

# Analysis of changes in fractographs quantitative indicators of the polymer composite fracture surfaces under the influence of the natural climatic factors

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**Abstract.** Analysis of changes in fractographs quantitative indicators of the polymer composite fracture surfaces in the aging process in temperate continental climate is carried out on the basis of the offered technique. It is found out that the strength of polymer samples at tension depend on the square of the fracture surface mirror zone.

## 1. Introduction

The environment consists of conditions where the most materials and products, regardless of their functional purpose, are used. Joint impact of a climatic factors set over a specific time period may be accompanied by significant change of material properties and, as a result, its performance decrease, which entails the need to perform reconstruction works. In the recent decades, the issue of climate change caused by continuous human activities – industry, land use, and livestock sector, deforestation, and natural fluctuations of the Earth's climate attracts ever growing attention of the global community [1].

This research leads to a continuous change in the operating conditions of materials, which, in its turn proves the need to carry out early research of their resistance to climatic impacts. One of the possible solutions to the problem of materials resistance in the conditions of constantly changing climate is the development of mathematical models predicting the material properties change depending on quantitative values of the environmental factors. It should be mentioned that the transition to quantitative values of the climatic factors as decisive parameters for determining the climatic aging can be the essential step for obtaining a universal model for calculating residual life of a product for all climatic zones, terms, and operating conditions.

The development of modern building materials science based on the introduction of the "green" technologies concept requires application of materials, on the one hand, it may be reused and, on the other hand, feature enhanced energy efficiency which is attained by significant increase of the service life [2, 3]. Since the most structures interact with the environment indirectly, but through protective coats (typically polymer-based ones), the development of polymers which will be resistant to natural climatic factors has the highest expediency [4-6].

The main difficulty of the climatic resistance evaluation of polymeric materials consists, on the one hand, in a large number of operating environmental factors, and, on the other hand, in the lack of sufficient number of determined interdependencies between the polymer properties at nano-, micro -



and macro-levels. And while the complexity of climatic effects can be significantly reduced by taking into account only the basic environmental factors for the climatic resistance evaluation [6-8], the identification of dependencies between the material properties at different scale levels is currently a very topical field of the modern materials science. Special attention is drawn to the analysis of the polymeric material fracture surface exposed to natural climatic factors. The aim of this work is to establish the relationships between the quantitative values of the fracture surfaces and the integral strength characteristics of epoxy polymer samples.

## 2. Fracture surfaces of the composite materials

Previous investigations of the polymeric material fracture surface at tensile stress allowed to classify the surface state on depending on the fracture type 0 to smooth and rough types [9,10].

According to [11], heterogeneous fracture of polymer materials is caused by a defect which is a hub of stresses and includes 3 crack propagation stages: absence of crack growth, slow crack growth, rapid crack propagation. Slow crack growth is caused by thermomechanical activation processes, the nature of which is similar to the homogeneous nature of fracture – tensile of a chain, plastic deformation, pore opening, untangling and fracture of the chain [11]. Thus, the rate of these processes reaches its maximum at the crack top, where the rate is greater than elsewhere in the material. Therefore, the fracture surface area which is called the mirror area represents the initial stage of crack formation and features the lowest formation rate. In this case, the fracture source is a microdefect having a form of the pore, inclusion, or structural irregularity. Thus, the mirror area usually has form of a circle of certain diameter, with its center located close to the defect which has initiated the polymer fracture [9, 10].

## 3. Equipment and materials used

Fractographic study of the epoxy polymers was carried out using the optical stereo microscope Olympus SZX-16 ("Olympus Corp.", Japan). For the mechanical tensile tests of the polymeric composites, tensile machine of AGS-X series with TRAPEZIUM X software were used. The tests were carried out in accordance with GOST 11262 "Plastic. Tensile test method" at a temperature of  $23 \pm 2$  °C and relative humidity of  $50 \pm 5$  % on samples having the form of "eight" (2nd type).

The research object was polymer composites obtained on the basis of epoxy resin ED-20 (GOST 10587-84) and Etal-247 (TU 2257-247-18826195-07, cured with Etal-1440H (TU 2257-3570-18826195-03), Etal-1460 (TU 2257-010-18826195-99), and Etal-1472 (TU 2257-1472-18826195-05) hardeners produced by JSC "ENPZ EPITAL". The ratio of the components used is presented in Table 1.

