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ANALYSIS OF THE MOISTURE CONTENT EFFECT ON THE SPECIFIC INDEX AND DAMAGE ACCUMULATION KINETICS IN THE STRUCTURE OF POLYMERIC MATERIALS DURING NATURAL CLIMATIC AGING

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Abstract. We present the results analyzing the climatic resistance of epoxy polymers obtained on the basis of a modified Etal-247 resin cured by Etal-45M under the effect of natural climatic factors in a temperate continental climate. The kinetics of damage accumulation in the structure of polymer samples under tensile loads was studied on the basis of the results obtained using the author's method. The essence of the technique was to determine the coordinates of the "critical" points of deformation curves based on the time series fractal analysis methods, recorded with a high readings frequency (0.01 s). To estimate the level of accumulated failures leading to the destruction of samples under tensile loads, we used a parameter defined as the ratio of the number of points with a fractality index less than 0.5 to the total number of points on deformation curves (until reaching the level of "critical" tensile stresses). Time intervals of 0.16 seconds were studied with analyzed area shifted with a step of 0.01 sec. A specific index is proposed that characterizes the accumulated number of damages in the polymer sample structure per unit of its strength. Its achievement leads to the destruction of the composite under study. We have determined the moisture state effect and climatic aging duration on the damage accumulation kinetics in the polymer sample structure under tensile loads.

Keywords: climatic aging, polymers, moisture content, damage accumulation, specific index, fractal analysis.

АНАЛИЗ ВЛИЯНИЯ ВЛАГОСОДЕРЖАНИЯ НА УДЕЛЬНЫЙ ПОКАЗАТЕЛЬ И КИНЕТИКУ НАКОПЛЕНИЯ ПОВРЕЖДЕНИЙ В СТРУКТУРЕ ПОЛИМЕРНЫХ МАТЕРИАЛОВ В ПРОЦЕССЕ НАТУРНОГО КЛИМАТИЧЕСКОГО СТАРЕНИЯ

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Аннотация. Представлены результаты анализа климатической стойкости эпоксидных полимеров, получаемых на основе модифицированной смолы Этал-247, отверждаемой Этал-45М, под действием натурных климатических факторов в условиях умеренно-континентального климата. Изучена кинетика накопления повреждений в структуре полимерных образцов под действием растягивающих нагрузок на основе результатов, полученных с помощью авторской методики. Сущность методики заключалась в определении координат «критических» точек кривых деформирования на основе методов фрактального анализа временных рядов, фиксируемых с высокой частотой снятия показаний (0.01 сек.). Для оценки уровня накопленных отказов, приводящих к разрушению образцов под действием растягивающих нагрузок, использовался показатель, определяемый как отношение числа точек с индексом фрактальности, меньшим 0,5, к общему числу точек кривых деформирования (до достижения «критических» уровней растягивающих напряжений). Исследовались временные интервалы продолжительностью 0.16 секунд со смещением

анализируемой области с шагом 0.01 сек. Предложен удельный показатель, характеризующий накопленное число повреждений в структуре полимерных образцов к единице его прочности, достижение которого приводит к разрушению исследуемого композита. Выявлено влияние влажностного состояния и длительности климатического старения на кинетику накопления повреждений в структуре полимерных образцов под действием растягивающих нагрузок.

Ключевые слова: климатическое старение, полимеры, влагосодержание, накопление повреждений, удельный показатель, фрактальный анализ.

1. INTRODUCTION

Polymer materials are widely used almost in all existing industries. By analogy with other materials, the main requirement for polymer-based products and structures is to ensure uninterrupted operation throughout the entire service life. However, the solution to this problem is extremely complicated under the effect of natural climatic factors, as practically all products and structures are exposed to the effect thereof, regardless of their functional purpose. This is due to the high complexity of climatic effects both for analysis and for replication in laboratory conditions with sufficient accuracy and completeness [1 – 6].

Since the complete reproduction of the environment impact in artificial conditions at the moment is not possible due to the insufficient level of instrumental and technical development, the main source of reliable information on the phenomena that arise in the polymer structure during climatic aging are natural climatic studies [7 – 13]. One of these phenomena is the reversibility of changes in the physical and mechanical properties of epoxy polymers, depending on the content of adsorbed moisture. According to the data given in the scientific literature, as well as the author's studies [14 – 16], the spread of strength parameters in the limiting moisture states (moisture saturated and dried) reaches 30% for polymer composites and 50% for unfilled epoxy resin-based polymers. As a result, during the operation of polymer composites, one should take into account both irreversible changes in properties caused by degradation of the surface layers of the product, disordering of filler fibers, photodestruction and chemical transformations of the polymer matrix, as well as reversible changes caused by the processes of sorption and desorption of atmospheric moisture.

