

## Frequency dependence of the self-heating effect in polymer-based composites

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### Materials

#### ABSTRACT

**Purpose:** The self-heating effect caused by viscous energy dissipation in polymer-based composite structures subjected to harmonic loads is considered to have a great influence on the residual life of the component. The purpose of the conducted investigations is the determination of the dynamic mechanical behaviour of a polymer-based composite material under different excitation frequencies and temperatures.

**Design/methodology/approach:** The dynamic mechanical analysis was employed for measurements of temperature and frequency dependence of the complex rigidity parameters. Obtained loss rigidity curves for different load frequencies enable the determination of the glass-transition temperatures and finally frequency-dependence of the loss rigidity determined on the basis of the kinetic molecular theory and Williams-Landel-Ferry (WLF) hypothesis.

**Findings:** The dependency between glass-transition temperature and excitation frequency has been investigated. The activation energy of the phase transition as well as the temperature dependence of the shift factor was calculated. The glass-transition temperature and constants of WLF equation enable the determination of temperature and frequency dependence of the loss rigidity according to the time-temperature superposition principle.

**Research limitations/implications:** The ranges of temperatures were limited to 30-150 °C and excitation frequencies to 1-200 Hz, the behaviour of the composite material outside these ranges can be estimated based on the theoretical assumptions only. Obtained dependencies are correct only for linearly viscoelastic materials.

**Practical implications:** Obtained dependencies can be useful for estimation of the mechanical and thermal degradation of polymer-based composites and can be subsequently applied for the determination of fatigue, crack growth and residual life of composite structures.

**Originality/value:** The determination of temperature and frequency dependence of the loss rigidity gives an opportunity to obtain the self-heating temperature distribution of the polymer-based composite structures under harmonic loading.

**Keywords:** Composites; Self-heating effect; Dynamic mechanical analysis

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## 1. Introduction

Constantly growing demands concerning safety, efficiency and reliability of extremely-loaded components e.g. in automotive and aerospace applications are resulting in increased interest in the use of polymer-based composites. Particularly endless fibre-reinforced composite materials with their outstanding, load-adapted strength and stiffness are considered to be attractive for the design of novel lightweight components with tailored mechanical property profiles [1].

In automobile and aircraft structures, dynamic loads are commonly present causing the fibre-reinforced composite materials to exhibit their viscoelastic behaviour. This behaviour causes energy dissipation, which is transformed into heat and results in loss of process efficiency. Due to relatively small values of heat transfer coefficients of amorphous polymers used in the most matrix systems the dissipated heat accumulates in the structure. When the dissipative heat and the thermal convection conditions are balanced, a self-heating effect in the steady-state can be observed, the temperature field is stabilised and demonstrates a quasi-linear character against the time. In the case of thermal instability the non steady-state of self-heating is noticeable. Resulting from this effect temperature generation is rapidly increasing above the glass-transition temperature, which causes a material stiffness reduction. Such transitions may initiate or accelerate damage processes and result in a malfunction of polymer-based composite components [2]. Therefore, it is necessary to determine the behaviour of these structures at various operational temperatures and excitation frequencies in order to describe the changing material behaviour. The constitutive model of such loaded structures need to be changed from viscoelastic to viscoelastoplastic or viscoplastic.

The viscoelastic behaviour of polymer-based composite materials is determined by the matrix properties, due to low glass-transition ( $\alpha$ -transition) temperature of most amorphous matrices in comparison to the reinforcement material. Therefore the properties of the reinforcing component can be neglected in thermal problems.

Polymers used as composite matrices (e.g. epoxy resins) exhibit viscoelastic response during oscillatory excitations and their material parameters are both temperature- and frequency-dependent. For the investigation of the loss rigidity of the composite material as well as of its temperature and frequency dependence, the dynamic mechanical analysis (DMA) was selected. It provides the possibility to measure complex material parameters in significant modes of oscillatory loading [3,4]. Applying a constant load or displacement, DMA gives also a possibility to obtain creep (or relaxation) characteristics of viscoelastic materials.

