Influence of Atmospheric Plasma on Mechanical Strength Fiber Reinforced Geopolymer Composite

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Abstract: Improving the adhesion of the matrix to the reinforcement brings an increase in the quality of the composite interface and, at the same time, improves the mechanical properties of the composite system represented by the geopolymer matrix reinforced with chopped fibers. The article's topic is to monitor the effect of plasma surface treatment of mineral fibers on improving the adhesion of the geopolymer matrix to reinforcing fibers by examining the mechanical properties of the composite. The experiment was performed with glass fibers of type AR, E, R, and basalt fibers. A bending test was used to monitor changes in mechanical properties. In the research, we assumed that the plasma surface treatment of fibers should improve the overall mechanical properties of the composite system. Special attention was paid to the statistical evaluation of the results (Horn method for the analysis of small data files).

Keywords: mineral fibers; atmospheric plasma; geopolymer matrix; flexural strength; interface.

1 Introduction

A characteristic manifestation of the composite material is the synergistic effect, which arises from the combination of the continuous and dispersed components of the composite. The essence of the synergistic effect is creating an interface. It is the transition phase forms between the matrix (continuous phase) and the reinforcement (dispersion phase). The strength of the interphase determines the magnitude and character of the stress transfer from the matrix to the reinforcement; it includes the properties of the partial phases and adhesion [1].

The adhesion between the materials determines the quality of the interface in composite materials and directly contributes to the resulting composite properties. It is formed by a set of mechanisms, including absorption and wetting, electrostatic interactions, reaction bonds, and unbound interactions [2–4]. Glass and basalt fibers belong to the group of mineral fibers. Chopped fibers, continuous fibers (rovings), textiles: warp knitted (multiaxial fabrics, geogrids), braided sleeves and tapes, fabrics with plain and twill weave, hybrid fabrics, and three-dimensional woven fabrics are used as reinforcement for composites. Glass and basalt fibers have very similar densities, strengths, melting points and the technical application in the production of composites [5].

The surface treatment of fibers is a commercially applied functional coating (sizing), which simultaneously meets the requirements for protection and fibers processability and the formation of a chemical bond between the fiber and usually the polymer matrix. Organosilanes are used for the surface treatment of glass fibers [6]. These are compounds that contain silicon in their structure and function as adhesives. They have excellent reactivity with silicon-containing substances, e.g., aluminosilicates [7].

Plasma is an electrically conductive medium that contains positively and negatively charged particles, neutral atoms, and molecules. The high concentration of charged particles created by ionization, with extremely high energy compared to liquid, gaseous and solid states, forms the so-called fourth state of matter – plasma [8, 9]. Cold atmospheric plasma is a type of plasma that is often used in practice. Its advantage lies in the very high electron temperature of the plasma itself and the relatively low temperature of the gas. As a result, charged particles present in the plasma can interact with the surface of the substrate or energetically activate reactive gas molecules [8, 10].

The presented research aims to characterize the properties of composite materials after surface modification of glass and basalt fibers using atmospheric plasma. A partial goal of the presented study is to process, evaluate and compare the achieved results statistically.

2 Experiment

2.1 Used materials

The following were used to produce composite samples: BAUCIS LK cement (Czech Republic) activated with potassium water glass; silica sand was used as a filler. The components were mixed in a 1:1:0.9 ratio; was added 2 wt.% fibers:

- chopped fibers type AR (ARGF): Cem-Fil, 2400 tex, density 2.68 g/cm^3, fiber diameter 14 μm, length 12 mm;
- chopped fibers type E (EGF): manufacturer Johns Manville, PR440 2400 907, 2400 tex, density 2.6 g/cm^3, fiber diameter 16 μm, length 12 mm;
- chopped fibers type R (RGF): 2400 tex, density 2.6 g/cm^3, fiber diameter 12 μm, length 12 mm;
- chopped strands (BF): manufacturer Deutsche Basalt Faser GmbH. 2400 tex, density 2.6–2.8 g/cm^3, fiber diameter 13 μm, length 12 mm.

