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**DOI**

[10.1177/0021998319845045](https://doi.org/10.1177/0021998319845045)

**Publication date**

2019

**Document Version**

Final published version

**Published in**

Journal of Composite Materials

**Citation (APA)**

Hagenbeek, M., Dias, M. M., Sinke, J., & Jansen, K. (2019). Creep behaviour of FM906 glass-fibre epoxy as used in heated fibre metal laminates. *Journal of Composite Materials*, 53(26-27), 3829-3840. <https://doi.org/10.1177/0021998319845045>

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# Creep behaviour of FM906 glass-fibre epoxy as used in heated fibre metal laminates

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Journal of Composite Materials  
2019, Vol. 53(26–27) 3829–3840  
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DOI: 10.1177/0021998319845045  
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## Abstract

An innovative deicing system for aircraft leading edges has been developed which integrates heater elements into fibre metal laminates. Such an electrical system can lead to weight reductions and more efficient performances compared to conventional bleed air systems. However, the combination of thermal and mechanical loadings also raises new questions on the durability of such a structure, in particular due to the repeated heating to elevated temperature. The linear viscoelastic creep behaviour, including the effects of temperature and ageing, is therefore investigated for manufactured FM906 glass-fibre epoxy composite as used in heated GLARE. A master curve is derived based on the time–temperature and time–age superposition. The effect of physical ageing during loading is included in a long-term creep prediction.

## Keywords

Deicing, heated GLARE, viscoelastic creep, physical ageing, long-term durability, time–temperature superposition, time–ageing time superposition

## Introduction

### *Heated fibre metal laminates*

Fibre metal laminates (FMLs) have been successfully introduced into aerospace. GLARE, glass-fibre reinforced aluminium, for example is nowadays used in the Airbus A380. The material offers improved fatigue resistance and damage tolerance over monolithic aluminium. A heating functionality can be added by integrating electrical heater elements in between two glass-fibre epoxy layers of the alternating metal and glass-fibre epoxy lay-up. The heater elements are thereby protected from environmental influences by the outer aluminium layer and electrically shielded by surrounding glass-fibre epoxy layers. This so-called heated GLARE can be used for de- or anti-icing of aircraft leading edges. Figure 1 shows the schematic lay-up of heated GLARE. The proposed system can lead to weight reductions and result in more efficient performances compared to conventional bleed air systems. However, the repeated heating to temperatures above 60°C may age the material considerably faster. This paper will therefore investigate the effect of combined thermal and mechanical loading on the creep behaviour of FM906 glass-fibre epoxy composite.

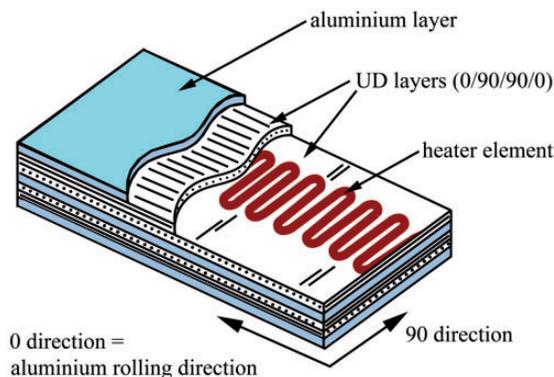
There are two major thermal loading conditions for heated leading edges. Every flight the service temperature ranges between –55 and 70°C,<sup>1</sup> which are relatively slow thermal fluctuations. The highest risk of ice accretion is present at flight operations within the –20 to +10°C temperature range. Ice can form in the presence of visible moisture or high humidity above 0°C, when the aircraft structure remains at 0°C or below, for example after a long flight at high altitude.<sup>2</sup> In case the de- or anti-icing system is turned on, a second thermal loading condition is present with a temperature range of –20 to 80°C.<sup>3,4</sup> This thermal cycle typically has a high frequency in the order of minutes. The temperature of the structure can thus vary from –55 to 80°C.

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**Figure 1.** Schematic lay-up of a heated GLARE laminate.

The conventional 120°C curing manufactured FM94 glass-fibre epoxy prepreg used in GLARE has a  $T_g$  of 103°C in dry condition.<sup>5</sup> Although the service temperature is indicated by the manufacturer up to this temperature level, beyond 70°C the stiffness of FM94 glass-fibre epoxy prepreg significantly decreases.<sup>6</sup> To enable higher service temperatures, manufactured FM906 glass-fibre epoxy prepreg is used in heated GLARE. The FM906 prepreg is a 180°C curing epoxy system with a  $T_g$  of 135°C and an estimated service temperature of 120°C.<sup>7</sup>

The difference in thermal expansion of the individual constituents leads to residual stresses in the laminate after curing. These stresses are superimposed with the mechanical and thermal loadings in service. As elevated temperature can accelerate creep,<sup>8</sup> a concern arises about the creep behaviour of the GLARE material under the more severe and frequent elevated temperature conditions. At the same time, the residual stresses induced by curing decrease by increasing temperature. The elevated temperatures during icing conditions are expected to accelerate physical ageing of the epoxy too.<sup>8,9</sup>

### Creep in FMLs

For FMLs, the literature on creep is limited. Creep research has been performed by Pindera on ARALL (aramid fibre reinforced aluminium) at 121°C and mechanical stress levels between 207 and 345 MPa.<sup>10</sup> Daghigh et al.<sup>11</sup> investigated the effect of creep in basalt FMLs at 200°C. The mechanical and thermal levels used by Pindera and Daghigh are not expected to be present in the case of a deicing leading edge application.

For aircraft leading edges with loading conditions in the linear elastic regime of the Glare material and below 120°C, the effect of creep in aluminium and glass-fibre is considered negligible. Thus, in cases where the FML loading is mainly taken by the aluminium and

glass-fibre, the effect of creep is considered to be small as well. However, creep effects in FMLs can still be present in areas of high local shear, as a result of buckling, residual stresses, or stress concentrations, for example. Depending on the exposure times, creep effects can already be present in the glass-fibre epoxy at moderately elevated temperature or even room temperature (RT) in these cases.<sup>12</sup>

Testing at ambient temperature might require months or years to complete. For laboratory testing of polymers, the time–temperature superposition (TTS) principle is therefore used to accelerate testing. By testing at elevated temperature, the long-term viscoelastic responses can be investigated on a much shorter and more practical time scale.<sup>13</sup> To assess the effect of creep for matrix-dominated properties of heated GLARE, the current creep study will be performed at elevated temperatures and will focus on the FM906 glass-fibre epoxy system.

