MŰEGYETEM 1782

BUDAPEST UNIVERSITY OF TECHNOLOGY AND ECONOMICS
FACULTY OF MECHANICAL ENGINEERING
DEPARTMENT OF POLYMER ENGINEERING

THESES

ANALYSIS OF LONG TERM CREEP BEHAVIOR OF POLYMERS AND POLYMER COMPOSITES

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BUDAPEST
2013.

The review of this PhD dissertation and the protocols of the defense
can be found at the Dean’s Office, Faculty of Mechanical Engineering,
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1. Introduction

Polymers and polymer composite structural materials are used in a wider and wider range in different industrial applications. The mechanical properties of polymers depend on different environmental variables such as temperature, humidity and what is more important, time besides their structure. As opposed to other materials, for instance metals thermoplastic polymers exhibit significant creeping and stress relaxation even at low temperatures and that makes the job of the designer difficult. Creeping is the time dependent, continuously increasing strain response of polymer materials to stress load constant in time.

Polymer and polymer composite parts are usually designed for maximum allowed deformation and not for load dependent failure, i.e. breakage or fraction due to stress peak. Creeping occurs due to long term loading at a stress level lower than tensile strength and yield point, and increases as the melting point is approached, i.e. temperature increases. The life cycle of parts exposed to constant or repetitive loading may end not only with actual failure but with function loss due to increasing deformation, in case dimensional stability is important regarding its purpose. If the part is exposed to an impact that reaches or approaches maximum allowed deformation, it is likely to fulfill its task in a reliable way for a long time. However, if this kind of loading is long term an adequately large safety coefficient has to be used (material surplus has to be designed) due to the scarcity and unreliably accurate information on the long term behavior of the material. For instance, a fan blade operating at high speed and maybe high temperature deforms, creeps. The gap between fan casing and blade edge may disappear due to blade deformation, and hence friction appears between the blade and the wall resulting in a burnt down motor or blade breakage. This phenomenon can be prevented if the blades are changed after a certain number of hours run. Precise design requires failure details and information that forecast failure.

Measurement of creeping behavior for single materials and mapping the process helps the designer reduce the safety coefficient and this way material surplus as the required quantity of information is available for him/her. The Hungarian standard on the determination of creeping behavior of polymers (MSZ EN ISO 899) is composed of two parts: the first part deals with specimens, measurement environment and methods of creeping phenomena due to tensile stress, while the second one details the same data on flexural stress type creeping. Although the equipment, specimens and important measurement parameters of creep tests are determined in standards, there is very few data available on the long term behavior of thermoplastic polymers, thermosetting resins and composites since these measurements are much more time consuming and expensive than conventional mechanical tests.

The measurement of long term behavior of parts would also delay their introduction on the market, therefore as a response to the requirements of industry creeping behavior is estimated from master curves that use the principle of similar impacts or from basic material models based on short term creep tests.

2. Analysis of literature, aim of dissertation

In the literature creeping behavior is usually described on the one hand with systems with three, four or more parameters composed of idealized material models – such as ideally elastic element following Hooke's law or ideally viscose element following Newton's law – based on material properties measured with short term creep tests; while on the other hand with master curve construction processes based on the principle of similar impacts (temperature-time, load-time, etc.). Viscoelastic material behavior can also be described with the Sherby-Dorn and Larson-Miller approximations. Master curves based on similar impacts can be constructed using Arrhenius function, William-Landel-Ferry equation and methods that combine those with offset along the logarithmic time axis ($\lg a_T$) or using ordinate offset factor ($\lg b_T$) that takes the temperature dependence of density into consideration.

Long term creep estimations based on master curves and heuristic relations are only valid for the given small loads, and do not provide information on failure deformation or life span therefore can only help designers to a limited extent in dimensioning a given product to its whole life span knowing the forces and environment and in estimating the expected failure time of the part.