The aim of this work is to quantitatively assess the kinetics of damage accumulation in the structure of epoxy polymer samples exposed to natural climatic factors, depending on their duration, the level of applied mechanical impacts, and the polymer material moisture state.

2. METHODS AND MATERIALS

The objects of the study were samples based on Etal-247 resin and Etal-45M hardener by ENPTs EPITAL JSC. Etal-247 is a modified epoxy resin with a mass fraction of epoxy groups of at least 21.4÷22.8% and a Brookfield viscosity at 25 °C in the range of 650÷750 cP. Etal-45M hardener is a mixture of aromatic and aliphatic di- or polyamines modified with salicylic acid.

The samples were subjected to natural exposure on the test stands of the environmental and meteorological monitoring laboratory, construction technologies and examinations of the Ogarev National Research Mordovia State University (Saransk, temperate continental climate). Physicomechanical parameters were determined for samples in a control state, as well as after 67, 151, 306, 531 and 765 days of natural exposure.

The samples were conditioned in accordance with GOST 12423-2013 “Plastics. Conditioning and Testing of Samples”. To establish the effect of the moisture state on the change in the physical and mechanical parameters of epoxy polymers under natural climatic factors, a series of 36 samples exposed in parallel was divided into 3 equal batches tested: immediately after re-

moval from the test site (series "without conditioning"); after moistening to constant weight at a relative humidity of 98±2% ("moisture-saturated" series) and after drying to constant weight at 60 °C ("dried" series) in accordance with GOST R 56762-2015 "Polymer Composites. Method for Determining Moisture Absorption and Equilibrium State".

Mechanical tensile tests of samples (type 2 according to GOST 11262-2017) (ISO 527-2: 2012) "Plastics. Tensile Testing Method") were made using an AGS-X series tensile testing machine with TRAPEZIUM X software. Test temperature was 23±2 °C and relative air humidity of 50±5%. The tensile testing machine clamp movement speed was 2 mm/min.

Quantitative values of accumulated failures are determined on the basis of the author's technique, which allows determining the coordinates of critical points of deformation curves "$\sigma - \varepsilon$" built by methods of fractal analysis [17 – 20]. As "critical" points, we consider the points of the deformation curves for which the $\mu(\sigma, \varepsilon)$ fractality index values calculated over the previous short time intervals using the least coverage method, are less than 0.5. Time intervals of 0.16 seconds were studied with analyzed area shifted with a step equal to 0.01 sec.

To estimate the level of accumulated failures leading to the destruction of samples under tensile loads, we used a parameter defined as the ratio of the number of points with a fractality index less than 0.5 to the total number of points on deformation curves (until reaching the destructing level of tensile stresses)

$$\omega(\sigma, \varepsilon) = \frac{m_{\mu(\sigma,\varepsilon)}}{n_{\mu(\sigma,\varepsilon)}} \cdot 100\%, \qquad (1)$$

where $m_{\mu(\sigma,\varepsilon)}$ is the number of points of the studied series for which the condition $\mu(\sigma, \varepsilon) < 0.5$ is satisfied; $n_{\mu(\sigma,\varepsilon)}$ is the total number of analyzed points.

To take into account the effect of changes in the strength parameters of polymers on the value of accumulated failures (until the sample reaches maximum tensile stresses), we introduce specific index θ defined as [13]

$$\theta = \frac{\omega}{R_s}, \qquad (2)$$

where ω is the relative number of accumulated damages determined according to (1) when the sample reaches the level of maximum tensile stresses [%]; R_s is the tensile strength of polymeric materials [MPa].

3. RESULTS AND DISCUSSION

The change in the sample mass after drying and moistening according to the above modes is shown in Fig. 1. Depending on the moisture state of the samples after being removed from the test stands during drying and moistening to constant weight, a decrease and increase in the weight of the samples by 0.67÷1.35% and 0.88÷1.94%, respectively, is observed. Depending on the duration of natural climatic aging, the range of change in the sample mass ranged (the sum of absolute values of the mass increase and decrease) from 2.09% to 2.73%, and the highest value was recorded for the samples in the control state.