### Creep response of FM906 glass-fibre epoxy

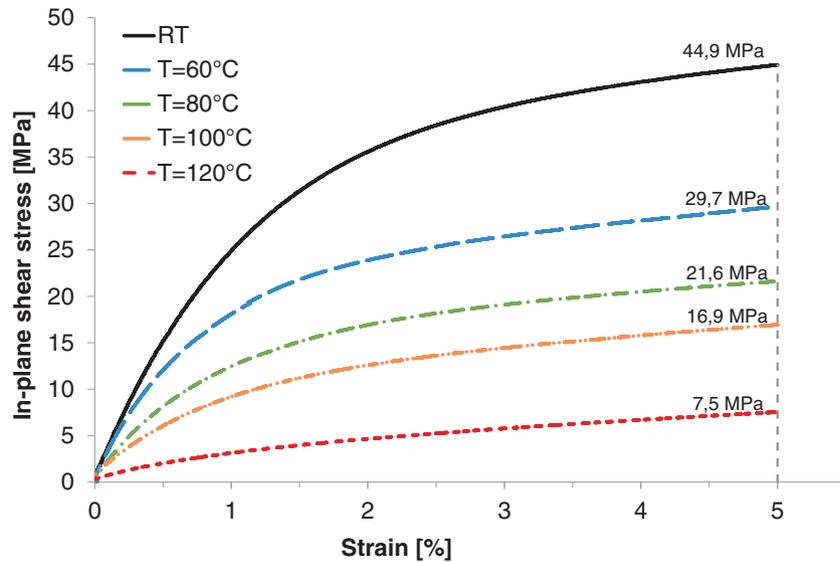
When the epoxy matrix in GLARE is loaded both thermally and mechanically over longer periods of time, a slow and progressive deformation of the material, known as creep, can take place. For linear viscoelastic materials, a given load increment results in a similar increment in the creep deformation. The creep strain can thus be normalised to the applied stress, and a material property called creep compliance  $S(t)$  is obtained

$$S(t) = \frac{\epsilon(t)}{\sigma_0} \quad (1)$$

where  $\epsilon(t)$  is the measured strain as a function of time and  $\sigma_0$  is the applied stress. The creep compliance as a function of time can be graphically depicted, which enables the quick comparison of creep data at different stress levels.

Prior to creep testing, a preliminary experimental investigation on the in-plane shear strength of FM906 glass-fibre epoxy as a function of temperature was carried out, and the results are shown in Figure 2. The in-plane shear response was determined by tensile tests on a 16-layer  $\pm 45^\circ$  laminate at RT, 60, 80, 100, and 120°C temperatures. The tests were performed in accordance to ASTM test standard D3518/D3518M.<sup>14</sup> The test specimens had a 250 mm length, a 25 mm width and a total thickness of 2.0 mm. The ultimate in-plane shear strength has been determined at 5% strain in accordance to the test standard.<sup>14</sup>

As can be seen in Figure 2, the shear strength at 80°C is reduced to 48% of the value at RT, and at 120°C even further to only 17% of the original



**Figure 2.** Experimental in-plane shear response of FM906 glass-fibre epoxy.

strength. To avoid premature damage and failure, any creep test at a given temperature needs to be performed below the corresponding static strength level.

### TTS

The TTS principle assumes that higher temperature accelerates the creep process and a direct equivalency between time and temperature exists. The theory was originally developed for pure polymers,<sup>13,15</sup> but several researchers have successfully expanded its use to polymer matrix composites.<sup>16–22</sup> By testing at elevated temperature, the creep is accelerated by a factor  $a_T$  and the required test time can be reduced. The resultant data are shifted to lower temperatures by the same shift factor value,  $a_T$ , in order to extend the prediction. The shifted time scale is called reduced time,  $t_{\text{red}}$ , and can be written as

$$t_{\text{red}} = a_T t \quad (2)$$

where  $t$  is the elapsed test time. The result of this shifting is a master curve depicting  $S$  versus  $t_{\text{red}}$ . A material for which this shifting along the time axis results in a satisfactory master curve is called a thermo-rheological simple material (TSM). If the curves do not overlap well by only shifting along the time axis, the material is called a thermo-rheological complex material (TCM).<sup>23</sup>

### Time–age superposition

Physical ageing is the slow evolution of a glassy polymer towards thermodynamic equilibrium by time-dependent changes in volume, enthalpy, and entropy,

as well as mechanical properties. All polymers undergo physical ageing at temperatures below their glass transition temperature,  $T_g$ .<sup>15</sup> However, unless the temperature is very close to the  $T_g$ , the ageing process will typically take years to complete. Physical ageing causes embrittlement and microcrack growth in the polymer. This results, amongst others, in increased shear strength, compression yield and hardness. In contrast, tensile strength, strain to failure and fracture toughness are reduced.<sup>12</sup> While an increase in temperature accelerates creep, physical ageing slows down creep.

Physical ageing distinguishes from other ageing phenomena (chemical ageing, damage evolution, etc.) due to thermal reversibility. Heating a polymer to a temperature above  $T_g$  erases the effects of physical ageing.<sup>15,23–25</sup> This is a phenomenon known as rejuvenation. Struik<sup>15</sup> showed that for TSM undergoing isothermal physical ageing, the short-term compliance response ( $S$ ) is related to a momentary compliance of the reference curve ( $S_{\text{ref}}$ ) by the equation

$$S(t; t_e, T) = S_{\text{ref}}(a_e t; t_{e,\text{ref}}, T) \quad (3)$$

where  $t_{e,\text{ref}}$  is the isothermal ageing time at which the reference curve was defined,  $t_e$  is the ageing time at which the short-term test is taking place,  $T$  is the common isothermal temperature at which both the short-term test and the reference curve were obtained, and  $a_e$  is the shift factor due to physical ageing.

In isothermal ageing tests, the short-term ( $S$ ) curves are obtained. The duration of the tests is generally chosen to be small enough so that physical ageing is assumed to be negligible during testing. Typically, this is accomplished by limiting the test duration,  $t$ , to a time corresponding to  $0.1 t_e$ <sup>15</sup> or  $0.3 t_e$ .<sup>26</sup> This is

called the snapshot condition. Creep tests that respect the snapshot condition are called short-term tests, and the individual curves obtained by following through these procedures are called momentary curves.