**Table 1.** Kind and ratio of the components in the epoxy composite materials compositions

Number of the composition	Epoxy resin brand	Hardener brand	Resin: hardener ratio
1	ED-20	Etal-1440H	100 : 56
2	Etal-247	Etal-1460	100 : 37
3	Etal-247	Etal-1472	100 : 23.75

Climatic researches of the polymer compositions were carried out within one calendar year (365 days). Samples were exposed at full-scale test site of environmental and meteorological laboratory of the National Research Mordovian State University named after N.P. Ogarev (temperate continental climate). Changes of the meteorological parameters were registered by automatic monitoring stations of the atmospheric air, allowing to record the following environmental parameters round the clock: temperature, relative humidity, atmospheric pressure, the wind speed and direction, amount of precipitations, pollutant concentration (carbon monoxide, ozone, nitrogen dioxide, nitrogen oxide, sulfur dioxide, hydrogen sulfide, ammonia), total solar radiation, ultraviolet radiation of A and B ranges [12].

#### 4. The technique for analyzing the changes in fractographs quantitative values of composite material fracture surface

The nature of polymer composite material deformation under mechanical stresses depends on the stress rate, temperature, humidity, chemical composition of polymers, and presence of defects (cracks, chips) on the surface and inside the samples [13-17]. Besides, composite properties change under the influence of various aggressive factors including climate, which affects the resulting fracture surface. [15, 18, 19] works have revealed the correlation between the relative elongation at fracture of epoxy composites and the average values of fracture fractographs brightness, which proves the possibility to detect the grid polymer deformations by colorimetry at the quantitative level. Revealing the interactions between the fractographic characteristics of the fracture surface and strength, strain, and other properties of the polymer composites can be considered as a researches topical field. Thus, special attention at research of the composite materials should be paid to techniques development for quantitative evaluation of the fracture surface fractographs indicators.

Let's examine the fracture surface of the epoxy composite test sample having the composition No. 1 which has not been subjected to climatic aging (Figure 1 a). After analyzing the figure, we see that the mirror (lower right corner of the picture), rough, and transition zones vary greatly by color and brightness, which enables to use colorimetric indicators for detection of the surface topography with subsequent zoning.

In this work, we used the "brightness" value which can be determined by NEXSYS ImageExpert software complex ("Novye ekspertnye sistemy" LLC) and with the help of author software systems to identify the relief of fracture fractographs colorimetric indicators [20, 21]. The results obtained using the software complex "Identification and analysis of porosity of the building materials" [20] are presented below. The source data for the analysis were images of fracture surface obtained with the help of a stereo microscope Olympus SZX-16. The further analysis of the generated data arrays was performed using cartographic methods.

It is known [22] that the visual analysis enables to reproduce the surface relief in a visual form, but the curvature, projections, and illumination add significant error to the resulting image. Another one widely applied group is a group of cartometric methods implemented on the basis of the determining the quantitative characteristics (length, area, height, volume). Besides the visual and cartometric methods, the graphical analysis is applied; this is a technique based on the mathematical analysis and related to statistical characteristics (mean, mode, median, excess, asymmetry). The surface relief can be represented in the several reliefs form of different particularization level [23, 24]. This approach to the image analysis allows to distinguish the objects of different sizes on the fractures that are required for calculation of mirrored and rough zones' areas. The separation procedure is called a topographic surface decomposition [23]. The relief under the study can be divided into the component reliefs:  $A(x, y)$  is a relief of high curvature objects,  $B(x, y)$  is a relief of transition curvature objects,  $C(x, y)$  is a relief of small curvature. Then the total relief  $Z(x, y)$  can be presented as the sum of component reliefs [25]:

