Materials Physics and Mechanics 42 (2019) 484-490 Received: June 5, 2018

STRESS RELAXATION AND CREEP OF A COMPOSITE MATERIAL BASED ON EPOXY RESIN FILLED WITH THE HEMP BOON

V.I. Ivlev*, A.F. Sigachyov, V.A. Yudin

Mordovia State University N.P. Ogareva, Bolshevistskaya, 68, Saransk, 430005, Russia

*e-mail: ivlevvi2010@mail.ru

Abstract. Samples of composite material based on epoxy resin and hemp boon as a filler were obtained and studied. The regularities of the processes of mechanical relaxation in this composite material are qualitatively similar to the regularities of these processes in pure cured resin.
Keywords: composite materials; epoxy resin; relaxation; mechanical stress, deformation, creep

1. Introduction

The development of composite materials is one of the most promising areas of physical, chemical and technical materials science. Of particular interest are composites with components of natural, including plant genesis [1-4].

Hemp belongs to the number of few plants on Earth, which man began to use even before the emergence of agriculture. The plant has long been grown to produce fibers, seeds and oil, as well as for medical purposes. In the second half of the last century, hemp was banned because of its narcotic properties. But recently breeders received non-narcotic varieties of cannabis, so there was a real opportunity to actively and diversely use the unique properties of this plant. One of the possible ways to solve this problem is to obtain innovative composite materials with fillers made from fiber and boon of hemp. These materials can possess not only unique mechanical, but also biological properties.

Recently, active experimental studies of composite materials with fillers from hemp fibers and boon have been carried out, including those crushed to produce nanostructures [5-7]. It has been established that the mechanical properties of hemp fiber are comparable with the properties of glass fibers [4,8-9]. The influence of various stages of hemp fiber processing on the properties of the fiber used to make composite materials has been studied [5]. It is shown that the introduction of hemp fiber increases the strength and elastic moduli of the cured epoxy resin several times [9].

In most of the mentioned works the characteristics of strength and elasticity of composite materials were determined. In some studies, stress relaxation and creep of composites with hemp were studied. So, the authors of [10] came to the conclusion that showed a strain softening behaviour of the composites with increasing hemp fibre concentration. The authors of [12] studied stress relaxation in a composite material based on polyethylene with fillers from wood flour, kenaf fibers, newsprint, and rice hulls. Judging by the graphs given in the work, the dependence of stress on the logarithm of time can be approximately represented by a straight line for all the materials. However, the functional dependence was not explicitly presented. Interesting also explored in [11] the effect of fiber surface treatment on their interaction with a polymer matrix for example on hemp fibers

http://dx.doi.org/10.18720/MPM.4242019_13

adhesion with linear medium-density polyethylene. Previously, we studied these processes in hardened epoxy and polyester resins [13].

Unfortunately, to date, many details of molecular processes that determine the mechanical, including relaxation, properties of polymer composites remain unclear. For their understanding, additional researches of new materials using a wide range of experimental techniques is necessary.

The purpose of this work is an experimental study of stress relaxation and creep of composite materials based on epoxy resin with a filler from hemp boon.

2. Materials and methods of research

Composite material was obtained using epoxy resin ED-20 with hardener polietilenpoliamin (PEPA) and hemp boon, provided by OOO «Mordovian hemp plants».

Samples for the studies were prepared by the following procedure. The resin was mixed with hardener in a ratio of 10: 1, then the mixture was filled with a boon. The resulting composition was thoroughly mixed and placed in a PVC molds of rectangular cross-section. The excess liquid resin and the resulting air bubbles were neatly squeezed out. Curing of the resin occurred at room temperature. Thus, specimens measuring 14×14×150 mm were obtained. Mechanical testing of the samples was carried out not earlier than a week after the preparation of the billet.

The mechanical properties of the samples were studied using the Shimadzu Autograph AG-X Series universal test machine [14]. Control of the test process and preliminary processing of the data obtained on this machine are carried out using the software TRAPEZIUM X * 1 [15]. The program TRAPEZIUM X * 1 allows to calculate some formal module, defined as the tangent of the slope of the strain curve $(d\sigma)/d\varepsilon = E$. Below this quantity will be called the effective modulus.

3. Test results and discussion

Samples of the obtained material were subjected to three types of tests: tensile to fracture, stress relaxation and creep.