If the material is loaded for a time that exceeds the snapshot condition, then physical ageing occurs during testing and therefore the tests are called long-term tests. The collection of momentary curves can be individually shifted by ageing shift factors,  $a_e$ , to a suitable reference (master) curve ( $S_{\text{ref}}$ ). From these physical ageing shift factors, we can define the ageing shift rate,  $\mu_e$ , as

$$\mu_e(T) = -\frac{d \log a_e}{d \log t_e} \quad (4)$$

where  $\mu_e$  is the ageing shift rate at temperature  $T$ , which is considered to be a material constant.<sup>15</sup> Once  $\mu_e$  has been determined, the ageing master curve constructed at a given reference age can be shifted to any other age (i.e. an age for which no experimental data are available) according to the equation

$$a_e = \left( \frac{t_{e,\text{ref}}}{t_e} \right)^{\mu_e(T)} \quad (5)$$

The approach above is referred to as time-ageing time superposition<sup>13,15,20</sup> and it can be combined with the TTS principle to predict the momentary response at a given ( $T$ ) using a reference curve defined at a reference temperature ( $T_{\text{ref}}$ ) as

$$S(t; t_e, T) = S_{\text{ref}}(a_e a_T t; t_{e,\text{ref}}, T_{\text{ref}}) \quad (6)$$

## Experimental procedures

### Material manufacturing

The material system investigated is a glass-fibre epoxy composite manufactured using FM906-27%-S2 glass-fibre epoxy prepreg from Cytec Engineered Materials. This prepreg has a nominal 27% resin content by mass and a 60% fibre content by volume (cured). The material is one of the constituents of heated GLARE together with aluminium sheets and copper heater elements. A 60 × 60 cm cross-ply laminate was manufactured by stacking UD prepreg layers to form a  $[0/90]_{4s}$  layup. The laminate was built up on a flat aluminium plate and covered by a thin release film and a flat 2 mm aluminium sheet to obtain a smooth surface on both sides. Vacuum is applied to the panel using a breather cloth and a vacuum bag covering the complete stack. The next step was to cure the material in the autoclave at a temperature of 180°C and a pressure of 6 bar for 1 h.<sup>1</sup> Figure 3 shows the vacuum, pressure, and temperature set points which are used for manufacturing of 180°C curing GLARE and the current cross-ply composite.

### Specimen configuration

Test specimens with a length of 250 mm and a width of 25 mm were then cut from the 2.0 mm thick manufactured laminated panels at an angle of  $\pm 45^\circ$  to the fibres using a water cooled diamond saw. This specimen size is in accordance to the ASTM Test Standard D3518/D3518M.<sup>14</sup> Dynamic mechanical analysis (DMA) at 1 Hz and 5°C/min showed a glass transition temperature  $T_g$  of 144°C defined as the temperature associated with the heat flow peak. To avoid slip in the test

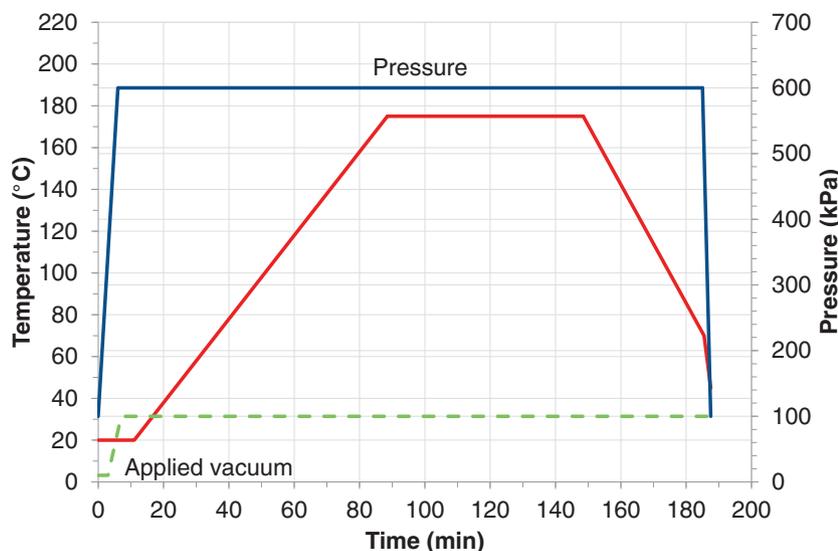


Figure 3. Autoclave cycle for FM906 glass-fibre epoxy as used in heated GLARE.

machine clamps, paper tabs were bonded to the specimen end surfaces. Two strain gauges, type KFG-5-120-C1-23 and bonded with CC-33A cyano-acrylate base adhesive (both by Kyowa), were used for strain measurement in both longitudinal and transverse direction at the centre of the specimen. The specimens were dried for at least 24 h at 40°C under vacuum prior to testing.

### Test setup

The in-plane shear creep response of the FM906 glass-fibre epoxy composite was determined by performing a tensile test on a  $\pm 45^\circ$  laminate in accordance with the shear test standard.<sup>14</sup> The test is performed on a 10 kN Zwick test machine equipped with a convection oven. The temperature was measured and monitored using a thermocouple at the centre section of the sample. For each test, with sequential ageing and creep for increasing test times, the temperature was kept constant ( $\pm 0.5^\circ\text{C}$ ) throughout the test. Prior to each test, the specimen was rejuvenated by heating it up above  $T_g$ , at 150°C, in a separate oven (Votch 0951).