According to the applied techno-climate aspect while the part lives its life during its production and usage, it collects the impacts it is exposed to. During tensile tests the specimen lives its life in an accelerated way, i.e. collects and stores the impacts on it. It is a reasonable assumption that this measurement provides a lot of information on the different loading methods related to the material and mechanical behavior, and what is more, long term behavior can be estimated from the results of short term examinations using adequate transformations. The applicability of the method developed by Vas and Nagy was checked on deformation driven tensile and very short term (20-30 minutes) stress relaxation tests of polypropylene samples. However, the relevant loading level dependent variable transformations and their parameter determination processes are not worked out. The method necessary for the determination of statistical characteristics (distribution, variation, confidence interval, quantiles etc.) required by the design of failure during long term loading was also not developed.

The more and more complex simulation processes and methods provide an increasing number of possibilities for designers from the design of products, tools and production and checking those with simulations to material behavior modeling. Sooner or later designers will require the life span estimation of products exposed to higher loading or used under extreme circumstances using a simulation process based on a real, simple but reliably described material behavior.

The aim of the present dissertation is the wide range experimental and statistical analysis regarding force driven tensile and creeping behavior of the method – developed by Vas and Nagy [1, 2] – that estimates the long term mechanical behavior and expected failure in case of constant loading based on short term measurements and the nonlinear transform ($T=(T_1,T_2)$) of the so called linear viscoelastic (LVE) difference curve and the execution of theoretical and experimental tasks necessary for practical applicability. Determination of the most favorable nonlinear variable transformations that are parts of the method and making them more accurate using tensile tests and mid-term creep tests carried out on a large number of specimens. Development of the statistical estimation method of creep failure. Determination of the applicability of long term three point flexural tests on creeping.

Aimed tasks:

- Carry out a large number of tensile tests and short term (lasting for several hours) tensile creep tests at different load levels, at room temperature on unreinforced, thermoplastic PP and glass fiber reinforced PP composite specimens with different fiber content.
- Analysis of measurements:
    - Analysis of the relation and their description using mathematical formulas of average tensile strength characteristics such as tensile strain and tensile strength as well as initial modulus of elasticity and fiber content.
    - Calculation of average creep failure strain from LVE difference curves, and the statistical, probability distribution based description of LVE estimations from the end points of these curves and the determination of estimated strain limit as a function of fiber content.
    - Statistical based approach of average smoothed room temperature tensile curves at given fiber contents and the determination of creep failure strain increase as a function of the initial strain values belonging to the uploading level.

  - In case of an average creep curve determination of transformation T_1 and the parameters of the first LVE approximation (LVE difference curve), approximation of the estimated creep failure strain as a function of fiber content, investigation of the creep failure strain distribution function, and estimation of statistical limit curves (quantiles, confidence interval).
  - Determination of time transformation function T_2 – needed for the estimation of long term creep curves – and its parameters based on creep failure strain estimated with transformation T_1 and short term creep tests as well as the estimation of the average value of creep failure time (life span) as a function of creep loading level.
- Carry out pre-controlled three-point flexural tests and flexural stress short term creep tests on the above mentioned materials at room temperature, and the comparison of results with those of tensile tests. My other aim is to prove the applicability of the method worked out for the description of tensile stress creep behavior for the estimation of flexural stress creep properties.
- Determination of master curves that estimate long term creep behavior based on tensile and flexural creep tests carried out on a tensile tester and on DMA equipment at different load levels and different temperatures as well as master curve based creep life span estimation using failure information estimated from short term measurements.

3. Review of applied materials and methods

The matrix material of unreinforced and glass fiber reinforced thermoplastic injection molded composite specimens was isotactic polypropylene homopolymer type H 949A produced by Tiszai Vegyi Kombinát recommended as the base material of thin-wall packing boxes, household, kitchen, medical equipment and toys. As reinforcement cut glass fiber surface treated for PP, type SV EC 13 473 produced in the Czech factory of Johns Manville in 2005 was used. In order to enhance interaction between the matrix and the reinforcing fibers, polypropylene grafted with maleic acid anhydride Orevac CA100 (Arkema, France) in the quantity of 2 weight% of glass fiber content was added to the extruded 5, 10, 20, 30 and 40 m% fiber content injection molding granulate produced in a Brabender Plasti-Corder (USA) computer controlled extruder at zone temperatures of 190-210-210-230°C. In order to prevent different thermal history unreinforced PP granulate used for injection molding was also extruded, and then ground on SB Plastics Machinery (Italy) equipment.