Theoretical investigations in the field of thermoviscoelasticity were introduced by Karnaukhov, Senchenkov and Kozlov [5]. In their works they propose governing equations of thermoviscoelasticity in the form of complete and approximate formulations. The self-heating effect was also investigated by Dinzart and Molinari [6]. They described the self-heating effect in axially loaded polymer-matrix beam subjected to pure bending loads.

Katunin's works were concentrated on the description of the self-heating effect in polymer-based composite materials as well

as in the development of the fatigue models of polymer-based composite plates. In [7] he presents an analytical model of the cross-section of a rectangular plate based on complex rigidity. The two-dimensional problem of steady-state self-heating effect of the rectangular plate subjected to pure bending was discussed in [8]. The self-heating effect in both steady- and non steady-states and its influence on the crack initiation was investigated using finite element analysis (FEA) [9]. Results on the crack initiation problem in polymer-based composites based on FEA were presented in many works (e.g. [10]), however they do not consider an influence of the self-heating. The fatigue behaviour considering self-heating effect was investigated using FEA; this analysis leads to a formulation of analytical model concept, describing the fatigue behaviour.

The aim of the investigations described in the current paper was to determine the dependencies between the loss rigidity and the temperature and excitation frequency of a rectangular plate made of glass-fibre reinforced epoxy. Obtained dependencies from the experimental results allow constructing the so-called master curve, which presents the dependency of given complex parameter simultaneously on the temperature and frequency. The experimental investigations were conducted using the DMA-thermoanalytical technique. Considering the above-mentioned dependencies in the analytical model [8] it is possible to determine temperature distribution caused by the self-heating analytically.

## 2. Problem statement and theoretical background

### 2.1. Motivation

The self-heating temperature distribution both in steady- and non steady-state can be obtained theoretically using available analytical models [5]. Using an approximate formulation of thermoviscoelasticity, analytical models that include complex parameters e. g. complex rigidity or complex modulus and their components could be derived. In the problem of the structural self-heating only the loss rigidity component is relevant, which can be explained by the viscous nature of this parameter and its influence on the hysteretic heating of the structure. According to the governing equations of thermoviscoelasticity, the loss rigidity is temperature- and frequency- dependent. Moreover, the value of loss rigidity parameter can be determined only experimentally and the DMA is considered to be the most adequate analysis for its determination. Obtaining the above-mentioned dependencies enable the formulation of the theoretical model that describes the behaviour of the investigated materials at various operational temperatures and excitation frequencies.

### 2.2. Dynamic mechanical analysis

Dynamic mechanical analysis is the combination of thermal and rheological analysis, which provides corresponding values of

thermo-mechanical properties under dynamic loads, especially for polymers and polymer-based composites. A dynamic mechanical analyzer gives the possibility to determine the time-temperature- or frequency-temperature-dependent complex parameters based on phase shift between stress and strain (represented by  $\tan \delta$ ). Due to various modes of operations like creep/relaxation, isostrain, multi-frequency, multi-stress/strain, controlled force/strain, the determination of  $\alpha$ -,  $\beta$ - and  $\gamma$ -transitions as well as thermal expansion coefficients is possible. The DMA can be conducted in a broad temperature range (typically -150-600 °C), which enable the evaluation of material thermomechanical properties for wide area of applications [11].

### 2.3. The constitutive model of thermoviscoelasticity

Linearly viscoelastic behaviour of the polymer-based composite materials can be expressed by Boltzmann-Volterra equation:

$$\varepsilon(t) = \frac{1}{E} \left( \sigma(t) + \int_0^t \Pi(t-\tau) d\tau \right), \quad (1)$$

where  $\sigma(t)$  and  $\varepsilon(t)$  are the stresses and deformations in an uniaxial stressed state (e. g. creep) at the moment of measurement  $t$ ,  $\tau$  is time elapsed until the moment  $t$ ,  $E$  is instantaneous Young's modulus,  $\Pi(t)$  is the relaxation kernel.

