Temperature aging effects on mechanical behavior of structural GFRP on interlaminar shear tests

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Abstract. Aging of polymer composites is a wide-spread problem, which causes deterioration of mechanical properties, shorter design service life of structures and their potential early destruction. The work presents the results of experimental researches, within which the impact of elevated temperatures on mechanical properties of airborne constructional fiberglass were investigated. Research results were obtained, which describe the impact of the preliminary temperature aging (in different modes) on mechanical properties, behavior and execution of different composite destruction mechanisms during interlaminar shear testing. Experimental data were obtained, which describe the temperature impact on the mechanical behavior of constructional fiberglass samples at interlaminar shear. Regularities of fiberglass properties changing after temperature aging at interlaminar shear were analyzed, based on the joint use of testing systems and an acoustic emission (AE) signals recording system. Dependences of AE signal energy parameters on the time, combined with diagrams of fiberglass samples loading were obtained. Results of the distribution of specific frequency bands obtained after the destruction of samples with different preliminary aging modes, are graphically represented.

1. Introduction

The study and analysis of the impact of elevated and lowered (service) temperatures on the mechanical properties and destruction mechanisms of composite materials is an essential line of research [1-2]. An important task is to determine temperature dependences of elastic and strength properties of polymer composites, which are used in critical structures [3-4]. The great attention is paid to the study of processes related to the degradation of mechanical properties of construction materials during their temperature aging [5].

For forecasting the service terms of structures made of polymer composites, an essential line of research is the study of issues related to aging of polymer composite materials. Aging of polymer composites is a wide-spread problem, which causes deterioration of mechanical properties, shorter design service life of structures and their potential early destruction. The problem of polymer composite aging in in aqueous media is currently being investigated all over the world [6-8]. The structures made of polymer composites are mostly impacted by atmospheric factors (temperature, humidity, solar radiation, temperature cycling, tropical and marine climate, etc.), which influence their physical, chemical and mechanical properties [9-17].

An acoustic emission (AE) method is used as an additional diagnostic method for preliminarily temperature-aged materials, which also evaluates their structural integrity [5]. Nowadays, this method is commonly used for investigations of material defect initiating and expanding processes. The analysis of AE signals allows evaluating of mechanisms of the damage accumulation in composite materials under loading. Besides, this method is used in some scientific works aiming to study the impact of
preliminary temperature aging on composite materials [18-21]. In this work, the AE method is used for the study and evaluation of the impact of different temperature aging modes on fiberglass samples.

The work includes a series of mechanical interlaminar shear tests of fiberglass samples based on the prepreg VPS-48 and epoxy binder; these tests were carried out under normal and elevated temperatures 22º, 120º, 160º, 200ºС, and after preliminary temperature aging at the same temperatures. The purpose of the work is obtaining of new experimental data related to changing of mechanical properties of constructional fiberglass under preliminary and direct impact of elevated (service) temperatures and interlaminar shear conditions.

2. Experimental procedure

All the tests were carried out at the Resource Sharing Center “Center of Experimental Mechanics” of Perm National Research Polytechnic University. Mechanical short beam shear tests (for interlaminar shear) under normal temperature were carried out considering recommendations provided by GOST 32659-2014, using an Instron 5965 electromechanical system, while tests under elevated temperatures were carried out using an Instron 5882 electromechanical system, which includes a temperature chamber with an operation temperature range of -100 to +350ºС. The testing loading speed was 1 mm/min for all sample groups.

The preliminary temperature aging of unloaded fiberglass samples was carried out in the temperature chamber, according to modes presented in Table 1. After the temperature aging, the groups of samples were tested under normal temperature.

| Exposure temperature, °C | No aging | 120 | 120 | 200 | 200 | 200 |
|--------------------------|----------|-----|-----|-----|-----|-----|
| Number of days           | -        | 5   | 15  | 5   | 15  | 15  |
| Number of samples        | 5 samples of each group |

After the preliminary temperature aging, the samples were tested using an AE signal recording system AMSY-6 from the test start and till the complete sample destruction. Broad-band piezoelectric sensors M31 (frequency band 300-800 kHz) and an amplifier (gain 34 dB) were used. The sensors were fixed on the samples using glue. A photograph of a sample equipped with an AE sensor is presented in a figure 1.

![Figure 1. A photograph of a sample equipped with an AE sensor](image-url)
3. Results and discussion

3.1. Mechanical test results

Results of the interlaminar shear tests carried out under different temperatures are presented in Figure 2 as loading diagrams and sample destruction modes. It is stated that during tests carried out at normal and elevated 120°C (temperature), the samples were destroyed according to the “stretching” mode (according to the classification of destruction modes provided by GOST 32659-2014); under elevated test temperature 160°C, the samples were destroyed according to the “single shear” and “multi-shear” modes. Under test temperature 200°C the samples were destroyed according to the “plastic shear” mode. The tests at the temperature 22°C were carried out jointly with AE signals recording. After temperature elevation to 120°C, values of interlaminar shear strength reduce by 36%, at the temperature 160°C they reduce by 57%, and at 200°C – by 90%.