Samples with dimensions $10\times10\times100$ mm^3 were prepared for the experiments. Four sets of fiber-reinforced composite samples were prepared as reference samples (REF), each set containing ten pieces of samples. The surface of the mineral fibers was modified with atmospheric plasma for about 3 minutes, and then the fibers were used to prepare composite samples. Each set of samples contained 30 samples. The time required for the maturation of the geopolymer composite materials is from 28 to 30 days.

2.2 Used methods

A Piezobrush®PZ2-i from Relyon-plasma GmbH was used to modify the surface of the fibers. The purpose of the modification is to activate the surface of the fiber. Improved interface quality ensures better adhesion of the geopolymer matrix to the fiber and strengthens the bond between the composite components.

To evaluate the achieved properties of the composite. The flexural strength test was performed according to the standard ČSN EN 14617-2 Artificial stone – Test methods – Part 2: Determination of flexural strength.

The change in the composite's properties was observed on the reference samples at various time intervals – after 30 days of maturation (P30), after three months (P120), and after half a year (P210) from the end of the maturing period. The measurement results were used to determine the degree of plasma treatment of the surface of fiber reinforcement and assess the improvement/deterioration of the interface quality.

The flexural strength was calculated according to Eq. 1:

$$\sigma_F = \frac{3}{2}\frac{F_{\max}\cdot L}{bh^2}\ [\text{MPa}], \tag{1}$$

where: σ_F – flexural strength [MPa]; $F_{\max}$ – maximal force [N], L – support span [m], b – width [mm], h – thickness [mm].

The retention ratio of flexural strength was evaluated according to Eq. 2. Calculations prescribe a Standard CSN EN 14617-2 Artificial stone - Test methods - Part 2: Determination of flexural strength [11].

$$\Delta R_F = \frac{R_{F0} - R_F}{R_{F0}}\,100\%, \tag{2}$$

where: ΔR_F – retention ratio [%], R_{F0} – reference value of strength [MPa], R_F – strength value after time exposure [MPa].

3 Results

The obtained results, including statistical data evaluation, are shown in Fig. 1. The research involves groups of samples with a small number of measurements, so the Horn method for the analysis of small data sets was used to evaluate the results. The result obtained from AR-type glass fiber composites after 30 days of maturation shows an increase in flexural strength of 80%. After 90 days (P120), there was approximately 48% flexural strength value. After the next 90 days (P210), flexural strength increased by 33%. The percentage increase was calculated according to Eq 2. The results, including statistical evaluation, show that plasma modification's positive effect on improving the mechanical properties of other types of fibers has not been demonstrated. The different time intervals (30, 90, and 180 days) did not affect the investigated mechanical properties.