The stress tensor  $\sigma_{ij}$  is represented by the following formula:

$$\sigma_{ij} = s_{ij} - \frac{1}{3} \sigma_{ij} \delta_{ij}, \quad (2)$$

where  $s_{ij}$  is the stress deviator and  $\delta_{ij}$  is the Kronecker's delta. By taking into consideration the temperature dependence and modifying Eq. (1) due to the transversal isotropy of the composite design, the deviatoric stress can be expressed as (cf. [8]):

$$s_{ij} = 2D_e(T) \varepsilon_{ij} + 2 \int_0^t D(t-\tau, T) \dot{\varepsilon}_{ij} d\tau. \quad (3)$$

Here  $D_e(T)$  is the temperature-dependent instantaneous rigidity of the polymer-based composite material (where  $T$  denotes temperature),  $\varepsilon_{ij}$  and  $\dot{\varepsilon}_{ij}$  are the strain tensor component and its first derivative, and  $D(t-\tau, T)$  is the time- and temperature-dependent resolvent kernel.

According to the approximate formulation of the theory of thermoviscoelasticity, the deviatoric stress can be expressed using complex parameters. Equation (3) can therefore be presented as:

$$s_{ij} = w(X) [D_e(T_a) + \varepsilon_{\max} \hat{D}(\omega, T_a)], \quad (4)$$

where  $w(X)$  is the deflection function dependent on the coordinates vector  $X$ ,  $T_a$  is the cycle-averaged temperature,  $\omega$  is the angular frequency,  $\varepsilon_{\max}$  is the maximal deflection and  $\hat{D}(\omega, T_a)$  is the complex rigidity, which can be decomposed as:

$$\hat{D}(\omega, T_a) = D'(\omega, T_a) + iD''(\omega, T_a), \quad (5)$$

where  $D'$ ,  $D''$  are storage and loss rigidities respectively, which physically denotes elastic and viscous response with respect to the generalized Maxwell model of viscoelasticity:

$$D'(\omega, T_a) = \omega \int_0^{\infty} D(t, T_a) \sin \omega t dt, \quad (6)$$

$$D''(\omega, T_a) = \omega \int_0^{\infty} D(t, T_a) \cos \omega t dt. \quad (7)$$

The self-heating temperature can be determined from the heat transfer equation:

$$c\rho \frac{\partial T(X, t)}{\partial t} - \nabla \cdot [\lambda \nabla \theta(X, t)] = Q_{sh}(X, t). \quad (8)$$

where  $c$  is the specific heat,  $\rho$  is the material density,  $\lambda$  is the thermal conductivity and  $Q_{sh}(X, t)$  is the source function,  $\nabla$  is a vector differential operator with respect to the coordinate vector  $X$ . In this problem the source function is presented by the dissipation energy based on the hysteresis loop area:

$$Q_{sh}(X, t) = \frac{2\pi}{\omega} \int_0^{\omega/2\pi} \sigma_{ij} \dot{\varepsilon}_{ij} dt. \quad (9)$$

By solving (8) with applying thermal boundary conditions it is possible to obtain the self-heating temperature distribution [7,8]. The investigated temperature distribution function according to the Fourier's theorem can be presented in the form of infinite double trigonometric series. For two-dimensional problems it can be presented as:

$$\theta_a(u, v) = 6\omega\lambda^{-1} w^2(u, v) \varepsilon_{\max}^2 D''(\omega, \theta_a) \cdot \sum_{m=1}^{\infty} \sum_{n=1}^{\infty} \frac{\sin \mu_m \sin \gamma_n \cos \frac{\mu_m u}{l} \cos \frac{\gamma_n v}{b}}{\mu_m \gamma_n \left( 1 + \frac{\sin 2\mu_m}{2\mu_m} \right) \left( 1 + \frac{\sin 2\gamma_n}{2\gamma_n} \right) \left( \frac{\mu_m^2}{l^2} + \frac{\gamma_n^2}{b^2} \right)} + \theta_0, \quad (10)$$

where  $\mu_m$  and  $\gamma_n$  are subsequent roots of the boundary value problem

$$\mu \tan \mu = \alpha l, \quad \gamma \tan \gamma = \alpha b, \quad (11)$$

$u$  and  $v$  are Cartesian coordinates,  $l$  is the length of the plate,  $b$  is the width of the plate and  $\theta_0$  is the ambient temperature. As it can be noticed in (10), the only parameter that needs to be determined is frequency- and temperature-dependent loss rigidity. The determination of this parameter is possible in the series of experiments using dynamic mechanical analyzer.