According to the results of the studies, it was established (Fig. 1, 2) that an increase in the moisture content of the control samples from 0.79 to 2.72% (moisture-saturated state) leads to a decrease in the ultimate tensile strength from 37.3 to 26.7 MPa, which corresponds to a residual strength of 71.5% (of the control values in an equilibrium-moisture state). Natural exposure of polymer samples of the studied composition without additional drying and moistening is accompanied by a decrease in strength parameters for the entire exposure time (765 days) by no more than 15%. In this case, the moisture saturation of the samples contributes to an additional decrease in the tensile strength, reaching 24÷35% of the initial value (before the beginning of natural exposure).

The strength parameters of dried samples with the duration of natural climatic exposure not exceed-

ing 306 days, are higher than those for samples not subjected to additional drying. Such restoration of properties is referred to as a reversible change in strength parameters due to the elimination of free moisture. In addition, with an increase in the natural exposure duration, we observed a gradual narrowing of the range of variation of the polymer sample strength parameters in the moisture-saturated and dried states. For time intervals of 531 and 765 days, the difference is only 0.3÷0.65 MPa (Fig. 2, a). At the same time, the tensile strength for samples not subjected to additional conditioning is 15-18% higher than the same parameter in the limiting moisture states. Obviously, for a given natural climatic impact duration, the presence of sorbed moisture in the polymer matrix structure acts as a mechanism that compensates for irreversible changes. At the same time, for the state of samples aged more than 531 days, it is obvious that there is a certain point of optimum moisture content corresponding to the highest ultimate tensile strength value. By analogy with the plasticizing effect of moisture, one can assume a gradual decrease in the contribution of the considered synergistic effect analyzed in [20], from the maximum value at the point corresponding to the moisture-saturated state to zero at the point corresponding to the dried state.

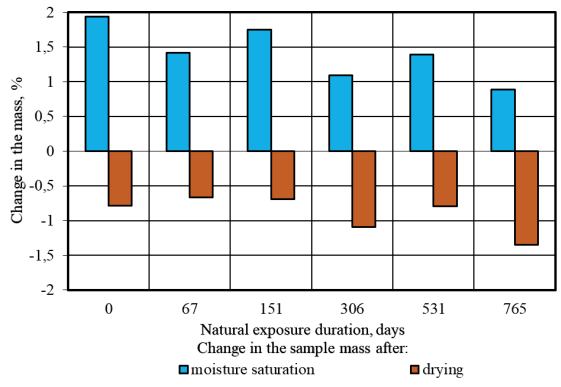

Figure 1. Change in the mass (%)of Etal-247 + Etal-45M polymer samples during their drying and moisture saturation to constant mass

Accumulation curves of failures depending on the level of applied stresses and elongation in tension for control samples in different moisture conditions after natural climatic exposure are shown, respectively, in Fig. 3 and 4. Numerical values of the levels of accumulated failures corresponding to the achievement of the maximum tensile loads by the samples are given in Table 1. It was found that the limiting level of accumulated damage for all types of moisture state of samples of the studied composition varies from 5.06 to 6.05%, increasing in the series: dried (5.06÷5.61%), moisture-saturated (5.23÷6.01%), without additional conditioning (5.53÷6.05%). The analysis results show (Fig. 3, a) that curves of failure rate accumulation depending on the level of applied stress for samples in equilibrium-moisture or dry states are similar. Moisture saturation of the samples leads to a significant (up to two times) acceleration of the process, depending on the level of applied stresses.

