Interfacial shear strength enhancement of polymeric macro-fibers via plasma treatment

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Abstract

This work deals with a utilization of a plasma treatment of polymeric macro-fibers used as reinforcement in the cement composites. Commercial fibers BeneSteel were plasma treated to reduce their weaknesses – smooth and chemically inert surfaces and thus to enhance an interfacial shear strength between them and the cement matrix. The low-pressure cold oxygen plasma treatment was done for both physical (roughening) and chemical (surface activation via active polar groups) surface modifications. A plasma exposition time differed between 5 and 480 seconds, while the surface changes were observed. It was shown that the most effective treatment time was equal to 30 seconds, as proven by a wettability measurement between fibers and demineralised water. To determine the interfacial shear strength between reference and chosen modified fibers, pull out tests from the cement matrix was performed. Finally, thus treated fibers were used as the reinforcement in concrete composites. Concrete samples having dimension equal to 100 × 100 × 400 mm were tested in the three-point bending test. We focused especially on the post-cracking response of tested samples. The results showed that the post-cracking residual flexural strength of samples reinforced with plasma treated fibers was higher by 30 %. It was find out that the utilization of plasma treated fibers is more effective if compared to reference ones. Copyright © 2017 VBRI Press.

Keywords: Plasma treatment, polymeric macro-fibers, cement composites.

Introduction

Nowadays, polymer fibers are used in civil engineering mainly as additional reinforcement of flat elements. Polymer fibers replace welded nets and steel wires in concrete floors and concrete slab, where redistribution of stress is making full use of plate strength at minimal cost. The use of polymer fibers in floor screeds is also very economical and very effective. Polymer fibers are very effective in impact-resistant prefabricated or complex composite prefabrications where the difficult or impossible positioning of welded nets is. Polymer fibers do not corrode and do not require cover layers [**1**].

Applications of polymer fiber reinforcement with cement matrixes are limited due to their poor interaction with matrix of the composite material. The polymer fibers have a circular shape, smooth surface, and a low cohesion with the cement matrix [**2**].

The purpose of fiber surface modification is to improve the cohesion with cement matrix and thereby reduce anchorage length of fibers i.e. reduction of mass and length of fibers used in the composites. In this work, we focused on oxygen plasma treatment of polymer fibers, namely BeneSteel 55. The plasma, an ionized gas composed of electrons, ions and neutral species, interacts with the surface of fibers [**3**]. This progressive modification is responsible for both, the chemical (interaction with radicals of working gas, O_2) and the physical (interaction with energetic particles) surface changes of thus treated fibers. The cohesion given by chemical bonds between treated fiber surfaces and reinforced matrix is enhanced by fiber surface activation (application of polar or chemical active groups responsible for wettability increase), while the physical cohesions is improved by surface roughening (morphology changes) [**4, 5**]. The plasma treatment was successfully used for surface modification of various (glass, carbon etc.) fiber materials [**6-9**]. We studied morphology changes by SEM, surface activation by contact angle and mechanical changes by cohesion between fibers and cement matrix [**10**].

Experimental

Materials and samples

We used commercial fiber BeneSteel 55 made from a special mixture of polypropylene and polyethylene. Material parameters were as follows: 480 µm diameter, 55 mm length, 5.17 GPa modulus of elasticity, 610 MPa tensile strength, 915 kgm^{-3} density.

The prismatic cement-paste samples for pull-out tests were made from Portland cement CEM I 42.5R based on a silicate clinker with gypsum additives. Water to cement ratio was equal to 0.4. The samples had the dimension equal to 25 × 25 × 20 mm and an embedded length of fibers was 25 mm (approx. a half of the fiber length). The samples were stored in a laboratory environment at 22 ± 1 °C and relative humidity 50 ± 2 % for 28 days.

The samples using for bending tests were composed CEM II 42.5 R, three types of aggregates, a plasticizer and fibers. The blended Portland cement was used because of its slower development of initial strength and for reducing the risk of technological cracks. The amount of cement was 490 kg/m^3. The water to cement ratio of these samples was set at 0.33. The amount of the coarse aggregate (fraction 8-16) was 745 kg/m^3, the medium aggregate (fraction 4-8) 100 kg/m^3 and the fine aggregate (fraction 0-4) 890 kg/m^3. The fiber amount was 0.75 % of the concrete mixture volume. The applied amount of fibers deteriorated the workability of the mixture. Therefore, the plasticizer additive was used. Prepared mixtures were cast in the rectangular mold having dimensions equal to 100 × 100 × 400 mm. After casting, these specimens were kept in the molds for 24 hours at room temperature of 22 °C. It was made six samples from each mixture. After demolding, these specimens were stored in a laboratory environment at 22 ± 1 °C and relative humidity 50 ± 2 % for 28 days.