### 3. Experimental procedure

The specimens were manufactured from glass-fibre reinforced polymer supplied by the company Epo GmbH in the form of unidirectional preimpregnated fibres. The lay-up of the composite

Table 1.

Material properties of the single UD-layer [18]

$E_1$ [GPa]	$E_2$ [GPa]	$G_{12}$ [GPa]	$\nu_{12}$ [-]	$\rho$ [kg/m <sup>3</sup> ]
38.283	10.141	3.533	0.366	1794

material, [0/ 60/ -60/ -60/ 60/ 0]<sub>s</sub>, was selected in order to achieve transversal isotropic properties. Table 1 shows the material properties of the single UD-layer of the composite material. The dimensions of the specimen are: width  $W=17.5\pm 0.1$  mm, width  $H=10.0\pm 0.1$  mm and thickness  $T=2.5\pm 0.05$  mm (Fig.1).

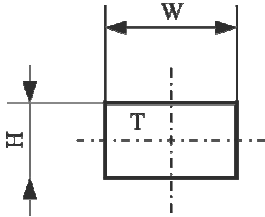


Fig. 1. Geometrical configuration of the test specimen

Dynamic mechanical analysis was conducted on the DMA-Q800 analyser of TA Instruments. The specimens were fastened in a single-cantilever clamp (Fig. 2.). The temperature range of the investigations in multi-frequency-strain operational mode was set to 30-150 °C and the corresponding heating rate to 3 K/min (conform to the requirements described in [12]). Tests in the above-mentioned configuration were performed for following excitation frequencies: 1, 2, 5, 10, 20, 50, 75, 100, 125, 150, 175 and 200 Hz. For each case, three specimens were tested in order to average the obtained results and hence to minimise the

statistical deviation. The investigations were performed under constant excitation amplitude of 20  $\mu$ m. Following parameters were measured and stored during the experimental investigations: temperature, frequency, storage and loss moduli. Selected results of conducted DMA measurements are presented in Fig. 3.