### Creep testing

To ensure that all tests were performed within the linear viscoelastic range, a preliminary study was carried out to check that proportionality conditions and Boltzmann's superposition would be satisfied.<sup>25</sup> In this case, the  $\pm 45^\circ$  specimen was repeatedly rejuvenated (at 150°C), quenched and subjected to a sequence of creep and recovery at tensile stress levels ranging from 5 to 25 MPa with increments of 2.5 MPa at a test temperature of 80°C. The test sample was subjected

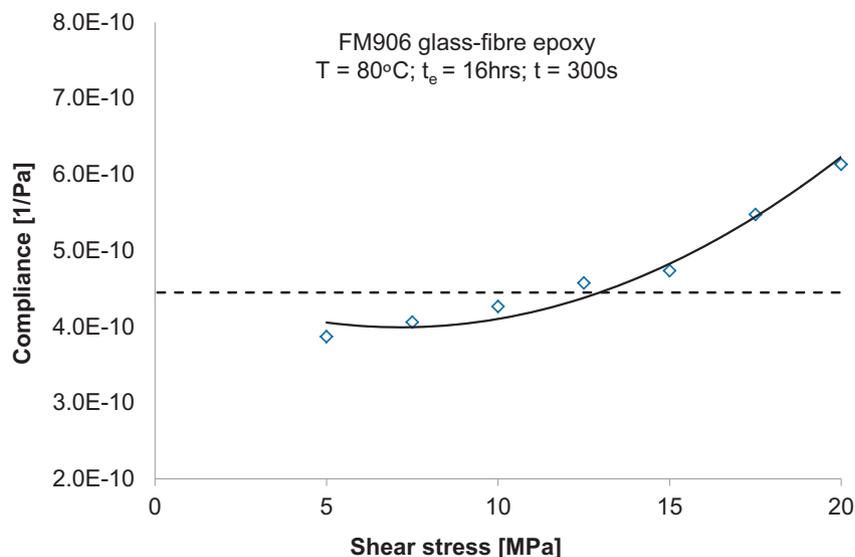
to 16 h of ageing prior to the sequence of 300 s tests. The linear–nonlinear threshold was taken as the compliance at the lowest in-plane shear stress of 5 MPa with a maximum variation of 15%. The isochronous curve of the FM906 glass-fibre epoxy composite and obtained threshold level (indicated as the dotted line) is shown in Figure 4. To facilitate the visualisation, a solid line is fitted through the data points.

For an applied load outside of the linear viscoelastic range of the material, the momentary compliance response will vary depending on the stress level. Based on this methodology, a tensile stress level of 10 MPa was chosen for this study, which is within the linear regime. The in-plane shear creep compliance response  $S$  of the  $\pm 45^\circ$  specimens is given by

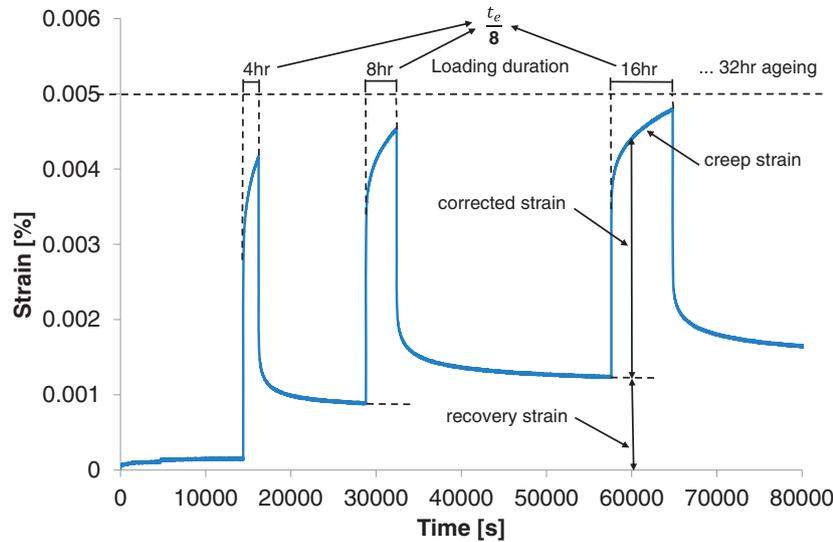
$$S = \frac{2A(e_x - e_y)}{P} \quad (7)$$

where  $P$  is the axial load applied on the specimen,  $A$  is the cross-sectional area of the specimen,  $e_x$  is the strain in the loading direction and  $e_y$  is the strain in the transverse direction.

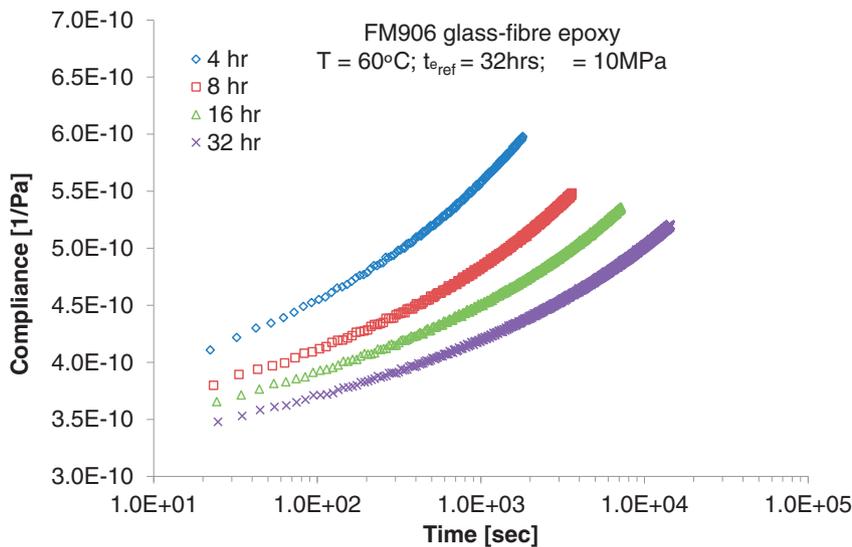
The creep test sequence for the determination of the effect of physical ageing on the creep response is illustrated in Figure 5. Prior to the test, the specimen was heated to 150°C and kept at this temperature for 15 min after which the specimen is rapidly quenched to the test temperature. The selected test temperatures were 60, 70, 75, 80, 85 and 90°C. Thus, all tests were carried out at sub- $T_g$  temperatures. These elevated temperature levels are expected to yield measurable changes in physical ageing within a lab time frame.



**Figure 4.** Determination of the linear viscoelastic range by means of multiple isochronous creep tests at 80°C.



**Figure 5.** Illustration of the creep test sequence for determining the effect of ageing.



**Figure 6.** Momentary creep compliance versus time for FM906 glass-fibre epoxy at 60 °C and 10 MPa shear load for 4, 8, 16, and 32 h of ageing.

