysis (shifting process)

Table 2, depicts the table of data interface of TRIOS software for time-temperature superposition method for producing master curve process. The first reference temperature was set to be 95°C for DMA analysis of sample of pultruded cross-arm composite with the presence of calcium carbonate.

Figure 8 shows shifting process of the pultruded cross arm composite sample for the reference temperature 95°C. Yao *et al.*, [48] specified that the data of frequency sweep was moved by using TTS of WLF models and the master curves were obtained. WLF models are used as it is embedded in TRIOS Software for current use of industry through TA Instruments. Every single parameter being put inside TRIOS Software, and there is no need for the researcher to calculate manually. Thus time-temperature superposition for life-prediction of the samples can be predicted successfully.

Figure 9 illustrates the master curve by using shift factor of the different composites function (storage modulus and loss modulus) according to the WLF equation, and the reference temperature at 95°C the prediction of service life for pultruded composite cross-arm sample was in the range of 10^6 s –111 days.

Figure 10 displays the shifting process of the pultruded cross-arm composite sample for the reference temperature 160°C which was said to be the glass transition temperature (determined during the preparation of DMA set up) and has been parallel to the result in a short-term test where at the temperature of 120°C, the pultruded composite cross-arm sample was fractured and failed. Compared with the experimental set up byYao *et al.* [48], also specified that the data of frequency sweep were

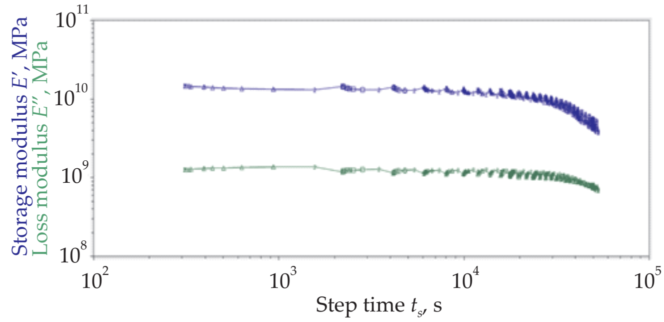


Fig. 8. Storage and loss moduli as a function of time at 95°C

T a b l e 2. Time (*t*), temperature (*T*) and heat flow of DMA analysis for coupons for reference temperature 95°C configurations

	Time *t* s	Temperature *T* °C	Heat Flow (Normalized) *Q* W/g
1	0.0299996	26.28	-0.033
2	0.229999	26.28	-0.033
3	0.429999	26.28	-0.032
4	0.630000	26.28	-0.032
5	0.830000	26.28	-0.031
6	1.03000	26.28	-0.031
7	1.23000	26.28	-0.030
8	1.43000	26.28	-0.030
9	1.63000	26.28	-0.029
10	1.83000	26.29	-0.029
11	2.03000	26.29	-0.029
12	2.23000	26.29	-0.028
13	2.43000	26.29	-0.028
14	2.63000	26.29	-0.028
15	2.83000	26.30	-0.027
16	3.03000	26.30	-0.026
17	3.23000	26.31	-0.026
18	3.43000	26.32	-0.025
19	3.63000	26.32	-0.025
20	3.83000	26.33	-0.025
21	4.03000	26.34	-0.024
22	4.23000	26.35	-0.023
23	4.43000	26.37	-0.022

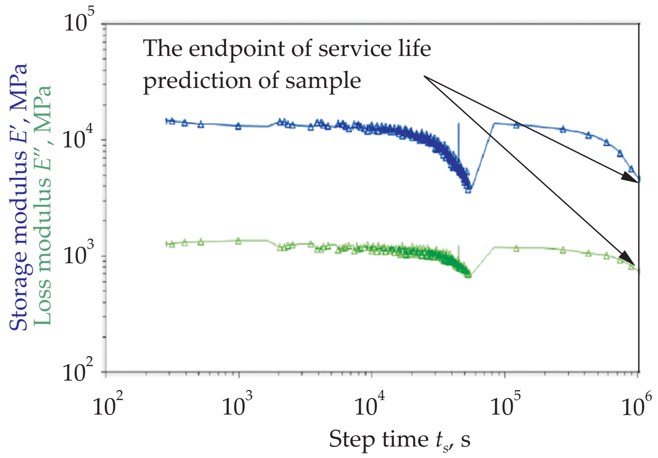


Fig. 9. The master curve of TTS for reference temperature 95°C

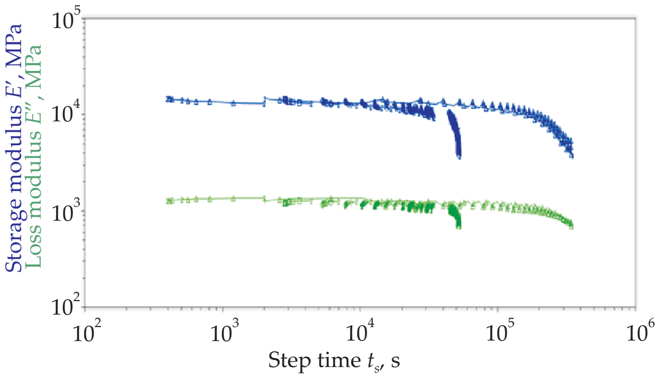


Fig. 10. The graph shifting process for coupon at reference temperature 160°C

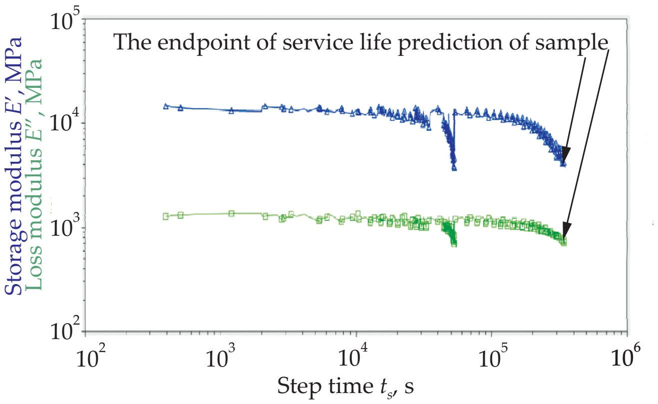


Fig. 11. The master curve of TTS for reference temperature 160°C

moved by using TTS of WLF models, and the master curves were obtained.

Figure 11 demonstrates the master curve by using shift factor of the different composites function (storage modulus and loss modulus) according to the WLF equation and for the reference temperature 160°C, the prediction of service life for pultruded composite cross-arm sample was 11 days.

CONCLUSIONS

Study on the pultruded composite cross-arm coupons (unsaturated polyester resin with fiberglass chopped strand mat composite) using conventional (flexural test jig + creep testing) and using time-temperature superposition principle (TTSP), shows that the prediction of longevity is possible. Conventional analysis for long term analysis, the experiment was done for all of the samples (dummy and tested) with and without the presence of calcium carbonate impregnated, had shown consistent results of strain deflection and was conventionally predicted to have a service life up to 25 years. It was proven that the material still was in a safe condition for installation. Therefore, the research has come up with a conclusion that the materials (pultruded composite cross-arm samples) can be used to the production of actual composite cross-arm in the future projects as the market price is economical and are said to be safely implemented by the contractors and the transmission line industry. As the main objective is to investigate the influence of calcium carbonate $CaCO_3$ in the glass fiber or unsaturated polyester composite on flexural and creep, it can be concluded that the presence of $CaCO_3$ slightly improve the overall performance of GFRP and it is used especially in the installation for cross-arm pultruded composite.

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