Damage accumulation after temperature aging in structural GFRP in interlayer shear tests

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Abstract. A series of quasi-static tests of the structural fiberglass reinforced polymers samples in interlayer shear by the short-beam method were carried out in this work. Mechanical tests were carried out after preliminary temperature aging with different temperature regimes and exposure duration. Diagrams of dependence of the residual interlayer strength degradation on the mode of preliminary aging are constructed. An investigation of the microstructure of GFRP specimens before and after quasi-static tests in interlayer shear was carried out. During the experimental studies the control of the damage accumulation processes and destruction was carried out using the acoustic emission method (AE). Analysis of the AE signal parameters made it possible to assess the prevalence of different destruction types of composites depending on the mode of preliminary aging.

1. Introduction
The conditions of the exploitative environment have a great influence on the destruction processes of composite materials. It is important to evaluate the duration and level of thermal effects in the design of structural elements as well as the features of the material of the real construction. In this regard the problem of studying the features of mechanical behavior, deformation pattern and destruction of structural composites during preliminary temperature aging, thermal moisture aging and moisture saturation in operating (polluting) environments becomes more actuality. In addition, to obtain characteristic dependences of changes in mechanical properties on the corresponding mode of preliminary action is important. [1-6].

Thus, the experimental study of the effect of elevated temperatures on the processes of damage accumulation and destruction of structural FGRP used for the manufacture of critical elements and structures is a relevant intervention of research in the solid mechanics.

2. Experimental procedure
In this work, an experimental study of samples of structural fiberglass/epoxy "STEF" after temperature aging at 120°, 200°С for 5 and 15 days is carried out. Mechanical tests for interlayer shear under static conditions were carried out using the short beam method on the basis of the shared research facilities “Center of Experimental Mechanics” PNRPU. Mechanical tests were carried out with the recommendations of ASTM D2344 on an Instron 5965 electromechanical testing system. The loading rate was 1 mm / min.
In order to study the influence of preliminary temperature aging on the mechanisms of destruction and the stages of damage accumulation in samples of various structural composite materials, the method of acoustic emission (AE) was used in this work [7-11]. The energy of AE was chosen as an informative parameter of the signals \( E, V^2s \). In addition, the values of the frequencies of maximum of spectrum \( F, \text{kHz} \), obtained using the fast Fourier transform, were estimated. AE signals were recorded using the Vallen AMSY-6 system (Germany). A broadband piezoelectric sensor with a frequency range of 300-800 kHz and a preamplifier with a gain of 34 dB were used. Cyanoacrylate glue was used to attach the sensors to the sample surface. A photo of a sample with an AE sensor is shown in figure 1.

The processes of damage accumulation and mechanisms of destruction of GFRP during mechanical loading are studied using the AE method. The expediency of using the frequency characteristics of the AE signal are noted. The most commonly used parameter is the values of the frequencies of maximum of spectrum [9-10, 12-15]. When analyzing these works, it was noted that the authors determined the frequency ranges of the spectrum maximum to describe the main mechanisms of damage accumulation in fiberglass/epoxy (matrix cracking, adhesion between the fiber and the matrix, fiber destruction). It is noted that lower values of the frequency ranges correspond to matrix cracking, high values characterize fiber destruction, and intermediate ones are associated with disruption of adhesion between the fiber and the matrix and delamination.

![Figure 1. A photo of a sample with an AE sensor.](image)

3. Result and discussions

3.1. Mechanical test results

The results of mechanical tests of structural fiberglass/epoxy samples in interlayer shear using the short beam method are shown in figure 2. An increasing dependence of the change in the residual ultimate strength during interlayer shear on the selected mode of preliminary temperature aging to the group of samples without thermal exposure (nominal) is noted.

Studies of the microstructure after temperature aging of the fiberglass/epoxy “STEF” samples before and after interlayer shear tests showed that at 120 °C - 5 days/15 days the matrix color changes to more yellow (5 days) and brown (15 days), there are no visible structural defects before testing. At 200 °C - 5 days/15 days the matrix color change to dark gray/black, defects in the form of surface cracks appear along the fiber-matrix boundaries.
After testing the samples of fiberglass/epoxy “STEF”, it can be noted that for a group of samples 120 °C - 5 days a macrocrack is formed due to shear of the layers. At 120 °C - 15 days, the destruction of the binder leads to the formation of many interlayer cracks, the development of which leads to the destruction of the sample. At 200 °C - 5 and 15 days, destruction and local brittleness of the binder and the formation of micro cracks occur, followed by shear failure of the samples. However, it should be noted that temperature aging lead to an increase in stiffness and an increase in strength values at interlayer shear by 15-30%, depending on the mode (figure 2).

![Figure 2](image)

**Figure 2.** Dependences of the change in the residual tensile strength at interlayer shear on the selected pre-exposure shear on the selected pre-exposure mode for fiberglass/epoxy “STEF” specimens.

![Image](image)

**Figure 3.** Structure of fiberglass/epoxy specimens after temperature aging under different mode before and after tests.

3.2. Acoustic Emission Signal Processing

To analyze the effect of preliminary temperature aging on the damage accumulation processes in samples of structural fiberglass, graphs of the distribution of the values of the frequencies of maximum of spectrum for all groups of samples under static load were plotted (figure 4).
For the “STEF” samples, it was noted that the values of the frequencies of maximum of spectrum can be divided into three ranges (25-80 kHz, 270-330 kHz and > 650 kHz). For samples tested after preliminary aging at a temperature of 200 °C and holding for 5 and 15 days, the largest number of recorded signals was recorded in the low frequency range (25-80 kHz). For the samples tested after preliminary aging at a temperature of 120 °C and holding for 5 and 15 days, the largest number of registered signals was recorded in the middle frequency range (≈ 300 kHz).

**Figure 4.** Graphs of the distribution of the values of the frequencies of maximum of spectrum for all groups of samples under static load.

### 4. Conclusions

New experimental data that illustrate the effect of preliminary temperature aging at elevated temperatures on the staging of damage accumulation in fiberglass samples during static tests in interlayer shear by the short beam method have been obtained. Non-monotonic dependences of the change in the residual ultimate strength during interlayer shear on the selected mode of preliminary temperature aging are noted. The most dangerous modes of temperature aging are determined, which lead to a change in the mechanisms of destruction and accumulation of damage and limit the implementation of the deformation and strength properties of fiberglass/epoxy “STEF”. Was noted that the values of the frequencies of maximum of spectrum for all samples can be divided into three ranges and related to the main mechanisms of destruction in composites.

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