1A type specimens were injection molded according to standard ISO 527-2 on an Arburg Allrounder 320C 500-170 machine using the following settings: injection molding volume (44 cm^3), switching over point (12 cm^3) and injection rate (50 cm^3/sec) was the same in case of all filling contents, injection molding pressure (700–1000 bar) and holding pressure (500–700 bar) was increased as a function of glass fiber content if it was necessary. The circumferential speed of the screw (15 m/min), the temperature of the tempered mold (40°C) and the temperature settings of the zones (175-180-185-190-195°C) were not changed during production.

Force driven tensile and flexural tests were carried out on the unreinforced and composite specimens produced. Furthermore, creep tests were also executed at different load levels at room temperature, and at the same load level but at different temperatures using a universal tensile tester and a dynamic mechanical analyzer (DMA). Master curves were determined based on the temperature-time, loading-time superposition from the creep test results using the principle of similar impacts.

Force driven tensile and three-point flexural tests provide the basic information for the creep estimation method developed. The creep curve that can be characterized with the same uploading time – if uploading time is known – separates from the approximating curves at time point t_0 (Figure 1). A kind of derivative of the tensile curve between uploading time t_0 and failure time t_{2B} provides the first linear viscoelastic (LVE) estimation of the creep curve. Its T_1 transform generates the second LVE estimation of the creep curve that can be interpreted as a compact creep curve and with the help of it creep failure strain can be estimated. Transform T_2 estimates the long term deformation and failure behavior of the creep curve based on the prolongation of the second LVE estimation in time.

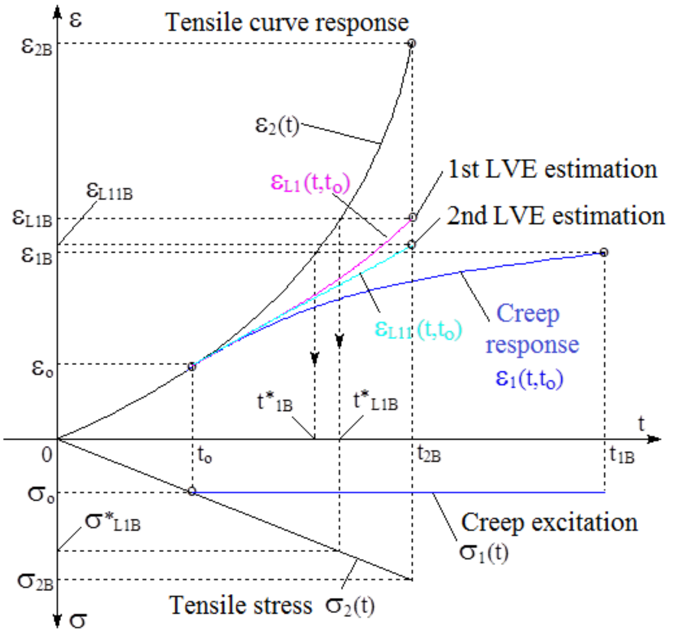

Figure 1 Presentation of the principle of the creep estimation method applied

If the statistical characteristics of the creep failure strain such as deviation interval, lower and upper quantiles, confidence interval are determined for any creep loading (Figure 2) the creep curve and the lower and upper estimation of the expected value of creep failure strain can be generated.

