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BASIC PROBLEMS OF RHEOLOGY OF WOOD-POLYMER COMPOSITES

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A modelling of the rheological behaviour of wood modified with polymers performed in the work was based on a conception of molecular and relaxative conditioning the viscoelastic properties of a polymer material. The kernel functions for composite integral state equations were considered as resulting from the adequate functions known for the individual components of the composite. The obtained exact solutions have taken the simplest form of the linear equations superposition for the stress relaxation function of the composite deformed along the fibres. More complicated form take the creep functions resulting from the deformation state equations which are not analytically integrable, in general. For the homopolymerous systems, like the wood-polystyrene composite investigated in normal, unchanging conditions elastic and unelastic properties could be predicted unambiguously.

1. INTRODUCTION

Structural elements made of polymer modified wood can work even when they subject to complex stress and changeable external factors such as temperature and humidity of environment. The problems concerning the improved dimentional stability of the wood-polymer composites, their reduced hygroscopicity, absorbability and swelling as well as improved parameters describing temporary mechanical properties of the composites have been widely studied and are well known e.g. [7, 11, 15, 16, 18, 23, 29, 33]. Rheology of composites have also been studied in many works [2, 5, 20, 21, 25, 26, 27, 41, 48], however, the question of stability of composite elements shape in arbitrary, assumed or allowed conditions determined by mechanical, physical and chemical interactions requires further research. The problem of the effect of structural changes in the composites caused by modification on their rheological properties has also not been solved yet.

The best from mathematical point of view and most frequently used description of rheological performance of materials in arbitrary conditions of mechanical interactions is the theory of integral equations worked out by Boltzmann and Volterra [50] employing the rule of superposition of interactions. This theory gives the following equations for the stress and deformation states of anisotropic materials:

$$\sigma_{ij}(t) = \varepsilon_{ij}(t) E_{ijkl} + \int_{0}^{t} R_{ijkl}(t-s) \varepsilon_{kl}(s) ds$$
 (1)

$$\varepsilon_{ij}(t) = \sigma_{ij}(t) J_{ijkl} + \int_{0}^{t} K_{ijkl}(t-s) \sigma_{kl}(s) ds$$
 (2)

where $\varepsilon_{kl}(t)$, $\varepsilon_{ij}(t)$, $\sigma_{kl}(t)$, $\sigma_{ij}(t)$ and $\varepsilon_{kl}(s)$, $\varepsilon_{ij}(s)$, $\sigma_{kl}(s)$, $\sigma_{ij}(s)$ are the tensors components of deformation (ε) and stress (σ) at time t and at time s prior to t. E_{ijkl} and J_{ijkl} are elastic moduli and compliance coefficients of a given material, respectively. $R_{ijkl}(t-s)$ and $K_{ijkl}(t-s)$ are the kernel functions of integral equations characteristic of a given material.

Phenomenological Boltzmann-Volterra theory is commonly used in description of properties of polymers characterized by different kernel functions [22, 30, 49]. In description of rheological properties of wood most frequently an exponential function is used which enables to derive rheological equations of a standard body [6]. However, much better agreement with experimental results at arbitrary external interactions has been obtained using Kohlrausch function [34] or its approximation Duffing function [19, 24].

The modification of wood through partial filling of empty space in wood may be a source of structural changes in wood substance resulting from localization and linking of polymer in wood cell walls as well as technology of the modification process [13]. In prediction of rheological properties of wood-polymer composites all the aforementioned factors should be taken into account, even structural changes on molecular level. The material structure and its rheological properties can be concluded about from the spectra of mechanical relaxation $H(\tau)$ and the related retardation spectra L(r) [46, 51]. These spectra determine the shape of kernel functions:

$$R(t-s) = \int_{-\infty}^{+\infty} \frac{H(\ln \tau)}{\tau} \exp\left(-\frac{t-s}{\tau}\right) d(\ln \tau)$$
 (3)

$$K(t-s) = \int_{-\infty}^{+\infty} \frac{L(\ln \tau)}{r} \exp\left(-\frac{t-s}{r}\right) d(\ln r)$$
 (4)