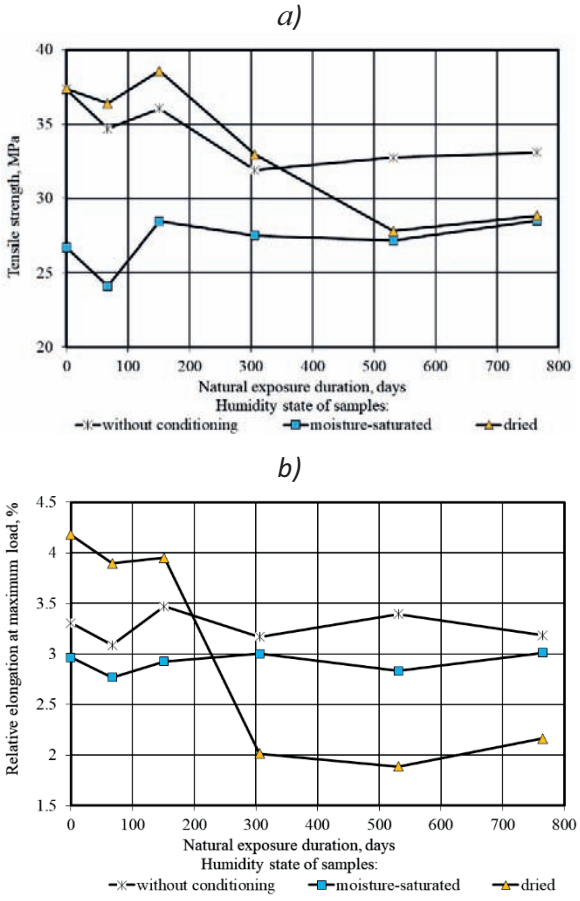

Figure 2. Change in elastic-strength parameters (a – tensile strength; b – relative elongation at maximum load) of polymer samples of the composition Etal-247 + Etal-45M during natural climatic aging in three moisture states (without conditioning, moisture-saturated, dried)

Table 1. Change in the cumulative failure rate of epoxy polymer (Etal-247+Etal-45M) during natural climatic aging in three moisture conditions (without conditioning, moisture-saturated, dried).

Humidity state of samples	Natural exposure duration, days					
	0	67	151	306	531	765
without conditioning	5,69	6,05	5,71	5,76	5,72	5,53
moisture-saturated	6,01	5,61	5,23	5,64	5,50	5,86
dried	5,53	5,61	5,51	5,06	5,43	5,54

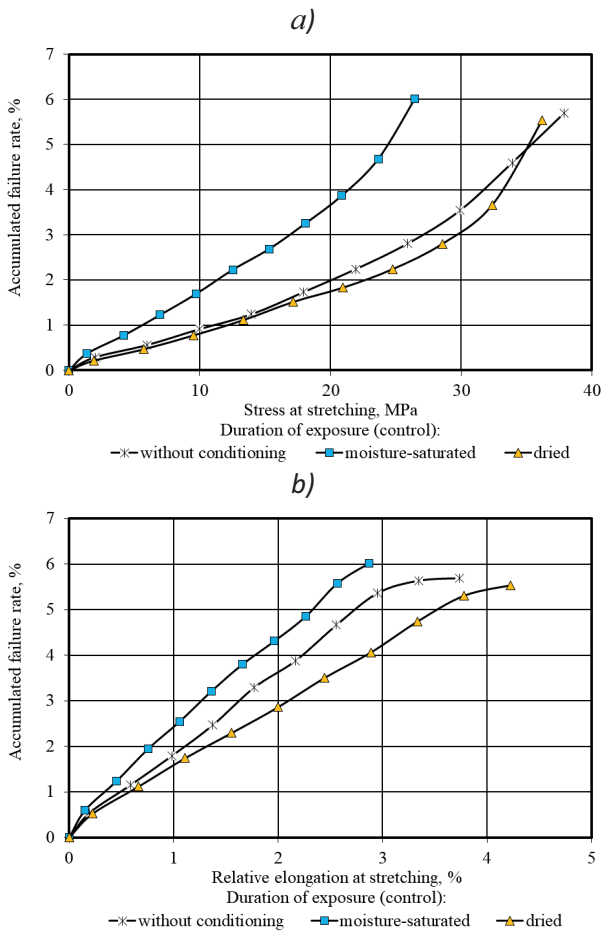

Figure 3. Failure accumulation curves for a series of polymer samples (Etal-247+Etal-45M) in different moisture states depending on the level of applied stresses (a) and relative elongations at stretching (b)

Analysis of changes in the curves for epoxy polymers (Etal-247 + Etal-45M) (Fig. 4) after natural exposure in a temperate continental climate for 765 days indicates that the nature of damage accumulation depending on the level of applied stresses for samples in equilibrium-humidity and moisture-saturated states are similar, especially at levels of tensile stresses not exceeding 40-50% of destructive ones. Moisture saturation of the samples after climatic aging over the entire studied time interval also leads to an acceleration of damage accumulation in comparison with the samples not subjected to additional conditioning. The greatest changes in the damage accumulation kinetics, depending on the natural exposure duration, were recorded for dried samples (Fig. 4, e, f). Thus, an increase in the climatic impact terms of up to 306 days and more leads to a significant acceleration of damage accumulation even at low stresses (Fig. 4, e). At the same time, the shape of the curve “accumulated failure rate –tensile stress” also changes – from concave to close to linear.