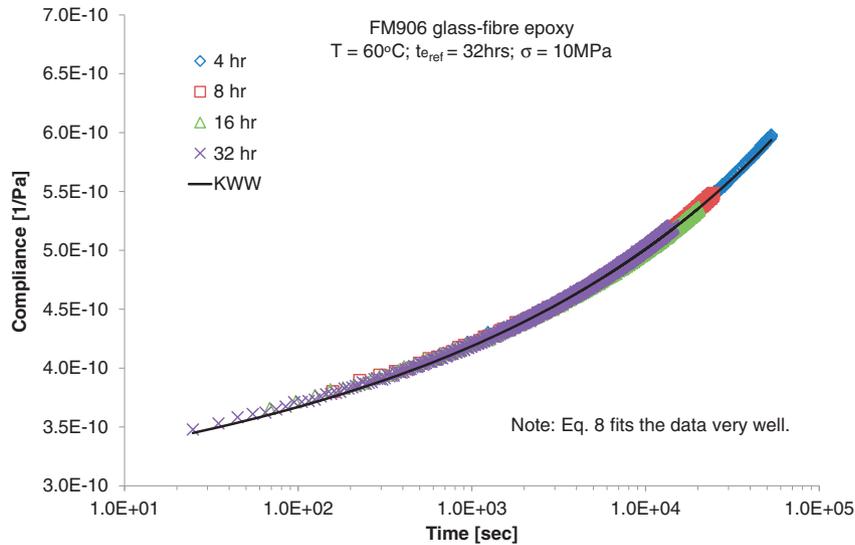
The physical ageing was defined to start immediately after the specimen reached the desirable test temperature. The physical ageing times selected for starting each creep segment were 4, 8, 16, and 32 h and the duration of each creep tests,  $t$ , was chosen to be  $1/8t_e$ , thus respecting the snapshot condition. After each creep sequence, the specimen was unloaded and allowed to recover until the start of the next creep test. A full recovery, however, is not achieved and the remaining strain is accounted for by subtracting the extrapolated recovery strain from the prior creep curve as illustrated in Figure 5.

## Results and discussion

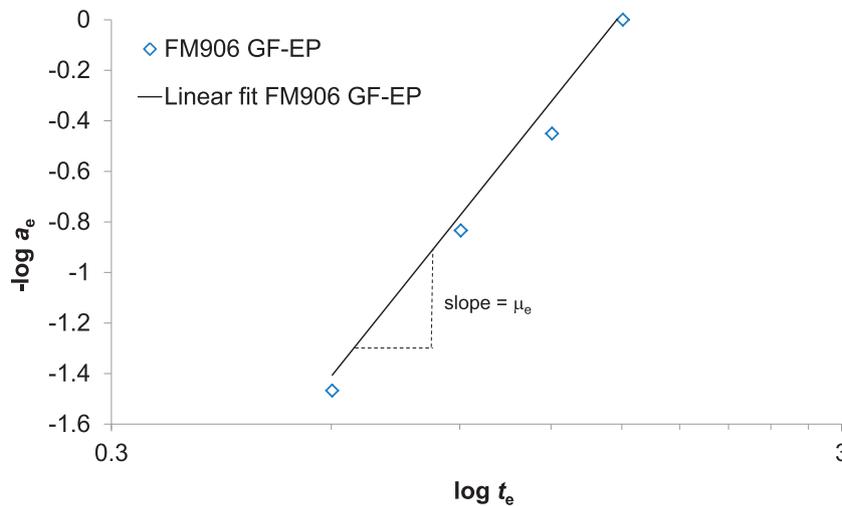
### Effect of ageing

In Figure 6, the momentary creep compliance versus time curve, as obtained from the tests, is shown for FM906 glass-fibre epoxy at 60 °C, 10 MPa in-plane shear load, and 4, 8, 16, and 32 h of ageing. Similar curves were obtained for the other test temperatures, 70, 75, 80, 85 and 90 °C.

Figure 6 confirms that physical ageing slows down the creep response by stiffening the polymeric resin. Physical ageing reduces the free volume and molecular



**Figure 7.** Master curve obtained by time–ageing time superposition of the momentary creep compliance curves at 60 °C and 10 MPa from Figure 6 as well as the KWW fit (full line based on equation (8)). The reference ageing time is 32 h.



**Figure 8.** The ageing shift factors obtained using time–ageing time superposition for FM906 glass-fibre epoxy at 60 °C and 32 h ageing reference curve.

mobility of the epoxy.<sup>12</sup> Increasing ageing times shift the creep curves towards the longer times and the compliance levels decrease. In Figure 7, the individual momentary creep curves obtained in Figure 6 were superposed to generate a single master curve for all data as well as the ageing shift factors. The 32 h curve was chosen as the reference ageing time. The ageing shift factor,  $a_e$ , versus the logarithmic ageing time,  $t_e$ , at 60 °C is shown in Figure 8.

A least square method was used to find the shift factors that result in a minimal mismatch in compliance of the reference and shifted creep curves. For convenience, the master curve can be approximated with a

three parameter fit model, depicted as a full line in Figure 7, known as the Kohlrausch-Williams-Watts (KWW) function<sup>27,28</sup>

$$S(t) = S_0 \left[ \exp\left(\frac{t}{\tau(t_e)}\right)^\beta \right] \quad (8)$$

where  $S(t)$  is the time-dependent creep compliance,  $t$  is the elapsed time after application of the load,  $\tau(t_e)$  is the physical ageing dependent retardation time, and  $\beta$  is the shape parameter. The value of  $S_0$  corresponds to the initial instantaneous elastic response  $S(t_0)$ .

The used KWW parameters to fit the superposed creep compliance curves of Figure 7 are given in Table 1.

The ageing shift factors of Figure 8 are fitted on a log–log scale yielding the (temperature dependent) ageing shift rate  $\mu_e$ . The ageing shift rate corresponds to the slope of the straight line (see also equation (4)) and amounts to 1.6 for 60°C.