As follows from the analysis of dynamic mechanical properties of wood studied in wide temperature and frequency ranges [1, 2, 3, 37] and its quasistatic properties [27, 44, 45] the wood relaxation spectra reveal multiband structure characteristic of polymers [14]. Two main groups of relaxation processes responsible for bands α and β appearing in mechanical and dielectric relaxation studies [3, 35, 36] are ascribed to group relaxation process related with glass-rubber transition of amorphous regions in wood and to local relaxation processes related with reorientations of polar functional groups, respectivelly. β -type processes of the most probable relaxation time of 10^{-7} s are unnoticeable in rheological quasistatic studies. Thus the shape of kernel functions (3) and (4), which describe theological behaviour of wood in the conditions of quasistatic interactions, are determined by α -type relaxational process. In this case the relaxation time at a temperature T is defined by WLF equation [8]:

$$\tau = \tau_g \exp\left[-\frac{U^*(T - T_g)}{T - T_g + T_0}\right] \tag{5}$$

where T_g is the temperature of glass-rubber transition of amorphous components of the system, R is the universal gas constant and the other parameters are empirical constants, $U^* \simeq 17.4$ and $T_0 \simeq 50$ K for most amorphous polymers. As follows from relation (5) valid for $T \geqslant T_g - T_0$, in temperatures below T_0 , the α -type relaxation process is frosen which should be manifested by a lack of stress relaxation and creeping process. Actually, polymers and multicomponent wood in particular are characterized not by a single T_g temperature but by a distribution of such temperatures depending on the composition and molecular structure of material and implying a distribution of relaxation times. Because of that quasistatic rheological behaviour would appear even when the processes corresponding to the mean glass-rubber transition temperature T_{gm} are frozen, since some temperatures from the set of T_g temperatures would fulfil the condition $T \geqslant T_{gi} - T_0$.

2. MODELLING OF TEMPORARY ELASTIC PROPERTIES OF COMPOSITES

Description of elastic properties of wood [4, 10, 42] by the components of elastic modulus matrix E and compliance coefficiets J occurring in eq. (1) and (2), makes us suppose that despite significant variation in wood species its elastic properties are in general determined by its density. Figures 1a and 1b present the results of elastic modulus determination for over 40 species of wood along the main three anatomical anisotropy directions as a function of density. The linear dependence obtained for the wood deformation along the fibres may be described by:

$$E_l = (1 - P)E_{wl} \tag{6}$$

where E_{wl} is the elasticity modulus of walls of the fibres deformed along their axis, P — is the porosity found from the relation $P=1-\rho/\rho_w$ where ρ is the wood density and ρ_w — the density of cell walls. The simplest model which predicts a nonlinear dependence of elastic modulus determined in perpendicular to the fibres axis on density is a model of wood composed of parallel fibres of square cross-section. Then, assuming that elastic reaction comes only from the walls parallel to the direction of action we come to relation:

$$E_n = (1 - P^{1/2}) E_{wn} \tag{7}$$

where E_{wn} is the modulus of elasticity of the walls parallel to the direction of action. By computer fitting of the experimental data to the dependences (6) and (7) we can obtain the constants E_{wl} , E_{wn} and E_{wt} characteristic of the assumed model and universal for wood. Assuming the most commonly used in literature density of wood cell walls to be $\rho_w = 1450 \text{ kg/m}^3$ and making use of the data from Fig. 1 we come to the following wall elasticity moduli:

$$E_{wt} = 31,7 \text{ GPa}, \quad E_{wr} = 7,7 \text{ GPa}, \quad E_{wt} = 4,9 \text{ GPa}$$

Appropriate model considerations [16, 32, 42, 43, 47] may also help predicting the elastic properties of wood-polymer composites. Taking simplifying assumptions analogous to those described above the elasticity modulus of wood-polymer composite

deformed along the fibres is expressed by the following relation:

$$E_{cl} = (1 - P_c) E_{cwl} + S E_P \tag{8}$$

where E_{cwl} and E_P are elasticity moduli of the modified wood cell walls in the direction and of bulk polymer, S is the volume contribution of bulk fraction of polymer in the composite, and P_c is the porosity of the modified wood matrix of the composite.