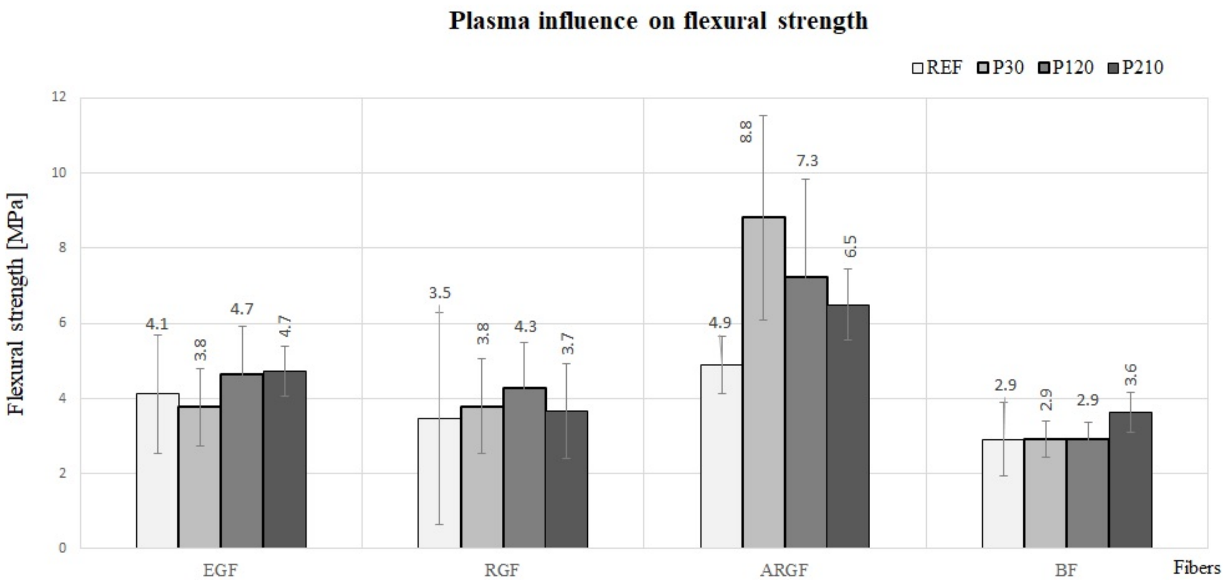

Fig. 1: Comparison of flexural strength results for reference samples and plasma-modified reinforcement.

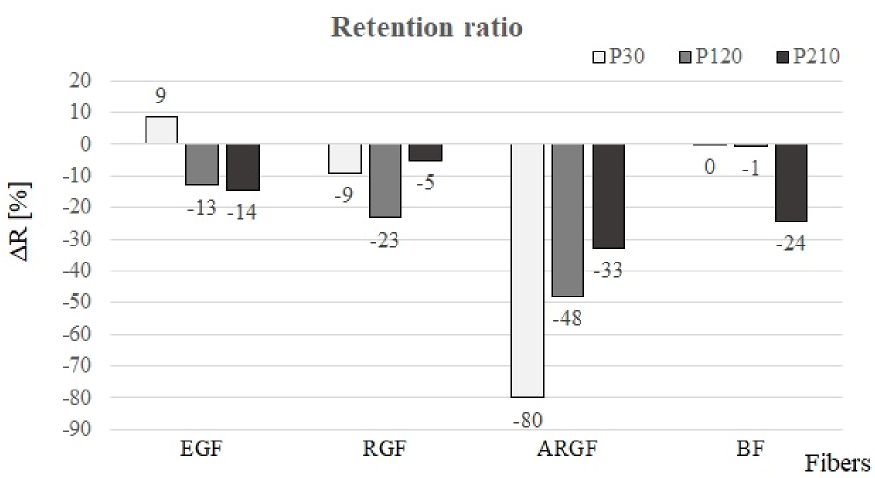

Fig. 2: Retention ratios for all type of mineral fibers after six months from preparation of samples.

The standard prescribes the evaluation of the bending test results by calculating the retention ratio (Eq. 2). The results are presented in the graph in Fig. 2. The calculated values show an increase in flexural strength of ARGF by 80% after a maturation period of 30 days against the reference value. After 90 days (P120), there was a decrease of approximately 18% in the flexural strength value against the value measured after 30 days (P30). After the next 90 days (P210), flexural strength decreased 26% against value P30. Flexural strength of BF increased after 210 days by 24%. The values of the retention ratios for exposure times P30 (0%) and P120 (1%) show no changes.

Fig. 3 and 4 show the amount of geopolymer matrix connected to the reinforcing fibers' surface. Despite plasma treatment, basalt fibers (Fig. 3) have little adhesion to the GP matrix. The surface of the fibers is clean and smooth, and the amount of matrix on the surface of the fibers is minimal to negligible. The exposure time (three and six months) had no significant influence on the adhesion of the fibers to the matrix. In the case of AR-type glass fibers (Fig. 4), an increased amount of geopolymeric material can be observed on the surface of the fibers. It indicates an excellent adhesion to the matrix even after a time delay. The results of microscopy