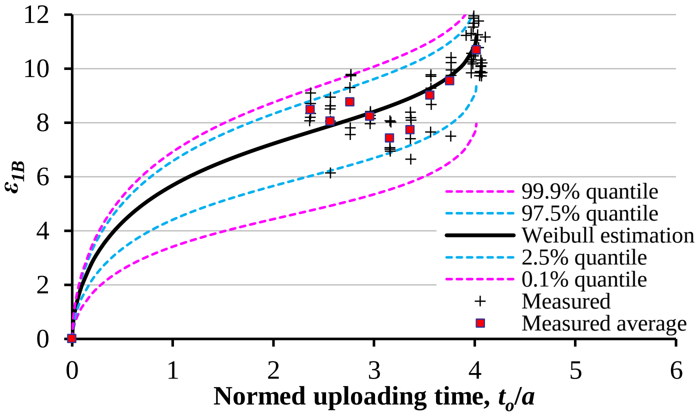

Figure 2 Quantile, average and confidence interval curves estimated based on the short term creep measurement of creep failure strain and Weibull distribution

With the help of the creep estimation method developed the estimated average creep failure time (average of t_{1B}), i.e. average life span is obtained as the intersection point of estimated creep failure strain (based on the determined second LVE estimation) and the master curve generated according to an already proven similarity principle (temperature-time or load level-time) (Figure 3).

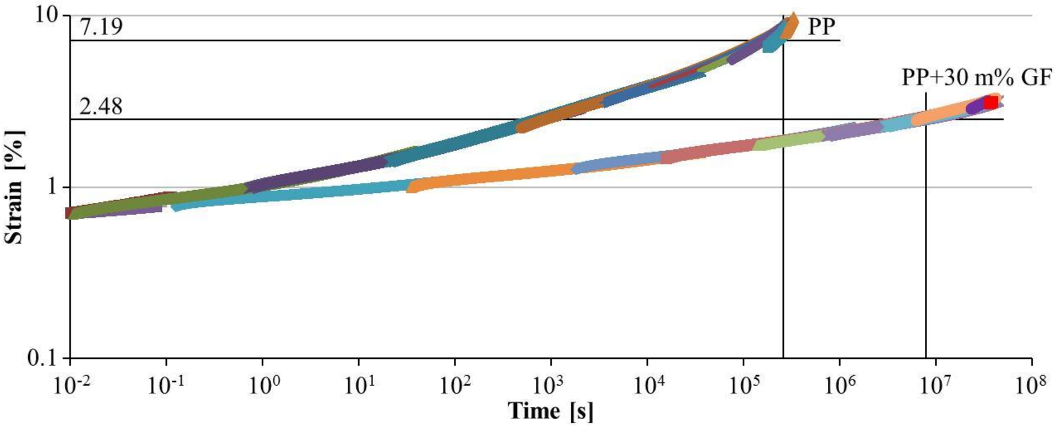

Figure 3 Determination of creep life span as the intersection point of estimated creep failure strain and the master curves (Loading: 50% of tensile strength, Temperature: 23±1°C)

It has also become possible to estimate long term creep curve and creep life span without master curves with the determination of transformation function T_2 and its parameters – that could be identified in a logarithmic-exponential form (Figure 4).