Based on the obtained experimental data, loading diagrams were developed for all groups of fiberglass samples after the preliminary temperature aging (Fig. 3, a). A photograph of the destroyed samples is presented in Fig. 3, b. Table 2 gives average values with specified statistical dispersions.

![Figure 2](image1.png)

**Figure 2.** Results of mechanical interlaminar shear tests (short beam shear tests) of fiberglass samples VPS-48 at normal and elevated temperatures: (a) – 22°C; (b) – 120°C; (c) – 160°C; (d) – 200°C

![Figure 3](image2.png)

**Figure 3.** Typical loading diagrams for all groups of fiberglass samples VPS-48 during interlaminar shear testing after the preliminary temperature aging (a), a photograph of the destroyed samples (b)
The loading diagrams (figure 3.a) of fiberglass sample groups after the temperature aging at different modes show monotonic (from colder and shorter modes to hotter and longer ones) stiffening after aging. For 120-5 and 120-15 (°C-days) modes it is explained by the binder depolymerization. The samples turn more intensive and yellow. For 200-5 and 200-15 (°C-days) modes, this effect can be explained by the low-temperature polymer carbonation in the air; the samples turn brown and dark-brown, relatively.

For samples aged at the 200-15 mode, the destruction mode is changed (figure 3.b). the samples are destroyed due to the interlaminar lamination. Maximum shearing stresses were not reduced for the samples aged at the 120-5, 120-15, and 200-5 modes compared to the tested non-aged samples; the distinction between vales is within the statistical dispersion (table 2). However, it can be noted that after all aging modes, the material stiffness increases (figure 3.a).

### Table 2. The average values of the interlaminar shear strength for all groups of samples

| №  | Interlaminar shear strength, MPa | Test conditions and preliminary aging modes, °C/°C-days |
|----|---------------------------------|--------------------------------------------------------|
| 1  | 61.7±4.5                      | 22/ no aging                                        |
| 2  | 39.5±2.5                      | 120/ no aging                                      |
| 3  | 26.5±0.9                      | 160/ no aging                                      |
| 4  | 6.1±0.5                       | 200/ no aging                                      |
| 5  | 63.3±4.1                      | 22/120-5                                            |
| 6  | 62.4±4.1                      | 22/120-15                                           |
| 7  | 64.7±3.7                      | 22/200-5                                            |
| 8  | 58.0±6.1                      | 22/200-15                                           |

#### 3.2. Acoustic Emission Signal Processing

In this work, values of energy parameter (E, V^2/s) and spectral maximum frequency (F, kHz) were used as an informative parameter of AE signals [22-23]. The values of spectral maximum frequency were obtained using a special software unit and the fast-Fourier-transform algorithm.

Figure 4 presents diagrams of the temporal dependence of AE signal energy parameter; these diagrams are combined with loading diagrams of the samples, which were not preliminarily temperature-aged (a), and samples after the preliminary temperature aging at different modes and (120-5 – b, 120-15 – c, 200-5 – d, 200-15 – e). The diagrams of the energy parameter dependence characterize processes of the material damage accumulation and allow evaluating of impact of different temperature ranges on the fiberglass samples. For all samples, we can note that energy parameter recording starts after reaching a peak loading value. Hereby, the highest energy parameter values are observed for the non-aged samples, and the lowest values – for the samples, which were impacted by 200º C during 15 days. This indicates that this temperature aging level influences the material structure more significant than other aging modes. For the samples, which were at the preliminarily temperature-aged 120-5, 120-15, 200-5 modes, the energy parameter values are slightly lower than for the non-aged samples.
Figure 4. Diagrams of the temporal dependence of AE signal energy parameter which combined with loading diagrams of the samples: (a) – 22ºС; (b) – 120-5; (c) – 120-15; (d) – 200-5, (d) – 200-15

Figure 5 shows the distribution of specific bandwidths of the spectral maximum frequencies, which were obtained after the destruction of samples with different preliminary aging levels. The distribution of the frequency bandwidths changes depending on aging temperatures. This is caused by changing of binder properties and in damage accumulation modes depending on temperature aging modes. During the distribution data analysis, we can observe the similarity of the bandwidths obtained for the samples aged under the 120-5 and 120-15 modes, and for the samples aged under the 200-5 and 200-15 modes.
Figure 5. Distribution of specific bandwidths of the spectral maximum frequencies after different preliminary aging levels

4. Conclusions
Thus, we have obtained the new experimental data, which describe the impact of the elevated and lowered (service) temperatures under the interlaminar shear on the constructional fiberglass samples. The stiffness of the samples and their destruction modes change depending on a temperature aging mode. The impact of the preliminary temperature aging is confirmed during analysis of the acoustic emission signals.

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