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RHEONOMIC MECHANICAL PROPERTIES OF AN EPOXY COMPOSITION
ČASOVĚ ZÁVISLÉ MECHANICKÉ VLASTNOSTI JEDNÉ EPOXIDOVÉ KOMPOZICE

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The paper presents results of experimental investigation of time-dependent mechanical characteristics of a homogeneous isotropic epoxy composition, which can be used either as a construction material or as the matrix of a particulate or fibre composite. The experimental programme involved short- medium- and long-term creep and relaxation investigations of samples loaded by quasi-static pressure. The results were used to define values of Young modulus, relaxation modulus and viscoelastic compliance, within active pressure and also delayed recovery. The measurements can serve to assess the influences of some important mechanical and non-mechanical loads (level of mechanical loading, temperature, physical aging and climate effects) on rheonomic properties of the composition in an attempt to generalise the obtained knowledge.

Článek uvádí výsledky měření časově závislých mechanických charakteristik homogenní isotropní epoxidové kompozice, která může být pro konstrukční aplikace použita buď přímo, nebo jako matrice částicového příp. vláknového kompozitu. Experimentální program zahrnoval krátkodobé, střednědobé a dlouhodobé creepové a relaxační zkoušky vzorků zatížených kvazistatickým tlakem. Z jejich výsledků byly stanoveny hodnoty Youngova modulu, relaxačního modulu a vazkopružné poddajnosti za aktivního tlaku i při zotavení. Smyslem uvedených měření je posoudit vlivy některých významných mechanických i nemechanických zatížení (úrovně zatížení, teploty, fyzikálního stárnutí a klimatotechnologického zatížení) na časově závislé vlastnosti kompozice se snahou o zobecnění dosažených poznatků.

Keywords. *Epoxy-resin-matrix-composites, long-term properties, relaxation modulus, viscoelastic compliance, physical aging*

Klíčová slova *Epoxidové kompozity, dlouhodobé vlastnosti, relaxační modul, vazkopružná poddajnost, fyzikální stárnutí*

Introduction

„Epoxy resin“ is a term that covers a diverse range of molecular types, the common feature being the presence of epoxide groups through which curing the crosslinking occurs. The general descriptive term also covers hardeners that are used in conjunction with the actual epoxy resin and differ through possession of functional groups reactive with epoxide.

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Many different mechanical properties may be measured for polymer (epoxy) matrices and composites. Among these time-dependent characteristics, like relaxation modulus and viscoelastic compliance, may be important for a real description of mechanical behaviour over a long period of time. The selected characteristic representative of the epoxy resins is a composition consisting of solventfree low-viscosity bicomponent pigmented systems on the basis of a low-molecular epoxy resin with a content of non-toxic reactive diluents, additives, pigments, fillers and auxiliary admixtures, hardened by a cycloaliphatic polyamide hardener. This material is used for surfacing a number of building substrates, such as concrete, cement screed, plaster, asbestos cement, cement-and-chipboard, steel, stone, etc., is well suited for the manufacture of self-levelling flooring top layers and blended with fillers forms trowelled polymer mortar or polymer concrete mixes. The material is hygienically harmless, temperature-resistant to loads within the range of -30° C and +90° C with a reserve. Among other uses, it can be applied advantageously to light-, medium- and heavy-duty plants requiring floors with high mechanical and chemical resistance.

Three types of experiments have been performed on samples in series comprising five specimens each (at least three samples in the long-term part of the investigations): (i) short-term experiments according to CSN 64 0606 to measure characteristic quantities in compression (yield point, strength, Young modulus, relative compression), (ii) medium-term creep and relaxation experiments (duration five-seven hours) in laboratory conditions to measure viscoelastic compliance (in active compression and in recovery) and relaxation modulus, (iii) long-term creep experiments at a stable temperature 20°C (duration in general 150 days active loading and 50 days recovery) to measure viscoelastic compliance in compression and recovery. The experiments corresponding to items (i) and (ii) were realised in ITAM, the others at CLPHCHM.

The prismatic samples 4x4x12 [cm] were made from one mixing, cured at room temperature and postcured at 80°C for 4 h. Three subgroups of samples were used for measurements: a) samples immediately after postcuring, not influenced by physical and chemical ageing (they are indicated as REJuvenated), b) samples subjected to programmed effect of climato-technological loading (periodic effect of temperature and moisture in combination with light irradiation) indicated by CTL and c) samples after physical ageing in laboratory condtions (PHA in days).