$$Z(x, y) = A(x, y) + B(x, y) + C(x, y) \quad (1)$$

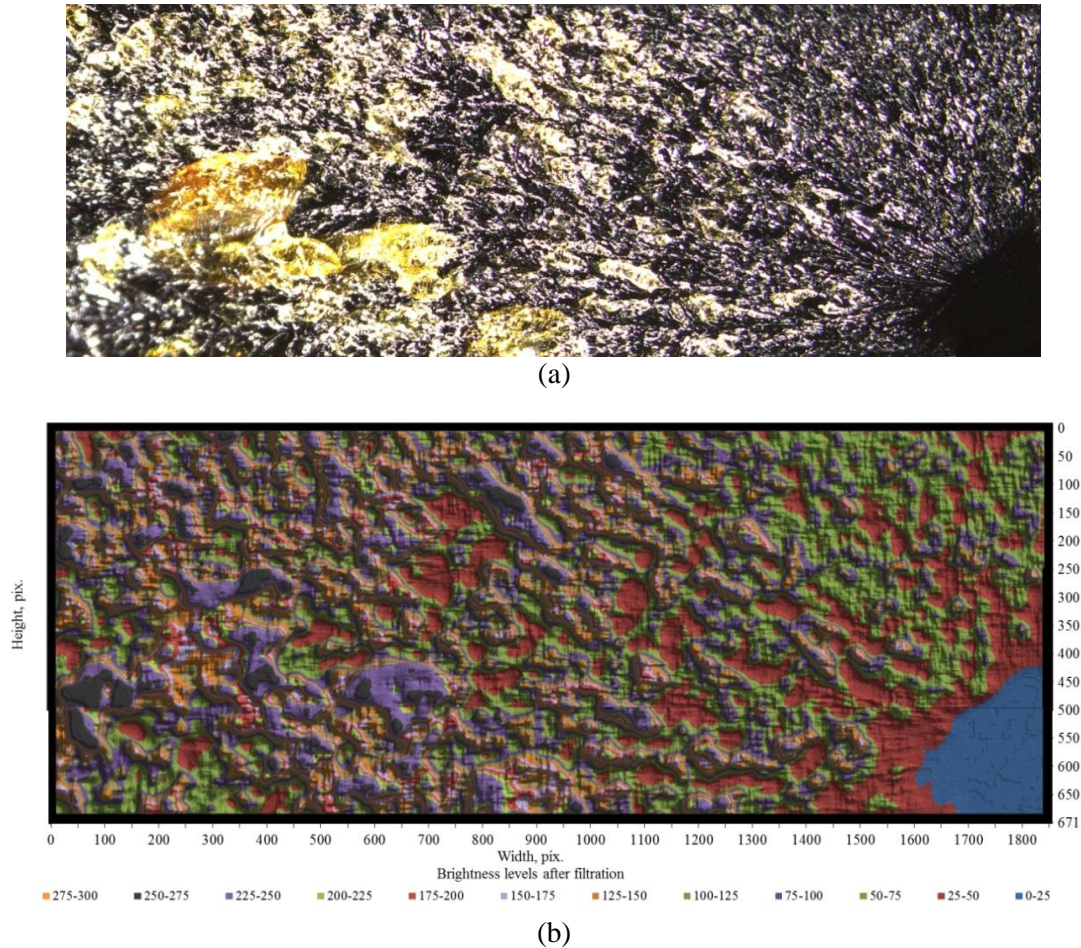
In order to determine the mirror surface on the fracture surfaces, a relief of small curvature objects should be determined  $C(x, y)$ ; this relief is obtained by averaging a point in the region  $S$  using a smoothing linear filter and can be expressed as follows [24]:

$$C(x, y) = \frac{1}{S} \int_S \Phi(x - x_0, y - y_0) Z(x_0, y_0) dx_0 dy_0, \quad (2)$$

where  $\Phi(x - x_0, y - y_0)$  is the core of the integral operator, normalization condition of which is:

$$\frac{1}{S} \int_S \Phi(x - x_0, y - y_0) dx_0 dy_0 = 1,$$

where  $x_0, y_0$  is the coordinates of a point on the original image for which the averaging according to a rectangular window occurs.



**Figure 1.** The fracture surface of the sample with composition No. 1 (a) and the brightness relief obtained by using a smoothing filter of  $25 \times 25$  pixels (b).

Example of the original image decomposition (Figure 1 (a)) of the sample fracture surface with the epoxy composite (composition No. 1) after the use of a smoothing filter of  $25 \times 25$  pixels is shown in Figure 1 (b).