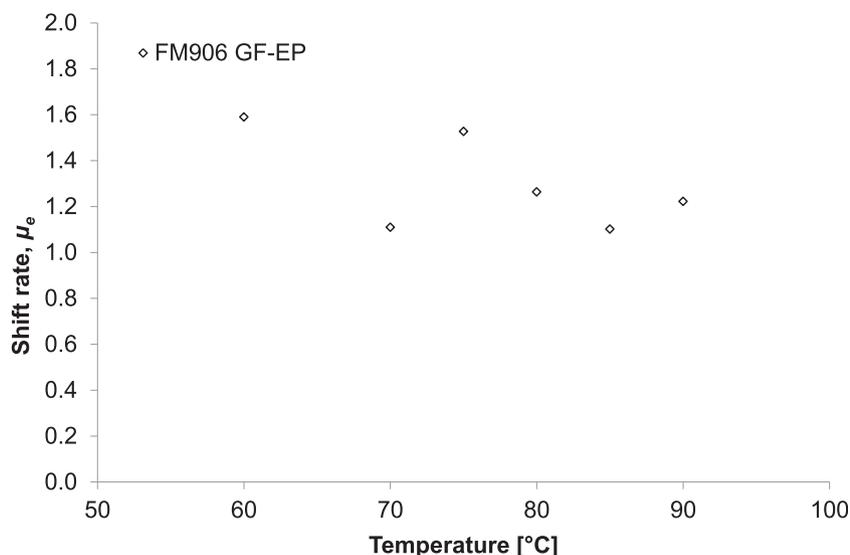
The time–ageing time superposition procedure has been repeated for all other test temperatures. Thus, the  $\mu_e$  as a function of temperatures are shown in Figure 9.

### Effect of temperature

For all tested temperatures, the KWW curves were seen to fit the data very well. The results of these fitted ageing master curves are shown in Figure 10. The reference ageing time here is 32 h and the in-plane shear load is 10 MPa. For clarity, only the curves for 60, 75 and 90°C are depicted in the graph. It can be seen that higher temperature increases the creep compliance at a given time, and thus accelerates the creep, as expected. By using the TTS principle, all the time–ageing time master curves for the different temperatures were collapsed into a single material master curve.

**Table 1.** Momentary curve parameters for FM906 glass-fibre epoxy at 60°C and 32 h ageing.

$\tau_e$	$S_0$	$\tau(s)$	$\beta$
[h]	[1/Pa]	[s]	[–]
32	$2.56 \times 10^{-10}$	$189.3 \times 10^3$	0.135



**Figure 9.** Ageing shift rate,  $\mu_e$ , as a function of temperature for FM906 glass-fibre epoxy.

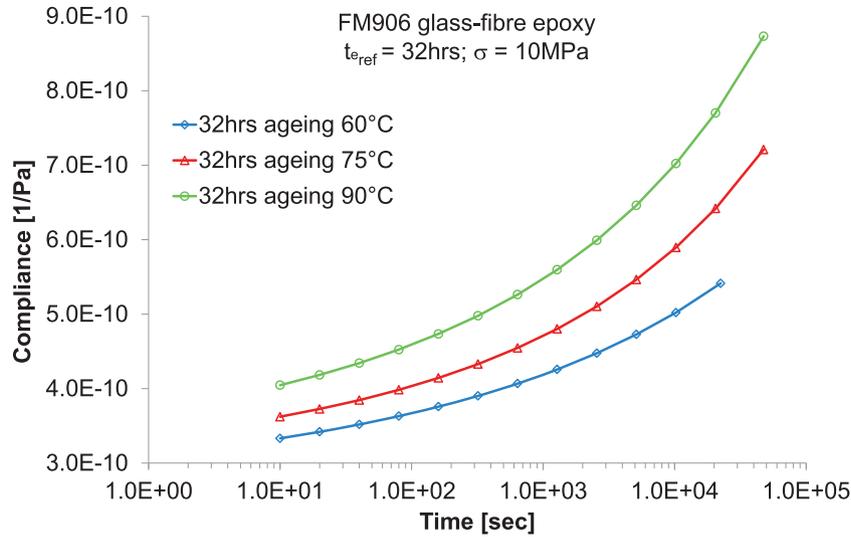
Figure 11 shows the collapsed experimental data after shifting the FM906 glass-fibre epoxy data for 32 h ageing at 75, and 90°C to the 60°C reference temperature. The determined shift factors are depicted in Figure 12 as a function of temperature. Since the temperature range was chosen below the  $T_g$ , the trend can be fitted using Arrhenius' law

$$\log(a_T) = \frac{E}{R} \left( \frac{1}{T} - \frac{1}{T_{\text{ref}}} \right) \quad (9)$$

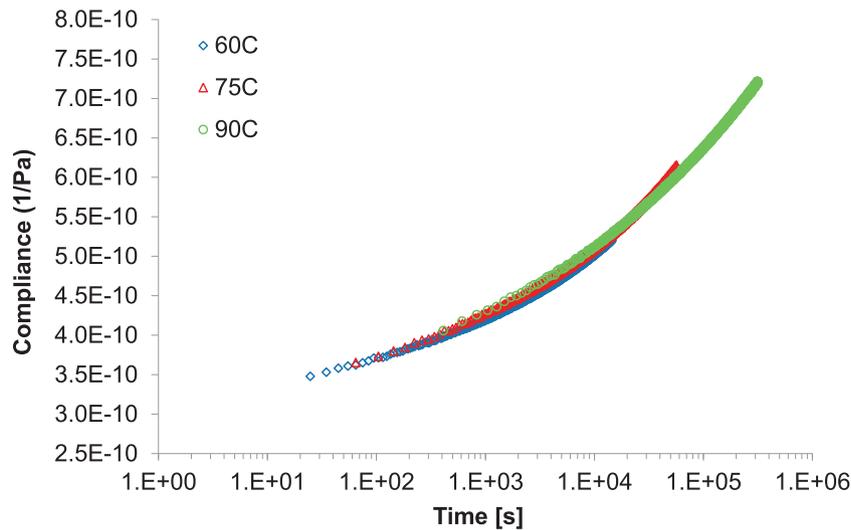
where  $E$  is the activation energy,  $R$  is the universal gas constant,  $T$  is the test temperature and  $T_{\text{ref}}$  is the reference temperature. An  $E/R$  of  $-5037.1$  was found using Matlab. Thus, as  $R$  is  $8.3144598 \text{ J mol}^{-1} \text{ K}^{-1}$ ,  $E$  is  $41.9 \text{ kJ mol}^{-1}$ . By using equations (2), (5), (8), and (9), it is now possible to model the creep behaviour of FM906 glass-fibre epoxy at each ageing time and temperature.