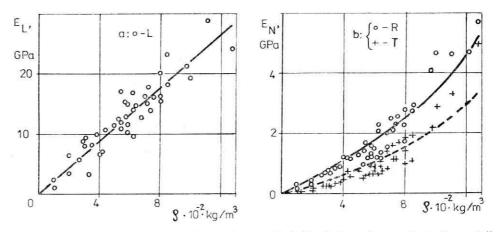


Fig. 1. Experimental (points after [10]) and theoretical (lines) dependences of elastic moduli of wood deformed along the fibres (a) and in the directions R and T perpendicular to the fibres axis (b) on wood density

Rys. 1. Doświadczalne (punkty wg [10]) i teoretyczne (linie) zależności modułu sprężystości drewna odkształcanego wzdłuż włókien (a) i w kierunkach R i T prostopadłych do osi włókien (b) w funkcji gęstości drewna

For main directions perpendicular to the fibre axis the composite elasticity modulus may be approximated by:

$$E_{cn} \simeq (1 - P_c^{1/2}) E_{cwn} + \frac{S}{P_c^{1/2} - (P_c - S)^{1/2}} E_P$$
 (9)

When the modification does not affect the wood cell walls structure, which is frequently assumed for wood composites with vinyl polymers [28, 31], the elasticity moduli of the modified wood cell walls in the above equations should take the values $E_{cwl} \simeq E_{wl}$ and $E_{cwn} \simeq E_{wn}$ characteristic of natural wood. In this case also $P_c = P$ and $S = L_{P\rho w}/\rho_P$ where ρ_w is wood density, ρ_P — bulk polymer density and L_P the degree of wood loading with polymer, defined as the ratio of the polymer mass to the mass of wood in the composite.

3. MODELLING OF RHEOLOGICAL PROPERTIES OF COMPOSITES

Description of unelastic behaviour of composites with constitutive eq. (1) and (2) requires the knowledge of the shape of the kernel functions $R_c(t-s)$, and $K_c(t-s)$, similarly as in the case of single component material. Moreover, kernel function

of the composite should be formed taking into account the shape and the parameter values of kernel functions of individual components of the composite. The above requirements are met in the spectral method of modelling of rheological properties of wood as a multicomponent sub-tance proposed in [35] and developed for wood-polymer composites in [20 and 21]. This method assumes that in deformation of composites the spectra of relaxation times are additive, analogously to the additiveness of elasticity moduli leading to equations (8) and (9). Therefore, the spectral function of relaxation times for a composite deformed e.g. along the fibres may be described as follows:

$$H_{cl}(\ln \tau) = (1 - P_c)H_{cwl}(\ln \tau) + SH_P(\ln \tau)$$
(10)

which is a linear superposition of the spectrum of relaxation times of the modified wood cell walls deformed along the the fibres $H_{cwl}(\ln \tau)$, and the spectrum of relaxation times of the polymer $H_P(\ln \tau)$.

The function of retardation times determining the shape of the kernel (4) of deformation state equation (2), may be found from the relaxation spectrum by the method of spectra transformation, as:

$$L(\ln r) = \frac{H(\ln r)}{\left[E_{\infty} - \int_{-\infty}^{+\infty} H(\ln u)(u/r)(1 - u/r)^{-1} d(\ln u)\right]^2 + \pi^2 H^2(\ln r)}$$
(11)

where $H(\ln r)$ and $H(\ln u)$ are analytical expressions for the relaxation time spectrum in which the relaxation time τ has been replaced by the retardation time r and the variable u.

Substituting eq. (10) into eq. (11) we come to the formula expressing the spectrum of the composite retardation times in terms of the retardation times spectra $L_1 = L_{cwl}(\ln r)$, $L_2 = L_P(\ln r)$; relaxation times spectra $H_1 = H_{cwl}(\ln \tau)$, $H_2 = H_P(\ln \tau)$ and the volume fractions of wood $V_1 = (1 - P_c)$ and polymer $V_2 = S$ in the composite:

$$L_{c} = \frac{\frac{1}{V_{1}H_{1}} + \frac{1}{V_{2}H_{2}}}{\frac{V_{1}}{V_{2}H_{2}L_{1}} + \frac{V_{2}}{V_{1}H_{1}L_{2}} + 2\left[\left(\frac{1}{V_{1}L_{1}} - \pi^{2}\right)\left(\frac{1}{H_{2}L_{2}} - \pi^{2}\right)\right]^{1/2} + 2\pi^{2}}$$
(12)