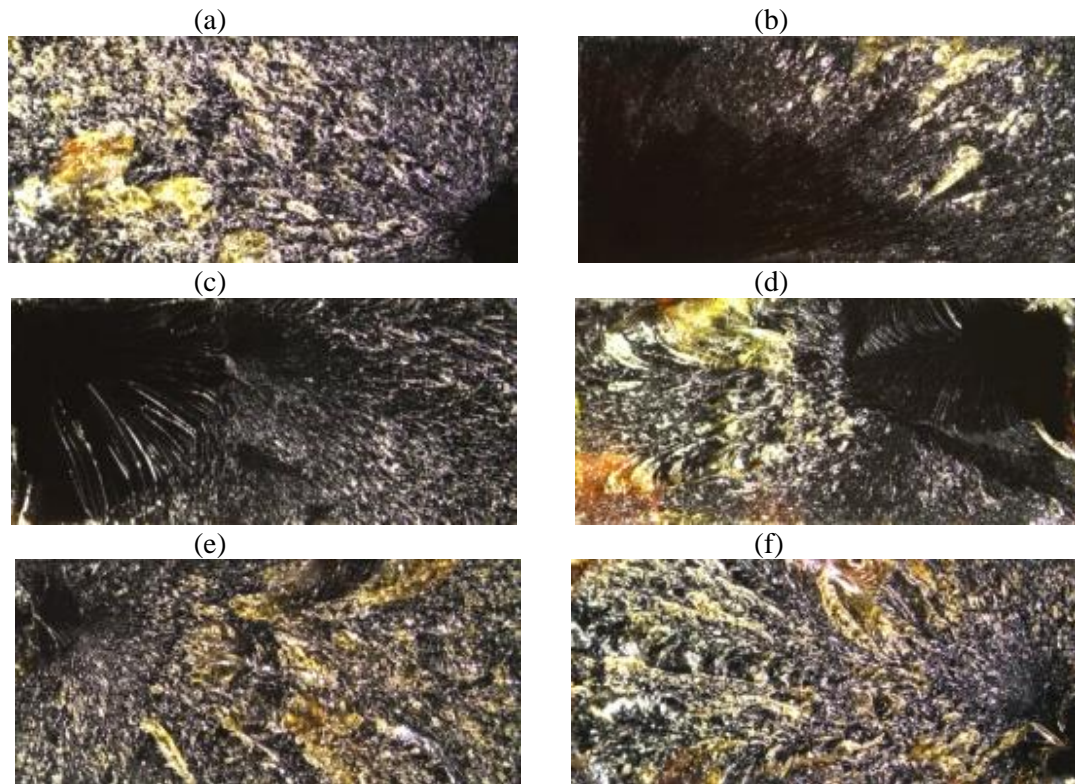
## 5. Results of the study

Numerical analysis results of the fracture surface on three studied compounds (see Table 1) are specified in Table 2. It is revealed that the composition No.1 (ED-20 + Etal-1440H), featuring the greatest reduction of strength properties after a year of in situ exposure (lower tensile strength limit of 34.3%) has (Figure 2, a, b) the greatest increase in the mirror zone area (from 3.1 to 37.2%). Thus, the samples of composition No.3 (Etal-247 + Etal-1472) have shown the highest stability of mechanical properties during the in situ exposure (change in properties after 365 days made only 0.2%), the mirror zone area almost has not changed (2.7 and 3.4%, respectively).

Generalization of the obtained results has shown (Figure 3) that the mirror zone area of ( $S_{mir.zone}$ ) samples is interrelated with the strength properties of the studied compositions at tension ( $\sigma_{tens}$ ) by the exponential dependence

$$\sigma_{tens} = 53.25 \cdot \exp^{-0.012 \cdot S_{mir.zone}} \quad (3)$$

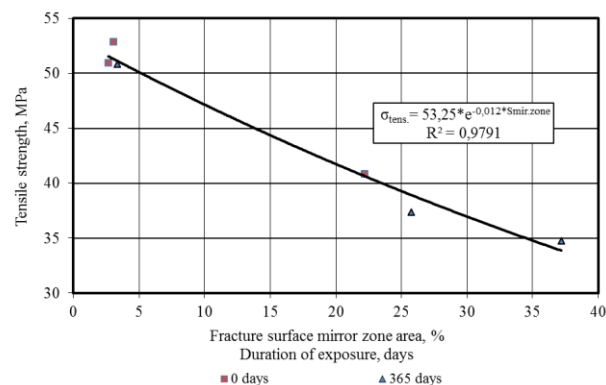




**Figure 2.** Epoxy polymer sample fractured surfaces in the initial state (a, c, e) and after 365 days (b, d, f) of in situ aging in the temperate continental climate: composition No.1 (a, b); composition No.2 (c, d); composition No.3 (e, f).

**Table 2.** Values of tensile strength and square mirror zone areas of the epoxy composite tensile destruction.

Composition Number	Tensile strength, MPa		Mirror zone area, %	
	0 days	365 days	0 days	365 days
<b>1</b>	52.9	34.7	3.1	37.2
<b>2</b>	40.8	37.3	22.2	25.8
<b>3</b>	50.9	50.8	2.7	3.4



**Figure 3.** A correlation between the strength properties of the epoxy composites at tension and the mirror zone area of the fracture surface.

It should be noted that the data presented in Figure 3 can also be approximated by linear and exponential dependencies with quite high accuracy ( $R^2 = 0.964 \div 0.979$ ):

$$\sigma_{tens} = 52.87 - 0.527 \cdot S_{mir,zone} \quad (4)$$

$$\sigma_{tens} = 60.64 \cdot S_{mir,zone}^{-0.145} \quad (5)$$

The range expansion of the studied epoxy composite compositions is required to clarify the kind of a mathematical dependence. Detection of the dependences between the macro properties of the epoxy polymers and the quantitative values of indicators characterizing their fracture mechanism will enhance the understanding of polymer material performance, in particular, under the influence of the environmental factors.

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