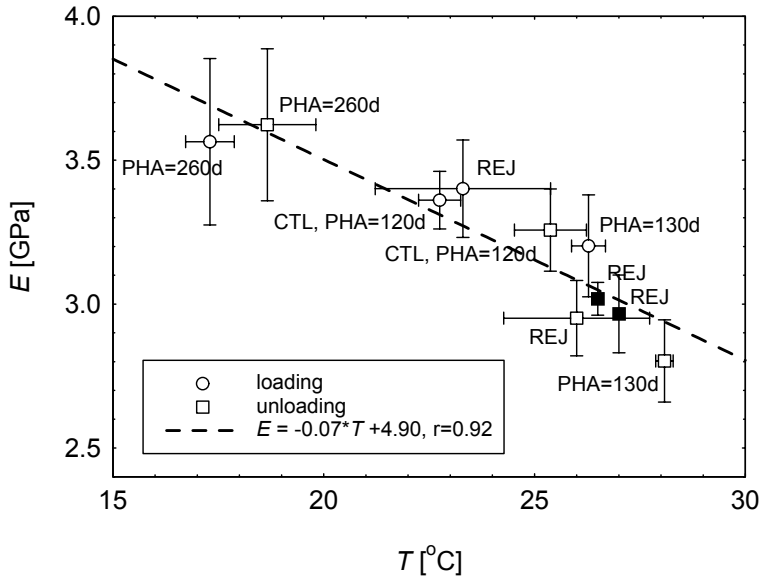

Fig.1 Young´s modulus

The CTL loading parameters were as follows: temperature range from -20°C to +60°C with 2 hours delay at the extreme temperatures, maximum relative humidity at positive temperatures, light exposure (wavelength 250-3000 nm, irradiation intensity 1,12 kW/m^2) at temperatures above +20°C, length of the loading 14 days (58 cycles).

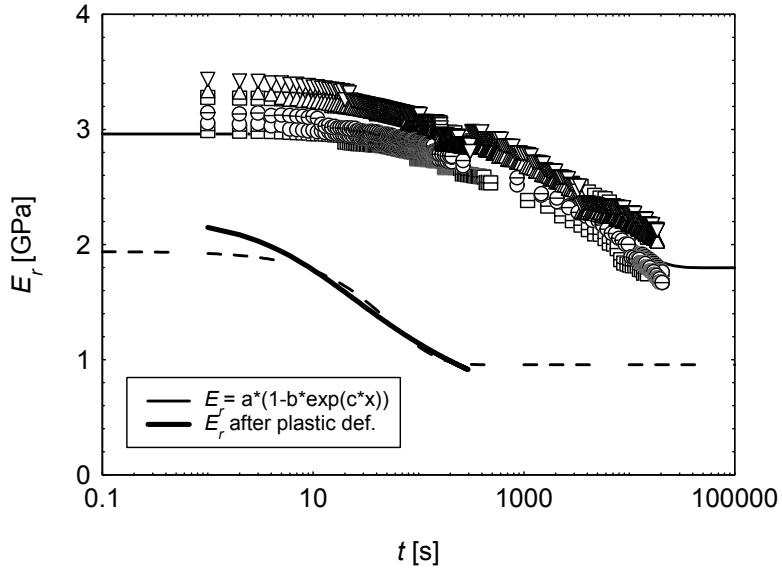

Fig.2 Relaxation modulus E_r (t)

Results

Among all mentioned influences, temperature has the most critical effect on Young´s modulus values. The power of the PHA in the given range (see Fig.1) and the CTL influence is inconsiderable in this respect. Fig. 2 shows the history of relaxation modulus E_r (t) of individual samples (marks) characterised by PHA=130 days and temperature T=26-28°C. Average values ofthe relaxation modulus of samples (REJ, T=26 °C) after plastic deformation ε= 10% are represented by the bold curve. The experimental data of the measurements are generalised by analytical regression curves. Of course, asymptotical (equilibrium) values (i.e. $E_r(\infty)$=1.8 [GPa] and $E_r(\infty)$=0.96 [GPa]) depend, among other factors, on interval of measurement and in the second lover value case can be understood (due to the shorter interval of measurement) only as a crude estimate. Fig. 3 presents comparison of the viscoelastic compliance $J_c(t)$ histories defined from medium-term experiments (5 measurements in the range from 1 to 2.10^4 [s]; PHA=260d) and from long-term experiments (7 measurements with data in interval between 15 min and 150 days; PHA=26d) at a temperature T=18-20°C. It seems that physical aging in laboratory conditions in the given range and temperature has negligible effect on viscoelastic compliance values. The medium-term data correspond well with long-term data, therefore the long-term history can be well predicted using only MTL experiments. A simple time function whose parameters are assumed to depend on the above mentioned and possibly on other influence factors $IF_{(n)}$ (n=1,2,…p) has been proposed [1] [2] to describe the viscoelastic compliance histories