The obtained relaxation (10) and retardation (12) times spectra for the composite make theoretical grounds for solving the equations of stress and deformation states for polymer modified wood. These equations take the simplest forms for step changes of uniaxial deformation:

$$\varepsilon(t) = \varepsilon_0 h(t) \tag{13}$$

and stress:

$$\sigma(t) = \sigma_0 h(t) \tag{14}$$

where

$$h(t) = \begin{cases} 0 & \text{for } t < 0 \\ & \text{for } t \ge 0 \end{cases}$$
 is the Haeviside function.

Now, when we omit the indices in the equation of stress state (1), take into regard the kernel function for the composite and the action function (13), equation (1) can be rewritten in the form:

$$\sigma(t) = E_{0c} \, \varepsilon_0 - \varepsilon_0 \int_{-\infty}^{+\infty} \frac{H_c(\ln \tau)}{\tau} \int_0^t \exp\left(-\frac{t-s}{\tau}\right) ds d(\ln \tau) \tag{15}$$

Calculating the internal integral of this equation, dividing its both sides by ε_0 and taking into account the relation:

$$\int_{-\infty}^{+\infty} H_c(\ln \tau) d(\ln \tau) = E_{0c} - E_{\infty c}$$
 (16)

we come to the following formula, having assumed the additivity of the moduli (8) and spectra (10):

$$E_c(t) = (1 - P_c) E_{\infty w} + (1 - P_c) \int_{-\infty}^{+\infty} H_{cw}(\ln \tau) d(\ln \tau) + SE_{\infty P} + S \int_{-\infty}^{+\infty} H_P(\ln \tau) d(\ln \tau)$$
 (17)

This dependence, where $E_c(t) = \sigma(t)/\varepsilon_0$ is a function of stress relaxation in polymer modified wood proves to be a linear combination of stress relaxation functions of individual components of the composite. To write exact time dependences of these functions in analytical form one should know the shape of relaxation time spectra of composite cell walls and bulk polymer. Assuming that the modification of wood does not affect the structure of cell walls and the composite is formed as a homopolymer, the spectral function $H_{cw}(\ln \tau)$ should take the form characteristic of natural wood. Then, the composite relaxation function may be easily found from experimental stress relaxation curves determined for pure components. Disciepancies between the composite relaxation function obtained in the above described way and the experimental curve may be treated as indications of the modification influence on the structure of wood cel walls. An example of the effect of modification on the structure of natural wood-polymer composite matrix may be a comparison between the experimental relaxation function (a) obtained for lime wood modified with polymethyl methacrylate [25] and the function calculated on the grounds of eq. (17) assuming homopolymer type of composite structure (b), Fig. 2. Instantaneous values of the experimentally determined elasticity moduli of the analysed composite are by about 50% higher than the values expected on the grounds of the initial values of the moduli and volume fractions of individual components. The parallel shift of the relaxation curve along the moduli scale proves that methyl methacrylate polymerization in wood may be accompanied by the polymer penetration into wood cell walls, its grafting with wood components and crosslinking. In consequence the nonrelaxating elasticity modulus E_{∞} of the composite cell walls increases without any significant alteration of the mechanism of the relaxation process which determines the degree of the modulus dispersion in the considered range of relaxation times.

Of particular practical importance in modelling rheological properties of modified

wood is the possibility to predict the creep funtion course. Assuming Haeviside type run of the uniaxial stress (14) taking into account eq. (4) and having performed some operations analogous to those done in derivation of eq. (17), the deformation state equation (2) brings us to the following creep function for the composite:

$$J_c(t) = J_{0c} + \int_{-\infty}^{+\infty} L_c(\ln r) \left\{ 1 - \exp(-t/r) \right\} d(\ln r)$$
 (18)

where $J_c(t) = \varepsilon(t)/\sigma_0$ and $J_{0c} = 1/E_{0c}$.