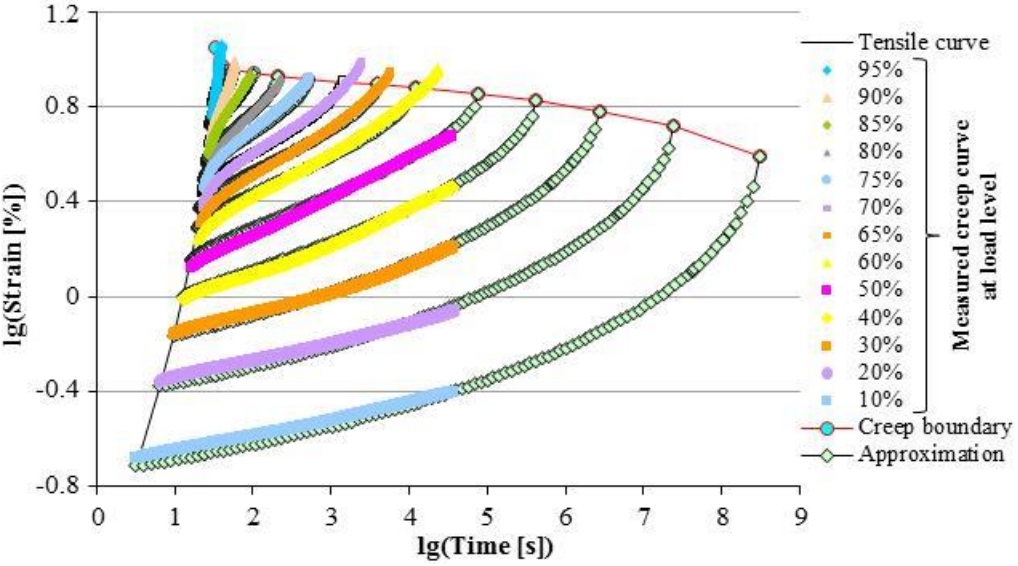

Figure 4 Estimation of long term creep curves and failure boundary curve based on transformation T_2 and short term creep curves

Meanwhile unreal creep life span forecasts were obtained (according to the relation of average life span and loading exponential model) that do not take aging into consideration but coincide with the life span determined as the intersection point of master curves well (Figure 5). Polymer materials age, degrade even without mechanical loading, and for instance the life span of PP is around 100 years according to experience [3, 4]. Modified estimation (logistic model) that takes physical and chemical aging into consideration provides real creep life span estimations.

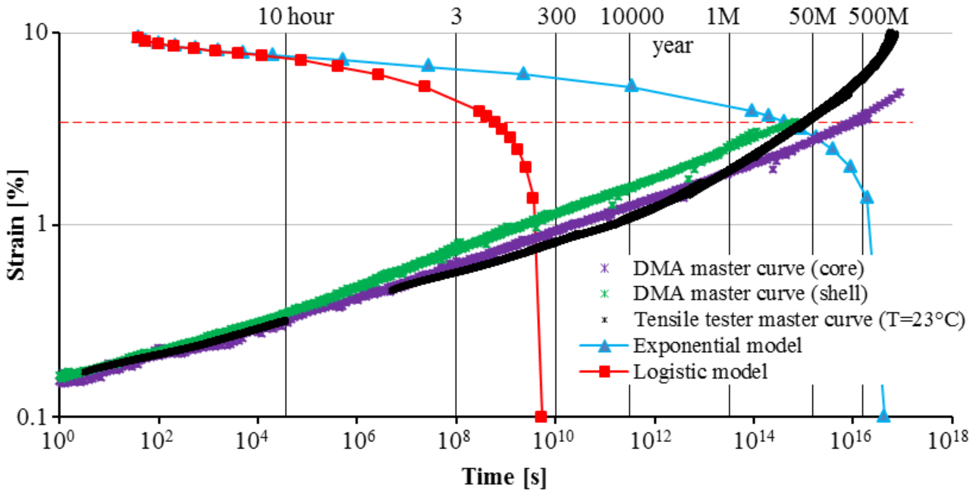

Figure 5 Presentation of master curves and creep life span forecasts (exponential and logistic model)

A practical significance of the process developed is that estimated average life span can also be determined as the time coordinate of the intersection point of master curves determined with a different method – provided that load and measurement method is known – and the average creep failure strain level. Failure statistics (such as deviation, confidence interval average value, as well as quantiles) determined for creep failure strain and life span enhance its applicability even further. A supplementary program module based on the process developed embedded into a three dimensional e.g. a finite element design program would make it possible to estimate, what is more to design failure strain and life span already in the design phase if the applied load and the workshop circumstances are known. The results achieved are summarized in the theses defined in 7 points.

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4. Theses

Thesis 1 ***Relation of average breaking strength characteristics and fiber content***

I have revealed that in case of tensile strength excitation tensile tests of constant rate carried out at room temperature the average failure strain ($\bar{\varepsilon}_{2B}$) of the examined unreinforced and glass fiber reinforced PP materials decreases exponentially while average breaking strength ($\bar{\sigma}_{2B}$) increases following the logistic curve in the examined range of fiber content weight percentage: $0 \le \varphi \le 40$ m% :

$$\bar{\varepsilon}_{2B}(\varphi) = \varepsilon_{2B0} + \varepsilon_{2B1} \cdot e^{-\frac{\varphi}{b_1}} + \varepsilon_{2B2} \cdot e^{-\frac{\varphi}{b_2}} \tag{1}$$