An analysis of similar curves for a series of dried samples plotted depending on the level of elongation under tension (Fig. 4, f) also clearly shows the change in the nature of damage accumulation. When physically bound water is removed from the studied epoxy polymer structure exposed to the natural factors site for 306 days or more, brittle fracture of the samples under tensile loads is observed.

The correlation dependence between specific index θ and the ultimate tensile strength of the epoxy polymer (Etal-247 + Etal-45M) in various moisture states (equilibrium-humidity, moisture-saturated and dried) with a sufficiently high reliability ($R^2 = 0.856$) is approximated by an exponential dependence (Fig. 5 , a) of the form

$$\theta = 0.492 \times \exp(-0.031 \times R_s). \quad (3)$$

Approximating the correlation dependence "$\theta - \Delta\varepsilon_s$" (Fig. 5, b) by the equation (without taking into account the data for dried samples after climatic exposure with a duration of 306, 531 and 765 days)

$$\theta = 0.484 \times \exp(-0.296 \times \Delta\varepsilon_s) \quad (4)$$

the reliability drops to 0.684.

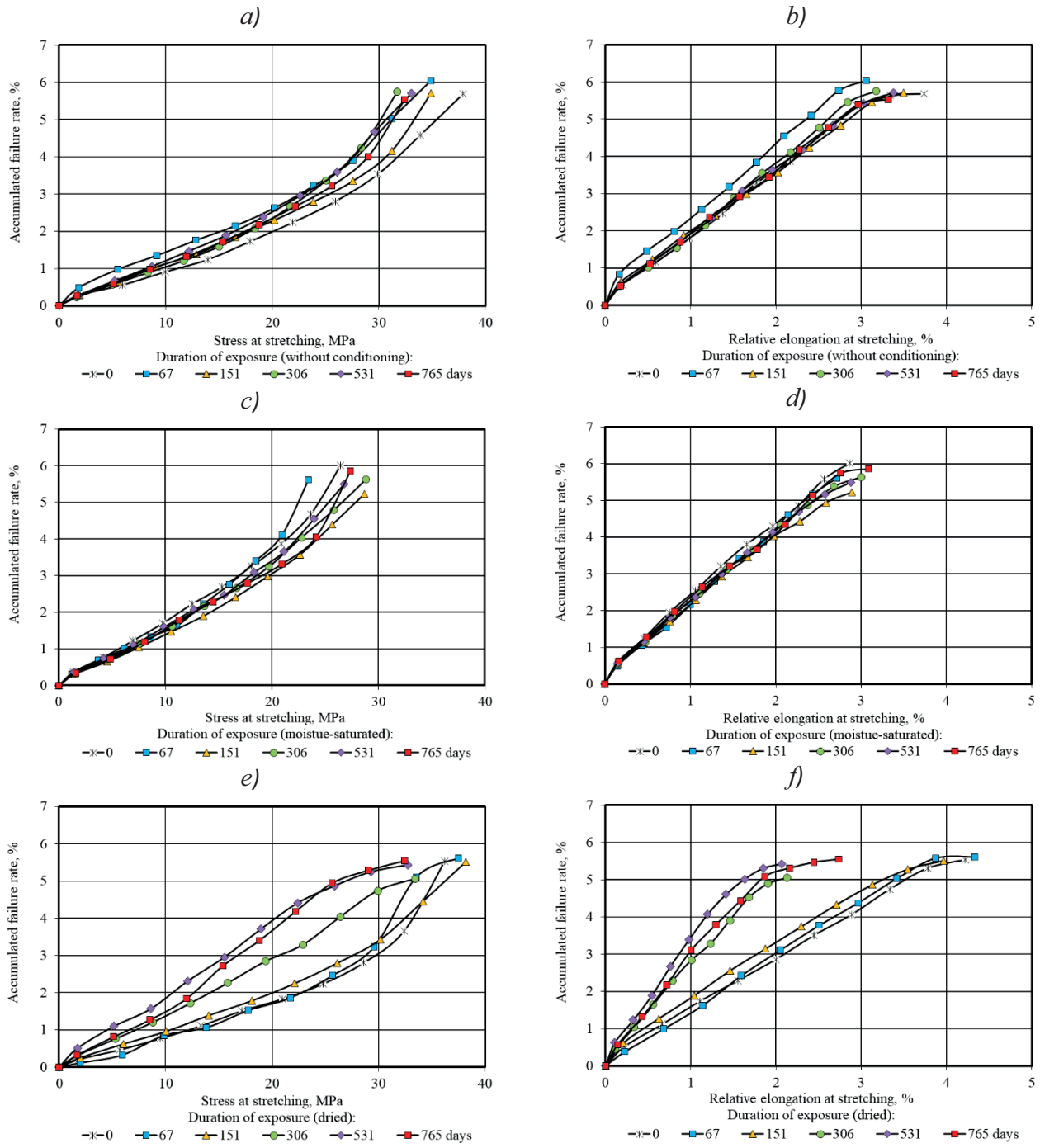

Figure 4. Failure accumulation curves for a series of polymer samples (Etal-247+Etal-45M) during natural aging in different moisture states (a, b – without conditioning; c, d – moisture-saturated; e, f – dried) depending on the level of applied stresses(a, c, e) and relative elongations at stretching (b, d, f)

The possibility of approximating the correlation dependences shown in Fig. 5 (a) by for all series of the studied composition depending on the moisture state of the samples and the duration of natural exposure, confirms the advisability of using specific index θ as a quantitative charac-

teristic of assessing the level of accumulated damage, where its achievement leads to the destruction of polymers. In this case, the analysis of Fig. 5 (b) is an additional confirmation of the change in the mechanism of destruction for samples, with aging duration exceeding 306 days.