Stretching diagram. Tensile tests were carried out at a constant rate of movement of the active gripper of 2 mm / min. As was to be expected, the stress and relative strain in the specimens of the composite material during fracture were found to be substantially smaller than for the pure resin. The reason for this is obviously that the pieces of boon have sharp edges that serve as sources of additional internal stresses that stimulate the appearance of microcracks.

Figure 1 shows the stretching curve (the relationship between the relative deformation ε and the stress σ) of one of the samples of the epocon-k material, in Fig. 2 the dependence of the effective modulus E on the stress. As can be seen, these dependences, as well as for pure cured resin [16], have a rather complex character, several characteristic areas can be distinguished on them. At first, a monotonic increase in the stress is observed with an increase in deformation with an almost constant value of the effective modulus. Then, with an increase in stress from 4 to 11 MPa, the module E gradually decreases by almost three times. At a stress of more than about 11 MPa effective modulus decreases only slightly until the sample breaks down.

The nonlinear character of the curves in Fig. 1 and 2 indicate a complex character of the deformation process with a repeated change in the deformation mechanisms. Discussion of the nature of these processes is the subject of independent research.

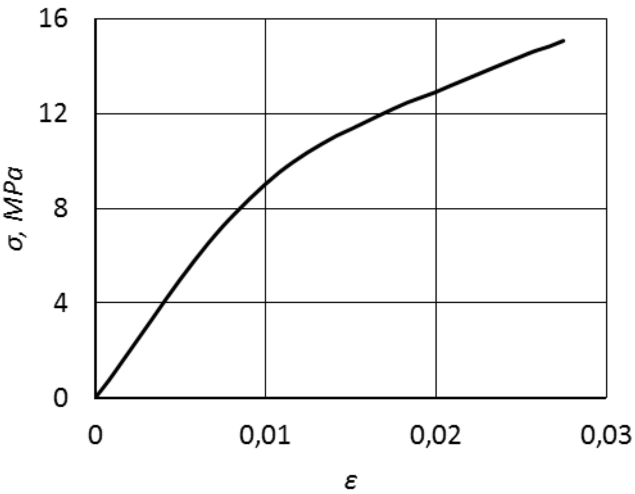

Fig. 1. Stretching diagram of the composite sample

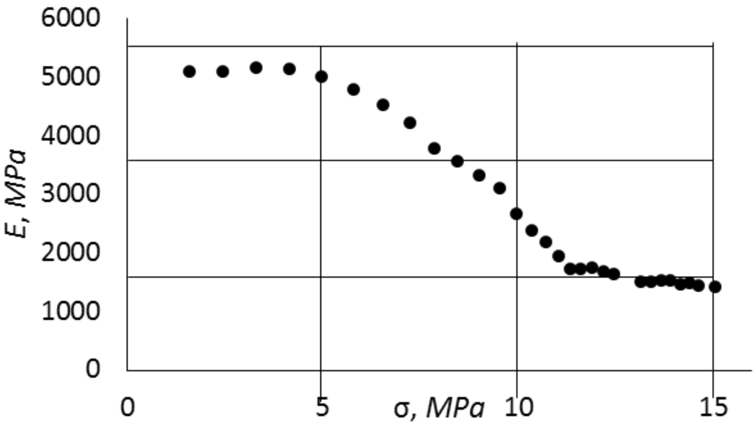

Fig. 2. Stress dependence of the effective modulus *E*

Stress relaxation. Stress relaxation in epocon-k samples was examined at room temperature. The sample was loaded stepwise to a predetermined stress level, then the grippers of the machine were fixed, and the deformation was remained for an hour. Then the load increased to the next level and the record of the relaxation curve continued.

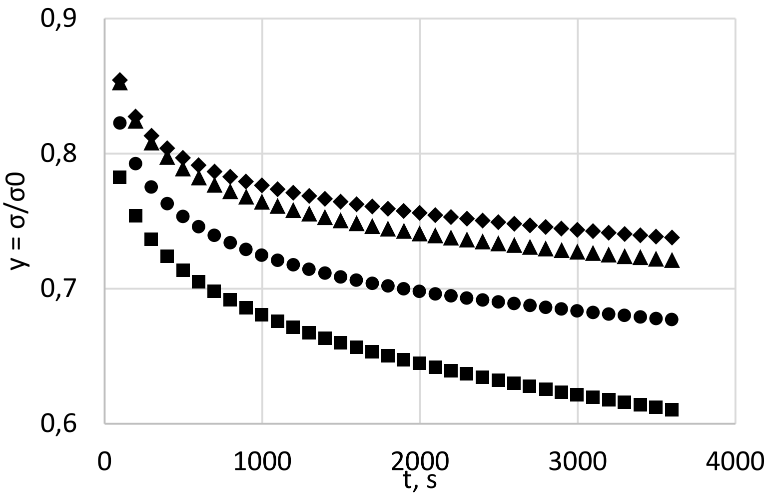

Fig. 3. Dependence of the relative stress $y = \sigma/\sigma_0$ on time at σ_0: 6, 8, 10 and 12 MPa (bottom-up)