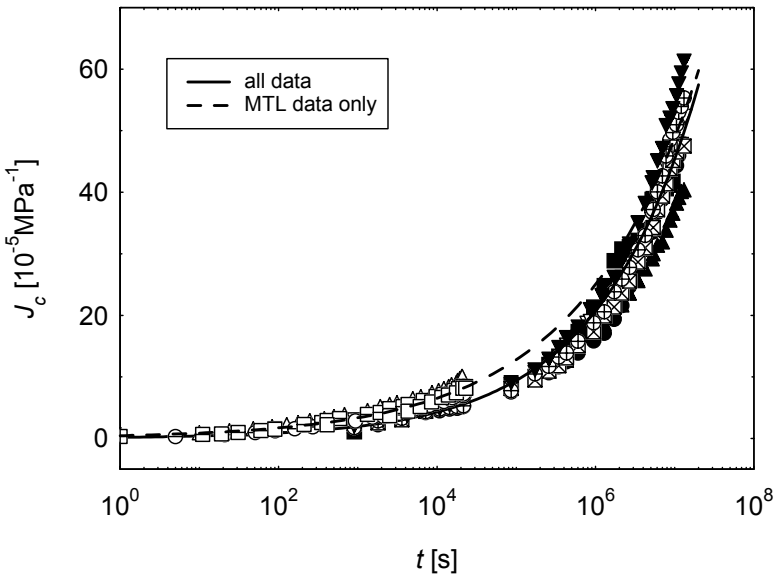

Fig.3 Viscoelastic compliance $J_c(t)$

$$J_c(t, IF_{(n)}) = P(IF_{(n)}) \exp[Q(IF_{(n)}) \log(t)] \qquad t \le t^*$$
$$J_c(t, IF_{(n)}) = \Psi(IF_{(n)}) \qquad t \ge t^* \qquad (1)$$

where $P(IF_{(n)})$ [MPa^{-1}], $Q(IF_{(n)})$ and $\Psi(IF_{(n)})$ [MPa^{-1}] are free parameters (material constants) which have to be determined from experimental data. t^* is a time point defining a qualitative change in the long-term viscoelastic response of a material. Eq.(1) defines in double-logarithmic coordinates a line with intercept $\log P$ and slope $0.434\,Q$. This is illustrated in Fig.4, which presents the history of delayed recovery viscoelastic compliance $J_{rc}(t)$ after completion of loading. In this case the asymptotic (equilibrium) value $J_{rc}(\infty)$ is known (in the contrary to $J_c(\infty)$ and $E_r(\infty)$), consequently the characteristic time t^* can easily be defined using (1). The samples whose experimental data are presented in Fig.4 are characterised by the physical aging in laboratory conditions PHA=130d and temperature T=28°C. Fig.4 illustrates large dispersion of individual data at the very beginning of measurements and relatively good regression afterwards.

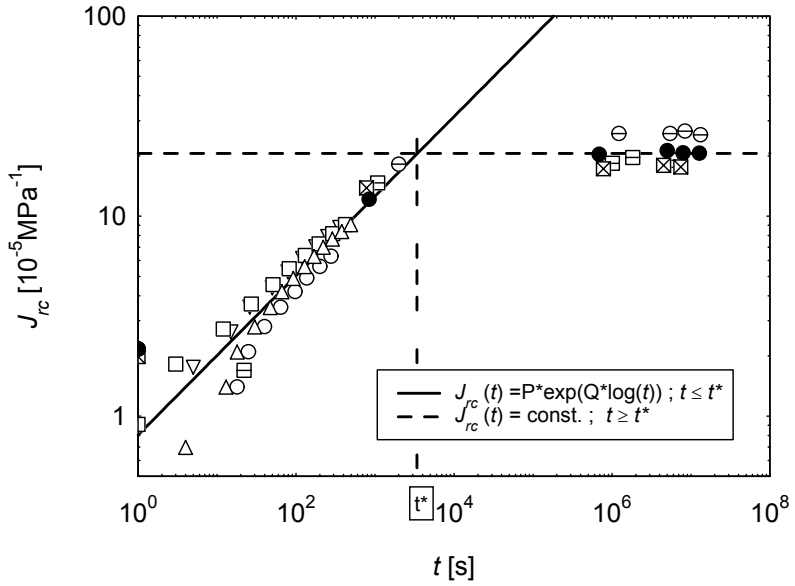

Fig.4 Viscoelastic compliance at recovery $J_{rc}(t)$

The return to the original equilibrium state is very rapid in this case due to the rather high temperature and small starting deformation for delayed recovery. In general, of course, it is impossible to eliminate a growth of non-reversible deformations during loading.

Conclusion

The results and the processing of the results demonstrate the applicability of an approach based on the use of the viscoelastic compliance measure defined by Eq.(1) to characterise quantitatively the time-dependent response of the material. The approach also has the potential to generalise the results. A follow-up set of measurements is under preparation.

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References

[1] Hristova, J., Minster, J.: *Filler fraction effect on creep response of crosslinked polyester matrix with mineral filler* – Journal of Applied Polymer Science, Vol. 89, (2003), 3329-3335

[2] Minster, J.: *Modelling of viscoelastic deformation of cortical bone tissue* – Acta of Bioengineering and Biomechanics, Vol.5, No.1, (2003), 11-20