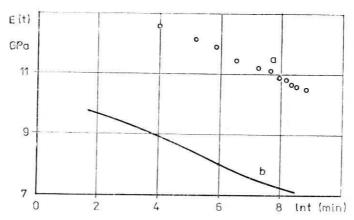


Fig. 2. Experimental stress relaxation function for wood -PMMA composite (a) after [25] and the function calculated for the assumed homopolymer type structure (b)

Rys. 2. Doświadczalna funkcja relaksacji naprężeń kompozytu drewno-PMMA (a) wg [25] oraz funkcja obliczona przy założeniu struktury typu homopolimeru (b)

Becouse of the complex form of the retardation times spectra (12) the creep function for the composite may be calculated by numerical integration of spectral functions obtained for the composite pure components. According the approximation analysis carried out in previous work [35], the symmetric distribution function of relaxation times:

$$H(\ln \tau) = H_m \operatorname{sech} \left[\alpha \ln \left(\tau / \tau_m \right) \right]$$
 (19)

was found to be corresponding to the following approximate form of the retardation times spectrum:

$$L(\ln r) \simeq L_m \operatorname{sech} \left[\alpha \ln \left(r/r_m \right) \right]$$
 (20)

where:

$$L_m \simeq \frac{1}{H_m} \left(\frac{1}{H_m J_0} - \frac{\pi}{2\alpha} \right)^{-2}$$
 and $r_m \simeq \tau_m \exp(0.9 J_0 H_m \alpha^{-2.5} / \pi)$

From the computer fitting of the experimental curves of creep function for pure

components of the composite to the following equation

$$J(t) = J_0 + (L_0/\alpha) [\pi/2 + \operatorname{arctg} P(\alpha, n)]$$
 (21)

where:

$$P(\alpha, n) \simeq (1 - \alpha^2) \operatorname{sh} \left[\alpha (n + 1 - \alpha)\right]$$
 and $n = \ln (n/r_m)$,

we could determine the parameters of the relaxation and retardation spectra of the composite components. Then, numerical integration of eq. (18) performed taking into account eq. (12), enabled the determination of the creep function for the homopolymer model of the composite. A good agreement between the theoretical dependence found in this way and the experimental creep curve has been obtained for example for the composite wood-polystyrene in the air-dry state [21]. However, when this composite was saturated with water, a significant discrepancy between the theoretically predicted and experimental dependence appeared, Fig. 3.

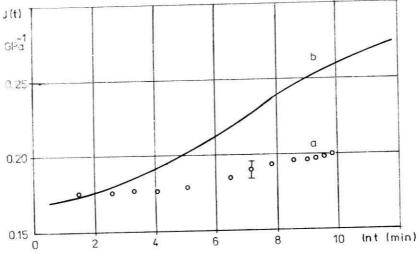


Fig. 3. The experimental creep curve for water saturated wood-polystyrene composite (a), after [20], and the model based dependence (b) calculated for homopolymer type structure

Rys. 3. Doświadczalna funkcja pełzania nasyconego wodą kompozytu drewno-polistyren (a) wg [20] oraz modelowa zależność (b) obliczona przy założeniu struktury typu homopolimeru

As follows from the figure, the initial values of complience coefficient calculated theoretically and experimentally determined are consistent, but the degree of complience dispersion obtained in experiment is over 5 times reduced when compared with theoretical predictions. This implies that in wood substance of the water saturated wood-PS composite a considerable number of relaxational processes has been frozen when compared to their number in freely swelling unmodified wood. The main reason for this phenomenon may be the effect of osmotic compression of wood substance in the conditions of its restricted swelling in the composite.

4. CONCLUSIONS

The presented attempt to develop a model of rheological properties of wood-polymer composites was based on assuming that viscoelastic behaviour of a polymer system is determined by its molecular relaxation characteristic. It was also assumed that the kernel functions of the integral state equations of the composite should be derived from the corresponding functions of individual composite components. When modelling the stress relaxation in the modified wood deformed along the fibres, the exact solutions take the simplest form of linear superposition. Far more complex and usually analytically unintegrable is the form of the deformation state equation from which the creep function of the composite is derived.

The elastic and unelastic properties of the homopolymer type system can be predicted in a definite way. An example of such a system may be wood-polystyrene composite exploired in the normal and unchangeable conditions. Because of the universal character of elastic constants and parameters of spectral funcions, the rheological properties of this kind of composite may be predicted already on the grounds of the known volume fractions of individual components.