Experimental methods

The low-temperature oxygen plasma treatment was done for the surface modification of tested fibers. The treatment was performed in the inductively coupled plasma system using RF Tesla VT 214 device. During treatment process, the driver power was 100 W. The base pressure in the chamber was 20 Pa. The O_2 flow was 50 sccm under pressure 50 Pa. Seven different times of plasma treatment were studied (5, 10, 30, 60, 120, 240 and 480 seconds) together with untreated samples. Fibers were weighed before and after treatment using laboratory scales Kern ALJ 120-4 (d = 0.1 mg).

The physical changes onto fiber surfaces were improved by the surface roughening (morphology changes), which it was observed and assessed by means of the scanning electron microscope (ZEISS Merlin at the University Centre for Energy Efficient Buildings, CTU in Prague).

The chemical changes were evaluated by a mutual interaction between fibers and demineralized water. Simply said, the fiber wettability by water is an indicator of a chemical interaction rate between fiber and cement matrix due to water presence in the cement paste. Contact angles between the two phases were measured using the horizontal optical set, according to [**11**].

The fiber pull-out tests from the cement matrix were performed to determine the bond and the adhesion between the two materials directly. The pull-out tests were carried out using the Web Tiv Ravestein FP 100 loading frame. Two sets of (reference and 30 seconds treated fibers) were tested to determine the effect of plasma treatment on fibers coherence with the cement matrix, while each set was represented by six samples in order to obtain statistically relevant results. The testing was displacement controlled at a constant rate of 2 mm/min with the maximum force equal to 60 N.

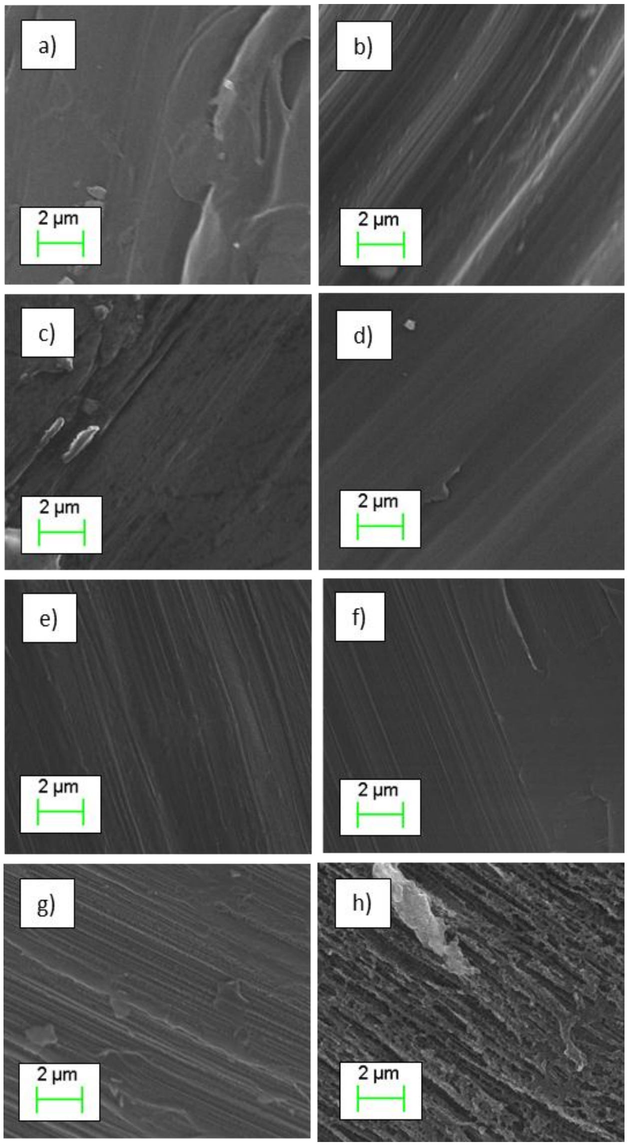

Fig. 1. SEM images of the modified fibers as a function of treatment time. (a)reference sample, (b) 5 second treatment, (c) 10 second treatment, (d) 30 second treatment, (e) 60 second treatment, (f) 120 second treatment, (g) 240 second treatment, (h) 480 second, scale bar is 2 µm.