### Effect of long-term ageing

In previously derived momentary creep master curves, the Boltzmann's superposition principle was used. The principle is valid if the material properties do not change (by ageing or mechanical loading) during testing. Therefore, the test times of all performed tests were chosen eight times shorter than the prior ageing time to limit the effect of ageing during the test. In an aircraft deicing structure, the FM906 glass-fibre epoxy composite is repeatedly exposed to high temperatures over the service life of the aircraft. Thus, the exposure time can become much longer than the initial ageing time, which results in a long-term creep testing. During long-term



**Figure 10.** The effect of temperature on the creep behaviour. The shown curves are time–ageing time master curves obtained from shifting the curves from different ageing time and fitting a KWW function. The ageing time here is 32 h and the in-plane shear load is 10 MPa.



**Figure 11.** Temperature shifted experimental data curves of FM906 glass-fibre epoxy for 32 h ageing at 75 °C and 90 °C test temperatures to 60 °C.

creep experiments, the material slowly stiffens, which results in a slower development of creep deformation and a deviation from the momentary curve.

To account for the cumulative effects of ageing, the total test time  $t$  can be reduced to the effective time  $\lambda$  as given by Vreugd<sup>29</sup>

$$\lambda = \frac{t_e^0}{1 - \mu_e} \left[ \left( \frac{1+t}{t_e^0} \right)^{1-\mu_e} - 1 \right] \quad (10)$$

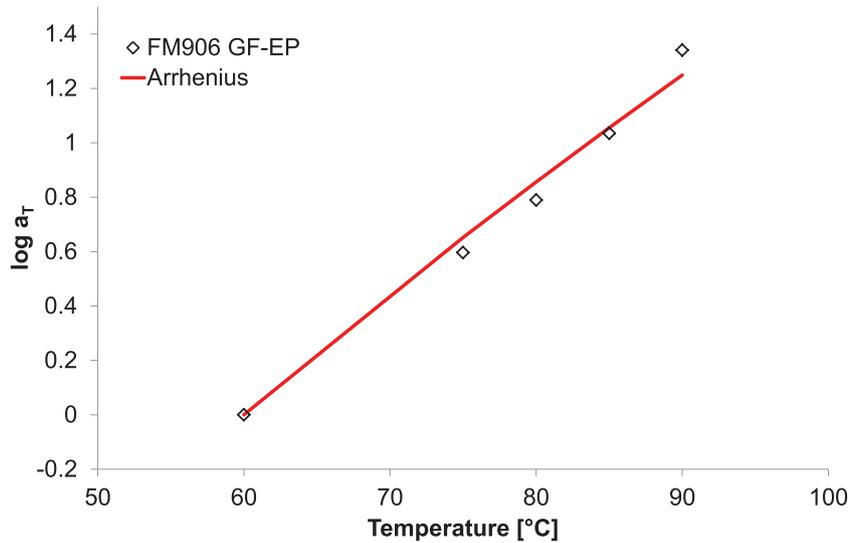
where  $t_e^0$  is the ageing time before the loading. By using this effective time instead of the elapsed test time, the

long creep compliance could be defined by

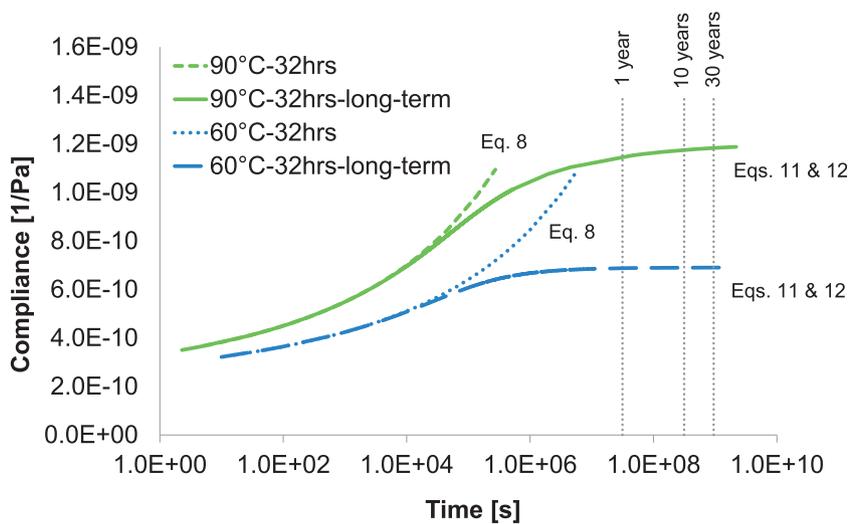
$$S(t) = S_0 \exp\left(\frac{\lambda}{\tau(t_e^0)}\right)^\beta \quad (11)$$

The relaxation time  $\tau$  calculated previously with Matlab for  $t_e = 32$  h (the reference curve from which the master curve was deduced) changed with the amount of ageing. The new  $\tau$  can be determined by

$$\tau(t_e^0) = \tau(t_{e,\text{ref}}) \left( \frac{t_e^0}{t_{e,\text{ref}}} \right)^{\mu_e} \quad (12)$$



**Figure 12.** Temperature shift factors and the fitted linear function using Arrhenius' law (full line). The reference temperature is 60 °C.



**Figure 13.** The predicted long-term creep behaviour of FM906 glass-fibre epoxy taking into account the effect of continuous ageing.

where  $t_{e,ref} = 32$  h. Thus, the prediction can be made for any ageing time. The shifting is taken into account in the equation. But to make the prediction at any temperature, the shift factor  $a_T$  is needed in addition. Finally, for a given temperature, the master curve shifting can be done and the long-term behaviour can be deduced, using the previous equation, for any specific ageing time. Figure 13 shows the predicted long-term creep behaviour of FM906 glass-fibre epoxy for 60°C and 90°C taking into account the effect of continuous ageing. The figure clearly shows that already after a couple of weeks the creep slows down considerably compared to what would be observed based on short-time measurements. This can be attributed to the

densification of the polymer and the associated decrease in molecular mobility. Similar curves can be found for the other test temperatures, but for clarity these curves are not depicted here.

Suppose a GLARE 5-3/2-0.3 laminate is loaded under 100 MPa in-plane shear in an aircraft deicing structure. Using classical laminate theory, this loading yields a 25 MPa in-plane shear loading in the glass-fibre epoxy layers.<sup>30</sup> After 10 years of continuous loading at an assumed deicing temperature of 90°C, this would result in a small creep shear strain of 0.03%, which is found by multiplying the compliance value of  $1.2 \times 10^{-9}$  found in Figure 13 times the 25 MPa in-plane shear load value.