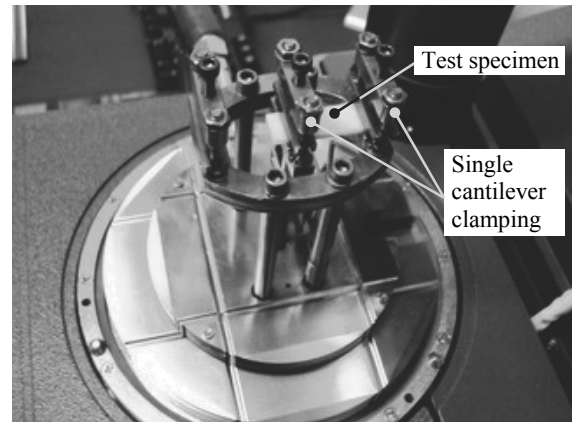


Fig. 2. Experimental setup of the DMA Analyser

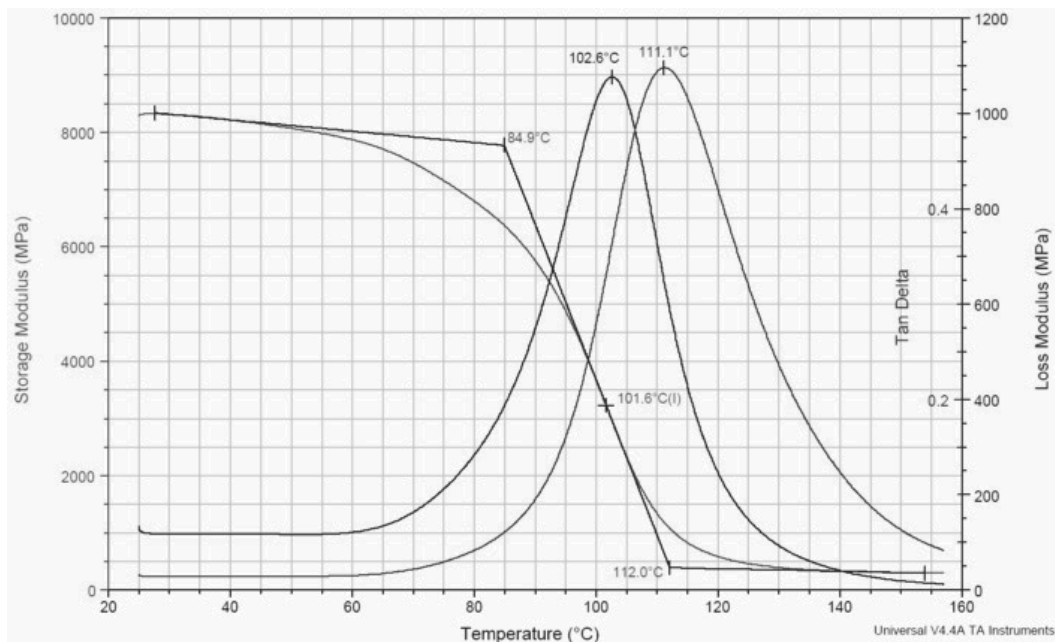


Fig. 3. Temperatures scan at frequency 10 Hz

Acquired characteristic material parameters have been applied for determination of the glass-transition temperatures,  $T_g$  based on the peak value of  $\tan \delta$  or loss modulus. Since the main focus of this study lies on the loss modulus (which can be used together with geometrical parameters for the calculation of the loss rigidity), this parameter was selected as the basis for the determination of the glass-transition temperatures. Results obtained from experimental investigations for different frequencies have been stored, and subsequently analysed in numerical computing environment.

#### 4. Results and discussion

Averaged glass-transition temperatures for above-mentioned frequency values were determined. They show that  $T_g$  increases with the increase of frequency. It is found that the relation between  $T_g$  and frequency follows the kinetic molecular theory has an exponential character and can be expressed by the Arrhenius equation (cf. [13]):

$$f = f_0 \exp\left(-\frac{E_a}{RT_0}\right), \quad (12)$$

where  $f_0$  is the empirically determined pre-exponential factor,  $E_a$  is the activation energy,  $R$  is the universal gas constant (8.314472 J/K·mol) and  $T_0$  is the reference temperature, in the present study  $T_0 = T_g$ . Fig. 4 shows the relationship between  $\ln(f)$  and  $1/T_g$ .

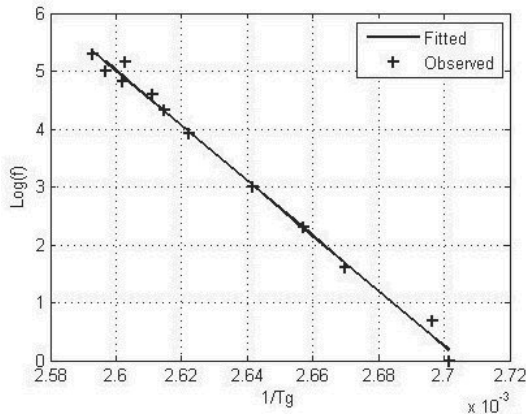


Fig. 4. Arrhenius relationship

Obtained data was fitted using linear regression algorithm. The  $R^2$  is equal 0.984. From the slope, an activation energy  $E_a$  of 395.37 kJ·mol<sup>-1</sup> can be calculated. Using this value it is possible to determine shift factors in function of temperature using modified Arrhenius equation (13) [14]. The shift factors are necessary for the construction of a master curve for the loss rigidity. According to the variability of  $T_g$  the averaged value of  $T_g$  was applied for the reference temperature  $T_0 = 105^\circ\text{C}$ .

$$\ln(a_T) = \frac{E_a}{2.303R} \left( \frac{1}{T} - \frac{1}{T_0} \right), \quad (13)$$

where  $a_T$  is the shift factor and  $T$  is the experimental temperature. The relationship of  $\ln(a_T)$  and  $T-T_0$  is presented in Fig. 5.

Results show, that the laminate reveals linear viscoelastic behaviour, so that the time-temperature superposition (TTS) principle can be applied in the investigated case. Following TTS the behaviour of linearly viscoelastic polymeric materials at the temperature  $T_0$  could be related to that at another temperature  $T$  by changing the frequency scale [14]:

$$D''(a_T \omega, T_0) = D''(\omega, T), \quad (14)$$

with  $\omega = 2\pi f$ .