$$\bar{\sigma}_B(\varphi) = \frac{\dot{\sigma}_0 \cdot t_\infty}{1 + (t_\infty / t_0 - 1) \cdot e^{-\varphi/\varphi_o}} \tag{2}$$

where $\varphi = \varphi_f$ [m%] is the weight percent of fiber content, while $\varepsilon_0 = 2.8\%$, $\varepsilon_{01} = 6.367\%$, $\varepsilon_{02} = 2\%$, $\varphi_1 = 2\ m\%$, and $\varphi_2 = 18.65\ m\%$ are constants fitted with the least squares method. In case of the materials examined the values of parameters obtained from regression fitting are the following based on logistic fitting: $\dot{\sigma}_0$ = 1.25 MPa/s, t_0 =32.1 s, φ_o=15.773 m%, and t_∞=93 s fitted constants and R^2=0.997.

I have also revealed that in case of tensile strength excitation tensile tests of the above mentioned materials average breaking deflection ($\bar{f}_{2B}$) and average breaking force ($\bar{F}_{2B}$) can also be described with equation (1) and (2), respectively. In case of flexure, the regression parameters for (1) are: f_∞=3.8 mm; f_{01}=5.8 mm; f_{02}=6.0 mm; φ_1=3.4 m%; φ_2=30 m% and the coefficient of fitting is R^2=99.83%, and for (2) $\dot{F}_0$ =20 N/s, t_∞=15.4 s; t_0=2.2 s; φ_o=15.15 m%, where the coefficient of fitting is R^2=99.63%

Publications related to this thesis are: [5-7, 9]

Thesis 2 ***Weibull based description of the LVE estimation of average creep failure strain***

I have revealed that the LVE estimation of average creep failure strain defined by the end points of the LVE difference curve calculated from the average breakage curve can be approximated with the following, Weibull distribution based formula in the examined range of fiber content at any strain loading (t_o):

$$\bar{\varepsilon}_{L1B}(t_o) = E(\varepsilon_{L1B}(t_o)) = \varepsilon_{L1B\infty}\left(1-e^{-\left(\frac{t_o}{a}\right)^k}\right), \quad 0\le t_o \le \bar{t}_{2B} \tag{3}$$

where $\bar{t}_{2B}$ is the average breaking time. The relation of asymptotic boundary strain ($\varepsilon_{1B\infty}$), scale (a), and modulus (k) parameters and fiber content can be described with the following formulae ($0\le\varphi\le40$ m%):

$$\varepsilon_{1B\infty}(\varphi) = \varepsilon_o + \varepsilon_1 e^{-\frac{\varphi}{b_1}} + \varepsilon_2 e^{-\frac{\varphi}{b_2}} \tag{4}$$

$$a(\varphi) = a_\infty e^{-be^{-c\varphi}} \tag{5}$$

$$k(\varphi) = k_o + (k_\infty - k_o)\left(1-e^{-c\varphi}\right) = k_\infty + (k - k_o)e^{-c\varphi} \tag{6}$$

where in case of the examined materials ε_0=7.2%; ε_1=3.9%; ε_2=1.4%; b_1=1.5 m%; b_2=20 m%; R^2=0.9545; a_∞=189 s; b=3.15; c=0.125/m%; R^2=0.9948; k_o=0.5875; k_∞=0.718; c=0.2/m%; R^2=0.714 are the fitted constants and the goodness of the fitting.
Publications related to this thesis are: [10, 11]

Thesis 3 *Weibull based description of average breaking curves*

I have revealed that average strain response (breakage curve) $\bar{\varepsilon}_2(t)$ of the examined unreinforced and glass fiber reinforced PP to constant rate tensile force excitation at room temperature can be described with the following Weibull based formula in the [0, $\bar{t}_{2B}$] time interval from unloaded state until breakage:

$$\bar{\varepsilon}_2(t) = \bar{\varepsilon}_{2B} - \varepsilon_{1B\infty}\left(1-e^{-\left(\frac{\bar{t}_{2B}-t}{a}\right)^k}\right), \quad 0\le t\le t_{2C} \tag{7}$$

where $\varepsilon_{1B\infty} > \bar{\varepsilon}_{2B}$ is the asymptotic boundary strain, *a* is the time constant (Weibull scale parameter) and *k* is the Weibull modulus parameter, the value of which are defined by (4)-(6) in the range of $0\le\varphi\le40$ m%.