Figure 3 shows the relaxation curves obtained at four values of the initial stress. Using the Excel program, the experimental points are plotted with trend lines. It turned out that the time dependence of the relative stress $y = \sigma/\sigma_0$ in all cases is approximated with the reliability criterion $R^2 > 0.99$ by a logarithmic function of the form

$$y = 1 - b \ln t, \tag{1}$$

where the constant b decreases from 0.049 to 0.031 with increasing initial stress of 6 and 12 MPa, respectively.

Figure 4 shows the time dependence of the value of the reduced parameter

$$\beta = \frac{\sigma_0 - \sigma}{\sigma_0 - \sigma_*}, \tag{2}$$

where σ_* is the stress at the end of the test, i.e. 1 hour from the beginning.

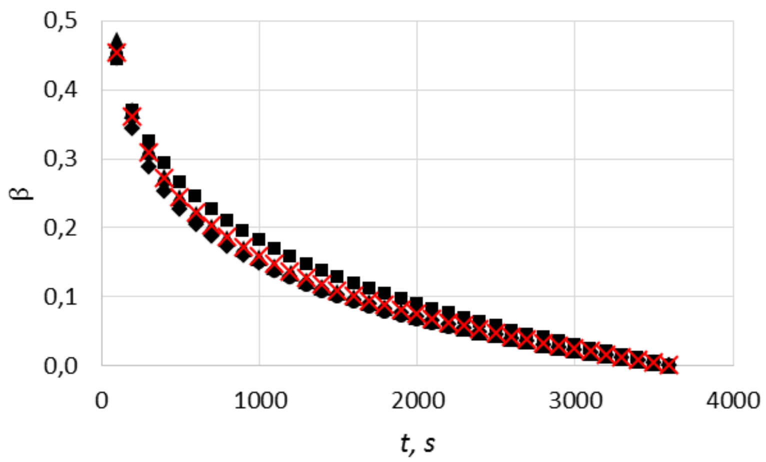

Fig. 4. Time dependence of the reduced parameter $\beta = \frac{\sigma_0 - \sigma}{\sigma_0 - \sigma_*}$

As can be seen, the obtained curves are almost superimposed on each other, in other words, parameter β is a universal characteristic that is actually independent of the initial stress. The averaging of these data (red crosses in the figure) gives a dependence satisfactorily approximated in Excel by the equation

$$\beta = -0.124 \ln t + 1.0134. \tag{3}$$

The free term of this equation must be equal to 1, but differs from it due to the error of the experimental data.

Creep. The creep of the composite was examined at room temperature. The sample was loaded stepwise to 4, 6, 8 and 12 MPa at a constant grippers rapidity and a creep curve was recorded for each load for one hour. The creep curves obtained in our experiments have the form shown in Fig. 5.

Analysis of the experimental data showed that the dependence of the relative strain on time for all stresses can be represented by a function of the form

$$\varepsilon = \varepsilon_0 + \alpha \ln(t/\tau), \tag{4}$$

where τ is a constant close to 1. The value of ε_0 is actually equal to the "instantaneous" deformation preceding the creep, and increases almost proportionally to the stress.

The value of the parameter α also increases with increasing stress (Fig. 6).

We can also introduce for creep a characteristic reduced parameter of the form

$$\eta = \frac{\varepsilon - \sigma_0}{\sigma_* - \sigma_0}. \tag{5}$$

This parameter, like the parameter β introduced above, depends little on the stress. The time dependence of the averaged value of this parameter is shown in Fig. 7 and is described by equation

$$\eta = 0{,}3012 - 0{,}034 lnt \qquad (6)$$

with criterion $R^2 = 0{,}9908$.