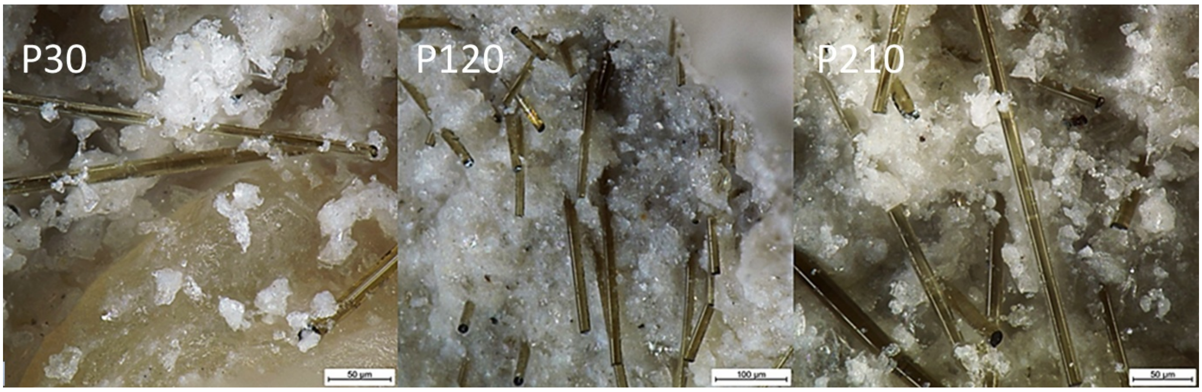

Fig. 3: Representation of basalt fiber surfaces after plasma modification (P30), after 3 (P 120) and 6 months (P210).

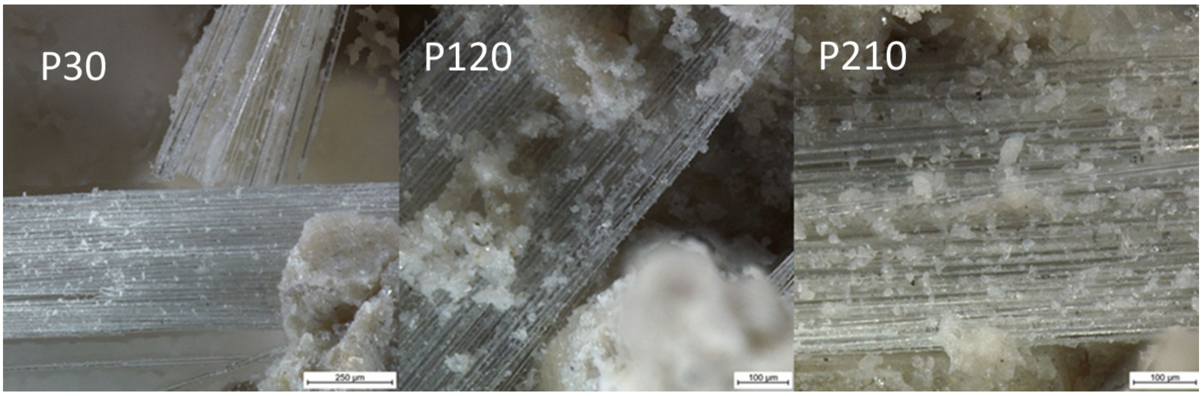

Fig. 4: Representation of AR-glass fiber surfaces after plasma modification (P30), after 3 (P 120) and 6 months (P210).

observation correspond to the results obtained in the flexural strength test; plasma treatment has been shown to affect the strength of the AR-type glass fiber reinforced geopolymer composite.

4 Conclusion

Atmospheric plasma surface treatment was used for mineral-type fibers, and a flexural strength test was performed after the maturation period. Plasma can be expected to roughen the fiber surfaces, which increases the contact areas and would result in increased adhesion between materials. The evaluation of the results showed a significant improvement in the flexural strength only of the geopolymer reinforced with glass fibers of the AR-type (with plasma surface treatment). For the other types of fibers, the improvement in the average flexural strength value between untreated and modified fiber reinforcement is statistically inconclusive. Therefore, it can be said that the atmospheric plasma surface treatment of glass (E-type and R-type) and basalt fibers did not lead to an increase in the flexural strength of the geopolymer composite system.

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