I have revealed that based on (7) the first LVE estimation of average creep curve and the strain increment until creep breakage can be given with the following formulae:

$$\bar{\varepsilon}_{L1}(t,t_o) = \varepsilon_{1B\infty}\left(e^{-\left(\frac{\bar{t}_{2B}-t}{a}\right)^k} - e^{-\left(\frac{\bar{t}_{2B}-t+t_o}{a}\right)^k}\right), \quad 0\le t,t_o\le \bar{t}_{2B} \tag{8}$$

$$\bar{\varepsilon}_{1B}(t_o) - \varepsilon_o = \varepsilon_{1B\infty}\left(1 - e^{-\left(\frac{t_o}{a}\right)^k} + e^{-\left(\frac{\bar{t}_{2B}}{a}\right)^k} - e^{-\left(\frac{\bar{t}_{2B}-t_o}{a}\right)^k}\right), \quad 0 \le t_o \le \bar{t}_{2B} \qquad (9)$$

Publications related to this thesis are: [10, 11]

Thesis 4 *T_1 transformation of the average creep curve and creep breakage strain*

I have revealed that in case of the examined unreinforced and glass fiber reinforced PP the first LVE approximation ($\bar{\varepsilon}_{L1B}(t_o)$) of the measurable average creep breakage strain ($\bar{\varepsilon}_{1B}(t_o)$) can be estimated with its T_1 linear transform without long term creep measurements, in case of any creep loading (t_o) in the fiber content range of $0 \le \varphi \le 40\%$:

$$\bar{\varepsilon}_{1B}(t_0) \approx \bar{\varepsilon}_{L11B}(t_0) = (1-c)\varepsilon_0 + c\varepsilon_{L1B\infty}\left(1 - e^{-\left(\frac{t_0}{a}\right)^k}\right) \qquad (10)$$

where $\varepsilon_o = \bar{\varepsilon}_2(t_o)$ is the initial strain loading, $\varepsilon_{L1B\infty}$ is the asymptotic strain boundary and c is the linear transformation parameter. The relation of value c and fiber content is:

$$c = c_o + \frac{c_1\varphi^n}{1 + b\varphi^n} \qquad (11)$$

where c_o=0.7; c_1=0.0243 (m%)$^{-n}$; b=0.0310 (m%)$^{-n}$; n=3/2=1.5 and R^2=2.48%.

The second LVE estimation ($\bar{\varepsilon}_{L11}(t,t_o)$) of the average creep curve ($\bar{\varepsilon}_1(t,t_o)$) is transform T_1 of the first estimation ($\bar{\varepsilon}_{L1}(t,t_o)$) the end point of which is the estimation of the average breakage strain (10):

$$\bar{\varepsilon}_{L11}(t,t_o) = T_1\left(\bar{\varepsilon}_{L1}(t,t_o)\right) = (1-c)\varepsilon_o + c\bar{\varepsilon}_{L1}(t,t_o) \qquad (12)$$

I have also revealed that curve (10) has maximum if c>1, i.e. creep breakage strain can be larger than breakage strain itself and the place of maximum shifts to the half value of the breakage strain if c values increase, while in case of 0<c≤1 the curve is strictly monotonously increasing.