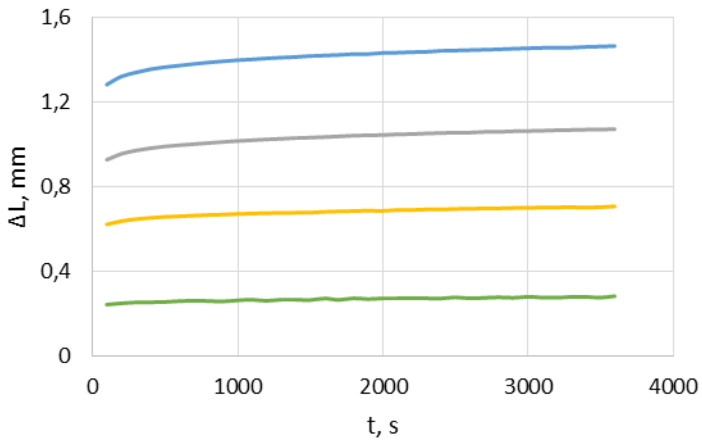

Fig. 5. Creep of composite material. The initial stress is 4, 6, 8 and 12 MPa (bottom up)

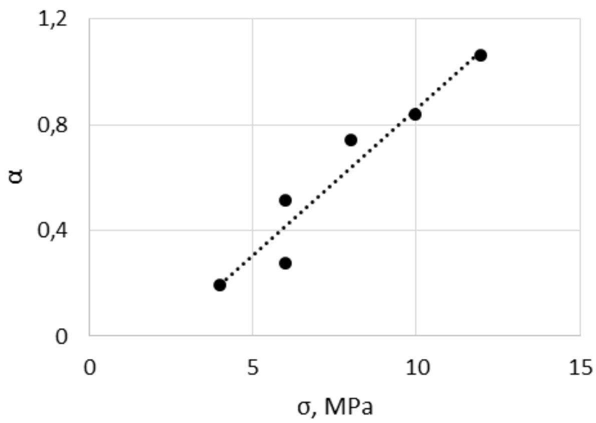

Fig. 6. Dependence of parameter α on stress

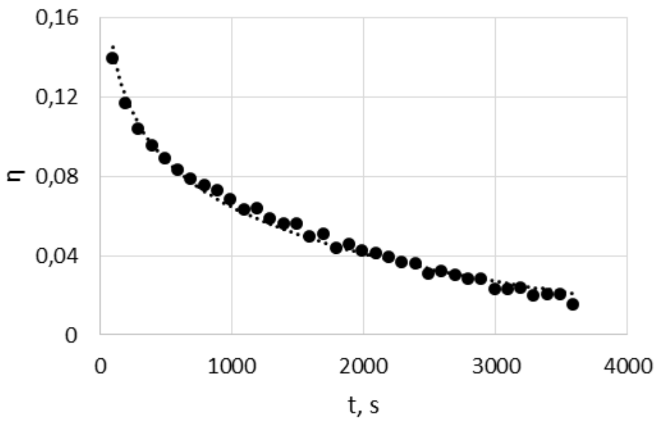

Fig. 7. Time dependence of the averaged parameter η

Earlier, we showed that the constant α in equation (4) can characterize the value of the kinetic structural element (segment) that determines the creep process - inversely proportional to it [4]. In a composite material, the constant α is approximately twice as large as in a pure epoxy resin. Consequently, the introduction of boon into the resin as a filler leads to a decrease in the size of the kinetic structural element.

It is noteworthy that the value of the reduced parameter β monotonically decreases to zero for some sufficiently large time value - on the order of 10^9 s (or more than 50 years) at σ_0 = 6 MPa. The validity of this functional dependence means that the relaxation of the stress does not end at some finite value, but occurs until it disappears completely. Such a conclusion agrees with the assumption that the main (and only) reason for the relaxation is the presence of structure defects (understood in a broad sense, and not just as crystal defects). A similar situation with long creep, characterized by the parameter η.

4. Conclusions

1. The non-linear dependencies between stress and deformation indicates a complex character of the process of deformation of a composite material with a repeated change in deformation mechanisms.
2. Relaxation of mechanical stresses and creep is described by a logarithmic function, which has the same form for amorphous cured epoxy resin and a composite material based on it.

Acknowledgements. *This work was supported by the Russian Foundation for Basic Research, project 18-48-130013 p_a «Comprehensive study of physico-chemical processes in composite materials based on epoxy resins and other synthetic polymers that are promising for use in construction»*

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