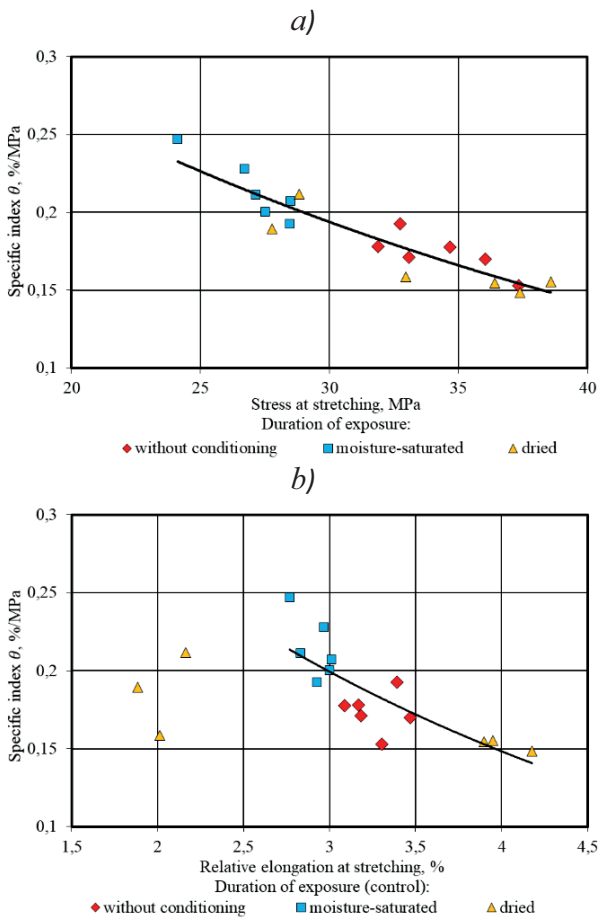

Figure 5. Correlation dependences between the specific index of the number of damages θ and elastic-strength characteristics (a – tensile strength; b – tensile elongation) of an epoxy polymer (Etal-247 + Etal-45M) in different moisture conditions.

4. CONCLUSION

The analysis of the research results showed that the sorbed moisture content is the main source of reversible changes in the elastic-strength parameters of polymer material samples. When the exposure time does not exceed 306 days, the decrease in the ultimate strength of the samples in the moisture-saturated state varies in the range from 16 to 34% of the analogous parameters for the dried samples. An increase in the duration of natural climatic exposure to 531 days and more leads to a change in the nature of the effect of adsorbed moisture on the strength parameters of epoxy polymers (Etal-247 + Etal-45M). In particular, the tensile strength of the samples tested immediately after removing the samples from the test benches (without additional conditioning) is 15-18% higher than the strength parameters in the limiting (moisture-saturated and dried) moisture states.

A sharp decrease in the deformative characteristics of the dried samples was also revealed when the time of natural exposure reached 306 days or more. The climatic effect in the time interval from 151 to 306 days is characterized by a two-fold decrease in the relative elongation during stretching of the dried samples (Fig. 2), which is presumably due to their additional embrittlement due to the removal of sorbed moisture, which in this case plays the role of a compensator for irreversible changes in the polymer matrix structure during natural climatic aging. In this case, a change in the damage accumulation kinetics is also observed depending on the level of applied tensile stresses.

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