## Conclusions

The linear viscoelastic creep behaviour of FM906 glass-fibre epoxy, as used in heated GLARE laminates, has been tested and modelled in master curves using both time-ageing time and TTS. Although the metal layers and glass-fibres in GLARE will minimise the effect of creep, shear and interlaminar shear modes can still cause creep behaviour in the epoxy. Continuous physical ageing slows down this process in the situation of long-term temperature and stress loading. The overall creep effect is thereby limited.

## Declaration of Conflicting Interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

## Funding

The author(s) disclosed receipt of the following financial support for the research, authorship, and/or publication of this article: This study is funded by the Dutch Technology Foundation STW and Fokker Aerostructures.

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## References

1. Vermeeren CAJR, Beumler TH, De Kanter JLCG, et al. Glare design aspects and philosophies. *Appl Compos Mater* 2003; 10: 257–276.
2. Airbus Industrie. Flight operations support. Cold weather operations, 2000.
3. Federal Aviation Administration (FAA). Airplane and engine certification requirements in supercooled large drop, mixed phase and ice crystal icing conditions. *Federal Regist* 2010; 75: 37311–37339.
4. Mohseni M and Amirfazli A. A novel electro-thermal anti-icing system for fiber-reinforced polymer composite airfoils. *Cold Regions Sci Technol* 2013; 87: 47–58.
5. Vlot A and Gunnink JW (eds). *Fibre metal laminates: an introduction*. Dordrecht, The Netherlands: Kluwer Academic Publishers, 2001.
6. Hagenbeek M. *Characterisation of fibre metal laminates under thermo-mechanical loadings*. PhD Thesis, TU Delft, Delft, Netherlands, 2005.
7. Pacchione M and Telgkamp J. Challenges of the metallic fuselage. In: *Proceedings of the 25th international congress of the aeronautical sciences (ICAS)*, Hamburg, Germany, 3–8 september 2006.
8. Müller B, Hagenbeek M and Sinke J. Thermal cycling of (heated) fibre metal laminates. *Compos Struct* 2016; 152: 106–116.
9. Hagenbeek M, Müller B and Sinke J. Effect of thermal cycling and ageing on heated fibre metal laminates and glass-fibre epoxy composites. *Adv Eng Mater* 2018. DOI: 10.1002/adem.201800084.
10. Pindera MJ, Williams TO and Macheret Y. Time-dependent response of aramid-epoxy-aluminium sheet ARALL laminates. *Polym Compos* 1989; 10: 328–336.
11. Daghigh V, Khalili SMR and Farsani RE. Creep behavior of basalt fiber-metal laminate composites. *Compos Part B* 2016; 91: 275–282.
12. Odegard GM and Bandyopadhyay A. Physical aging of epoxy polymers and their composites. *J Polym Sci Part B* 2011; 49: 1695–1716.
13. Ward IM and Hadley DW. Experimental studies of linear viscoelastic behavior as a function of frequency and temperature: time-temperature equivalence. In: IM Ward and J Sweeney (eds) *An introduction to the mechanical properties of solid polymers*. Hoboken, NJ: John Wiley & Sons Ltd, 2004, pp.95–120.
14. ASTM D3518. *Standard test method for in-plane shear response of polymer matrix composite materials by tensile test of a  $\pm 45^\circ$  laminate*. West Consohohocken: ASTM Standards, 2013.
15. Struik LCE. *Physical aging in amorphous polymers and other materials*, 1st ed. Amsterdam: Elsevier Science Ltd, 1978.
16. Sullivan JL. Creep and physical aging of composites. *Compos Sci Technol* 1990; 39: 207–232.
17. Gates TS, Veazie DR and Brinson LC. Creep and physical aging in a polymeric composite: comparison of tension and compression. *J Compos Mater* 1997; 31: 2478–2505.
18. Goertzen WK and Kessler MR. Creep behavior of carbon fiber/epoxy matrix composites. *Mater Sci Eng A* 2006; 421: 217–225.
19. Barbero EJ. Prediction of long-term creep of composites from doubly-shifted polymer creep data. *J Compos Mater* 2009; 43: 207–232.
20. Barbero EJ. Time temperature age superposition principle for predicting long-term response of linear viscoelastic materials. In: Guedes RM (ed.) *Creep and fatigue in polymer matrix composites*, 1st ed. Sawston, UK: Woodhead Publishing, 2011, pp.48–69Vol. 15.
21. Motta Dias MH, Jansen KMB, Luinge H, et al. The Influence of fiber-matrix adhesion on the linear viscoelastic creep behavior of CF/PPS composites. In: *1st international conference on ageing of materials and structures*, Delft, The Netherlands, 26–28 May 2014.
22. Motta Dias MH, Jansen KMB, Luinge JW, et al. Effect of fiber-matrix adhesion on the creep behavior of CF/PPS composites: temperature and physical aging characterization. *Mech Time Depend Mater* 2016; 20: 245–262.
23. Jansen KMB. *Thermomechanical modeling and characterization of polymers. Modelling of thermoviscoelasticity and non-linearities* [Course book]. 1st ed. Netherlands: Delft University of Technology, pp.81–98.
24. Hutchinson JM. Physical aging of polymers. *Prog Polym Sci* 1995; 20: 703–760.
25. Papanicolaou GC and Zaoutos SP. Viscoelastic constitutive modeling of creep and stress relaxation in polymers and polymer matrix composites. In: Guedes RM (ed.) *Creep and fatigue in polymer matrix composites*, 1st ed. Cambridge: Woodhead Publishing, 2011, pp.1–47.

26. Tomlins PE and Read BE. Creep and physical ageing of polypropylene: a comparison of models. *Polymer* 1998; 39: 355–367.
27. Kohlrausch R. Theorie des elektrischen Rckstandes in der Leidener Flasche. *Ann Phys* 1854; 167: 179–214.
28. Williams G and Watt DC. Non-symmetrical dielectric relaxation behaviour arising from a simple empirical decay function. *Trans Faraday Soc* 1969; 66: 80–85.
29. Vreugd J de. *The effect of aging on molding compound properties*. Doctoral Thesis, Delft University of Technology, Delft, Netherlands, 2011.
30. Hagenbeek M. *Estimation tool for basic material properties (ETMP)*. Technical Report B2v-00-29. Delft, The Netherlands: Delft Faculty of Aerospace Engineering, University of Technology, 2000.