The obtained values of shift factor allow to apply the Williams-Landel-Ferry (WLF) empirical hypothesis [15] for determining constants of WLF equation (15) proposed by Ferry in [16] using curve fitting.

$$\ln(a_T) = -\frac{C_1(T-T_0)}{C_2+T-T_0}, \quad (15)$$

Based on the measured values of the loss rigidity and the values of  $a_T$  it is possible to construct the master curve of the loss rigidity of the laminate. Therefore it is necessary to present the loss rigidity sequences in frequency domain (Fig. 6.) and to multiply frequency values by the shift factors. During this operation sequences are translated by horizontal shifting. Depending on the value of  $T_g$  sequences are shifted to the left (when  $T_g$  is less than the reference temperature) or to the right (when  $T_g$  is higher than the reference temperature). The obtained master curve is presented in Fig. 7.

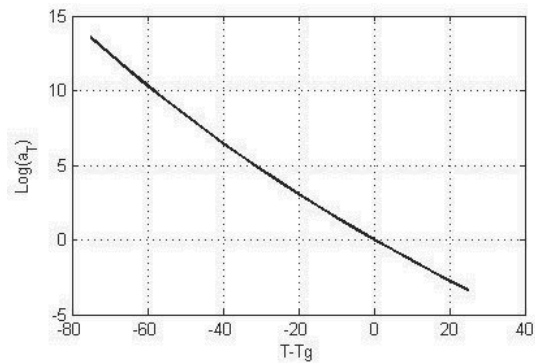


Fig. 5. Shift factor vs. temperature difference for the reference temperature of  $105^\circ\text{C}$

Obtained values of  $C_1$  and  $C_2$  for the investigated laminate are 54.4745 and 377.288 ( $^\circ\text{C}$  or  $\text{K}$ ) respectively. If condition of  $T_0 = T_g$  is satisfied the values of these constants can be assumed as “universal” ones (equal 17.44 and 51.6 respectively) for most polymers [17], but for some materials they may differ, which is confirmed, e.g. in [14]. In fact, there is a large deviation of these constants from polymer to polymer and it has been observed in many polymer systems. It is often attributed to specific character of the polymer system [18]. In the investigated study WLF equation constants does not vary with respect to  $T_g$  for different frequencies. The maximal differences of values of  $C_1$  and  $C_2$  in comparison with values obtained for reference temperatures of specified cases (see Fig. 4) are -0.16 % and -0.17 % respectively.



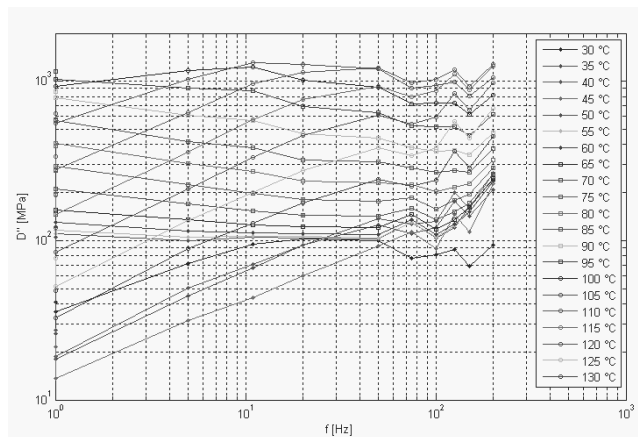


Fig. 6. Frequency dependency of the loss rigidity at different isothermal temperatures

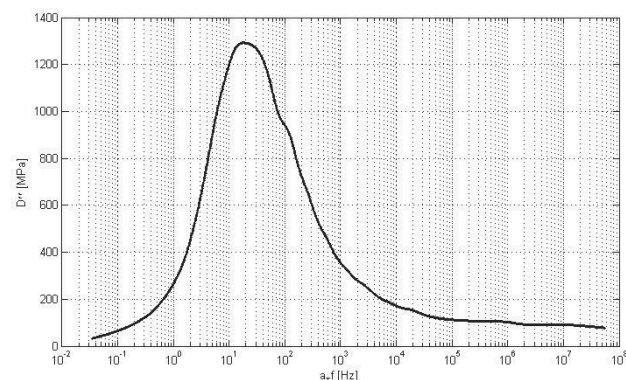


Fig. 7. Master curve of loss rigidity with respect to the reference temperature of 105 °C