Publications related to this thesis are: [10, 11]

Thesis 5 ***Statistical characteristics of creep breakage strain***

I have also revealed that the first LVE estimation of the creep breakage strain can be obtained in the examined fiber content range using the Weibull distribution below ($z>0$ is the variable of the distribution function):

$$Q_{\varepsilon_{L1B}}(z,t_o)=P\left(\varepsilon_{L1B}(t_o)<z\right)=1-e^{-\left(\frac{z}{z_o}\right)^{\beta}} \tag{13}$$

where scale parameter z_o that depends on creep loading is determined by the expected value:

$$z_o(t_o)=\frac{E\left(\varepsilon_{L1B}(t_o)\right)}{\Gamma(1+1/\beta)}=\frac{\varepsilon_{L1B\infty}}{\Gamma(1+1/\beta)}\left(1-e^{-(t_o/a)^k}\right) \tag{14}$$

while modulus parameter a β is independent from creep loading, only depends on fiber content and can be obtained as the inverse of the term below:

$$V_{L1B}=V\left(\varepsilon_{L1B}(t_o)\right)=\sqrt{\frac{\Gamma(1+2/\beta)}{\Gamma^2(1+1/\beta)}-1} \tag{15}$$

where V_{L1B} is the relative strain of ε_{L1B} that depends on fiber content in the way described below:

$$V_{L1B}=v_o+(v_1-v_o)e^{-v_2\varphi} \tag{16}$$

where v_o=0.028; v_1=0.1007; v_2=0.37/m% and R^2= 2.57%.

I have also revealed that the distribution function of measurable creep breakage strain can be described with a three parameter Weibull distribution ($z\geq(1-c)\varepsilon_o$ is the variable of the distribution function) obtained using transformation T_1 of the second $\varepsilon_{L11B}(t_0)$ LVE estimation:

$$Q_{\varepsilon_{1B}}(z)=P\left(\varepsilon_{1B}(t_0)<z\right)\approx P\left(\varepsilon_{L11B}(t_0)<z\right)=1-e^{-\left(\frac{z-(1-c)\cdot\varepsilon_0}{c\cdot z_0(t_o)}\right)^{\beta}} \tag{17}$$

I have revealed that based on three parameter Weibull distribution (17) deviation interval, lower (0.1%) and upper (99.9%) quantile boundary curves, and confidence interval boundary curves can be constructed for the average value at a given probability level for the creep breakage strain value and using those the long term deformation behavior and failure of a given product can be designed.

Publications related to this thesis are: [10, 11]

Thesis 6 *Determination of creep life span*

I have revealed that transformation T_2 that maps tensile test time values to the adequate creep time value can be realized with the following invertible, logarithmic-exponential transformation function (h):

$$t_2 = h(t_1) = t_o + (\bar{t}_{2B} - t_o)\left[\frac{\lg(t_1/t_o)}{\lg(\bar{t}_{1B}/t_o)}\right]^{\gamma}$$
$$t_1 = h^{-1}(t_2) = t_o\left[\frac{\bar{t}_{1B}}{t_o}\right]^{\left(\frac{t_2 - t_o}{\bar{t}_{2B} - t_o}\right)^{1/\gamma}} \tag{18}$$

where $\bar{t}_{1B}$ and $\gamma>0$ are the average creep life span and coefficient, respectively. The relation of average life span $\bar{t}_{1B}$ and uploading time t_o can be approximated with the following logistic curve:

$$\bar{t}_{1B} = \bar{t}_{2B}\,\frac{(1+\delta)e^{\left(\frac{\bar{t}_{2B}-t_o}{t_D}\right)^{d_1}}}{1+\delta e^{\left(\frac{\bar{t}_{2B}-t_o}{t_D}\right)^{d_2}}} \tag{19}$$

where $0<t_D$ [s] is the scale parameter, $0<\delta<1$ is the logistic impact parameter and $1<d_2<d_1$ is true for the exponents.

I have revealed that creep life span can be estimated with intersection point of the creep master curve determined at a given strain load or the estimation of the second LVE estimation of the creep curve produced with nonlinear time transformation T_2, and the average value of creep breakage strain estimated with (10) as the boundary level of creep strain and using the boundaries of the creep breakage strain confidence interval in a similar way lower and upper estimation can be made.

Thesis 7 *Average tensile strength and tensile creep characteristics*

I have revealed that the results of three point flexural tests and flexural type creep tests carried out with force excitation can be handled with theoretical and practical methods similar to those worked out for tensile loading regarding the average behavior, and this way the expected failure characteristics can be estimated without any significant change, in a similar way.

Publications related to this thesis are: [6, 8]

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