At the present state of knowledge, it may be difficult to predict the changes in wood matrix structure and related parameters of constitutive equations in the case of wood composites obtained by polymerization of the monomers showing greater than styrene ability to penetrate wood cell walls and to make copolymers with wood components. As follows from the presented analysis, direct information about such structural changes may be obtained from measurements of stress relaxation in the composites. An additional problem appearing in quantitative determination of the effects of modification is the indeterminacy of volume contributions of the composite components. Polar monomers penetrating wood cell walls and thus being responsible for wood swelling may change the porosity of the initial matrix $(P\Rightarrow P_c)$. This process is in turn accompanied by a decrease in the contribution of bulk polymer fraction in the system to an unknown value $S \leq S_0$.

Another problem in modelling of rheological properties of polymer modified wood is the possibility to predict the composite performance in the changing conditions of environment and in particular in the conditions of changing air humidity. The above presented example of nonadditivity of rheological properties of wood-polystyrene composite soaked with water, contrasted with the usually observed additivity of these properties in normal conditions points to the complexity of the phenomena that should be taken into account in changeable humidity conditions. The polymer in the system which usually assimilates much less water than wood and restricts wood swelling may be itself subject to stress because of the osmotic compression of wood substance and thus may affect the relaxation process. At the same time the presencess of polymer hinders the access of water to wood substance what significantly affects the kinetics of wood penetration by water molecules. The following evolution of the penetration front determines the changes in relaxation processes [12, 26, 29, 41]

as well as the course of irreversible destructive proceses leading to microcrackings appearance [38].

To take into account the above mentioned effects as well others related with the temperature influence on composite properties [17, 27, 39, 40], further experimental and theoretical investigation is reguired.

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PODSTAWOWE PROBLEMY REOLOGII KOMPOZYTÓW DREWNA Z POLIMERAMI

Streszczenie

Podjętą w pracy próbę modelowania reologicznych właściwości drewna modyfikowanego polimerami oparto na koncepcji molekularno-relaksacyjnych uwarunkowań lepkosprężystych właściwości układów polimerowych, wychodząc z założenia, że funkcje jądrowe całkowych równań stanu kompozytów powinny być wyprowadzane z odpowiednich funkcji określanych dla indywidualnych składników kompozytów. Uzyskane ścisłe rozwiązania przyjmują najprostszą formę liniowej superpozycji równań przy modelowaniu zjawisk relaksacji naprężeń w odkształcanym wzdłuż włókien drewnie modyfikowanym. Znacznie bardziej złożoną i w większości przypadków nie całkowalną analitycznie postać przyjmują równania stanu odkształceń, z których wyznacza się funkcje pełzania kompozytów. W sposób jednoznaczny mogą być przewidywane zarówno sprężyste, jak i niesprężyste właściwości układów typu homopolimeru, do których można zaliczyć, eksploatowane w normalnych i nie zmieniających się warunkach, kompozyty drewna z polistyrenem.

ОСНОВНЫЕ ПРОБЛЕМЫ РЕОЛОГИИ ДРЕВЕСИНЫ МОДИФИЦИРОВАННОЙ ПОЛИМЕРАМИ

Резюме

Исходя из принципа молекулярно-релаксационных обусловленностей вязкоупругих свойств полимерных материаллов в работе проведено моделирование реологических свойств древесины модифициг ованной полимерами. Ядерные функции интегральных уравнений состояния композиционных систем определено с учетом формы и параметров ядерных функций компонентов исследованных систем. В случае моделирования процесса релаксации напряжений при деформировании вдоль волокон получено решения уравнений состояния, которые принимают самую простую форму линейной комбинации решений получаемых для компонентов системмы. Более сложную и в большинстве случаев аналитически не интегрируемую форму принимают уравнения состояния деформаций, из которых получаются функции

ползучести композиционных систем. Достаточно точно могут быть прогнозированы как упругие так и неупругие свойства систем гомополимерного типа, к которым принадлежит модифицированная полистиролом древесина эксплуатированная в нормальных и не изменяющихся условиях окружающей среды.

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