The shape of the obtained master curve is in the good agreement with literature results for such materials [14]. The presented algorithm could be useful to determine viscoelastic parameters in form of Prony's series (16), which is based on the generalized Maxwell rheological model and often used in FEM commercial software:

$$D_{ij}(t) = D_{ij}^e + \sum_{n=1}^N D_{ij}^n \exp\left(-\frac{t}{\tau^n}\right). \quad (16)$$

During the experiment it was noticed, that the glass-transition temperature could be determined from the relation between force and temperature. The exemplary relation is given by Fig. 8. Note, that the relation obtained in Fig.8 is equivalent with the relation of the storage moduli versus temperature (compare with Fig. 3). The relation presented in Fig. 8 could be obtained without the necessity of the dynamic mechanical analysis, which is often expensive or not available. According to small differences between above-mentioned techniques, the precise force measurements in function of the temperature could be applied for

$T_g$  determination as well. The method of glass-transition temperature evaluation based on the analysis of storage modulus versus temperature relation is the most imprecise (usually  $T_g$  is determined based on the loss modulus or  $\tan \delta$  relations). Therefore the proposed method will be imprecise too. However, in most engineering applications such slight imprecision is negligible.

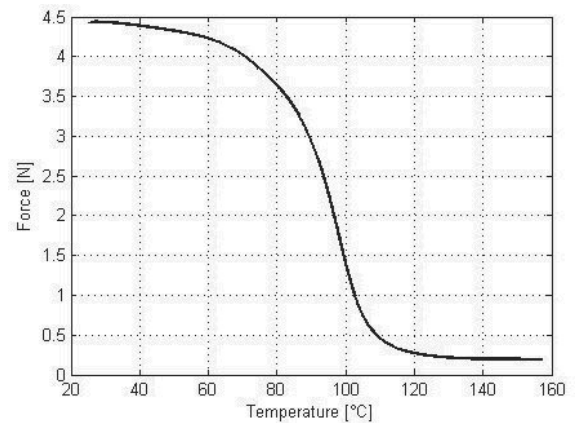


Fig. 8. Relation between force and temperature for 1 Hz

## 5. Conclusions

In this work the influence of temperature and frequency on the loss rigidity for linearly viscoelastic laminate was determined. DMA experiments conducted good agreement of measurements with theoretical models. Proposed algorithm of above-mentioned relations determination could be applied for evaluation of complex parameters of linear viscoelastic materials as well. Constants of the WLF equation were determined and were vary to the "universal" ones. Therefore, it is necessary to construct the shift factor vs. temperature curve to obtain adequate values of them and to investigate their dependence on the reference temperature, which may change the values of constants, especially for  $C_2$ . Obtaining the temperature and frequency dependence of the loss rigidity and taking into consideration the TTS principle it is possible to fulfill the analytical models of the self-heating temperature distribution and the character of increasing proposed in [7,8]. The proposed technique of the glass-transition temperature determination based on force measurements could be low-costly in comparison with DMA.

The self-heating effect could be used in problems of the damage detection and evaluation in polymer-based composite structures. Several research on this field was provided by scientific group of Prof. Wróbel [20-22]. In [20] the authors used pulsed infrared thermography for detection of defects. This method is based on the short-time heating of the composite structure by the internal heat source and the analysis of the obtained thermograms. The self-heating effect allows the damage detection without the heat source. The heating of the structure could be provided by choosing appropriate loading parameters.

In further research it is necessary to conduct the experiment of the self-heating of laminate for verifying theoretical studies. The temperature rises will change the frequency spectrum of cyclically loaded composite plates due to the stress relaxation processes in the material [23], thus it is necessary to determine the dependence of the modal characteristics in a frequency domain on the temperature, which gives a possibility to predict shifting of the structures' resonant frequencies.

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