Results and discussion

Table 1 shows the influence of the plasma treatment on fibers, namely the loss of weight and the contact angle changes. It can be seen, that plasma treatment up to 10 seconds have no effect on the fiber weight loss and more than 10 seconds of plasma treatment did not provide the better effect on the contact angles. Based on these findings, 30 seconds plasma treatment appears to be the

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best choice for next tests. **Fig. 1** shows SEM images of plasma treated fibers. The SEM images (**Fig. 1a** – **Fig. 1h**) show that prolonged treatment time resulted in deepening of longitudinal grooves originally presented (see **Fig. 1a**) on untreated fibers. No visible changes were seen between SEM images **Fig. 1a** – **Fig. 1d**. The SEM images **Fig. 1e** – **Fig. 1g** had grooves and the SEM image **Fig. 1h** had approximately two times deeper grooves than the **Fig. 1g**.

Table 1. Summary of plasma treatment duration and influence of plasma treatment on macroscopic properties of fibers.

Treatment duration [sec.]	Loss of weight [g]	Highest temperature in the chamber [°C]	Contact angle [°]
0 (Ref)	0	-	82.6
5	0	28.1	41.9
10	0	30.3	34.1
30	0.0008	35.7	36.9
60	0.0021	40.1	36.7
120	0.0043	45.2	41.4
240	0.0095	50.8	36.8
480	0.0229	56.1	34.5

The results of pull-out tests are imagined in the **Fig. 2**. It can be clearly said that the bond (before debonding of the fibers from the cement matrix) and the adhesion between the two materials (after debonding) were significantly increased.

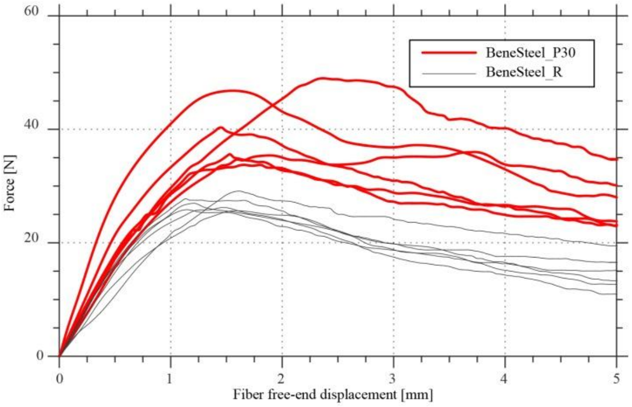

Fig. 2. Force-displacement diagram of cement samples reinforced with reference and 30 seconds plasma treated fibers.

The shear stress was evaluated from the size of the force and shear area in two cases. In the first case, shear stress was evaluated from the maximum force (τ_{max}) and in the second case, shear stress was evaluated from the force when displacement was equal to 3.5mm ($\tau_{3.5}$). **Fig. 3** shows values of shear stress between fiber and cement matrix. It can be stated that 30 seconds plasma treated fibers had approximately 2 times larger value of shear stress than references fibers and between 30 seconds plasma treatment and 480 seconds plasma treatment, a minimal change of the shear stress ($\tau_{3.5}$) occurred. Differences between the values were in the size of the standard deviations. The samples with 30 second and 480 seconds plasma treatment fiber had approximately the same value of the shear stress for maximum force.

From this reason, 30 seconds plasma treatment is economically more advantageous and 30 seconds plasma treated fibers were used in the last experiment on concrete samples.

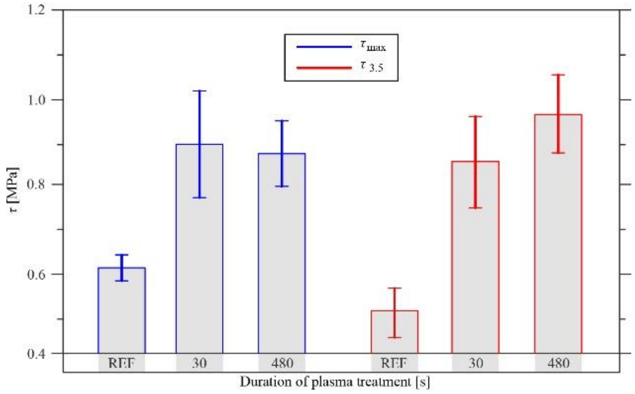

Fig. 3. Comparison of the shear stress (with standard deviation).

The result of bending test was the force and the displacement curve. **Fig. 4** shows average curve calculated from the bending tests of the samples containing the BeneSteel reference fibers (BeneSteel_R) and 30 seconds plasma treated fibers (BeneSteel_30). The force required to reach the elastic limit was 17.79 ± 0.89 kN in the case of samples with references fibers and 17.85 ± 1.16 kN in the case of samples with 30 seconds plasma treatment fibers. After cracking, the sample with references fibers was able to carry about one-third of the maximum load. The bending material exhibits elastic-plastic behavior with reinforcement. By comparing the average curves, it can be seen that the post-cracking residual flexural strength of samples with the modified fiber is higher in plastic phase by about 30%. The results obtained correspond with the outputs of the pull-out tests.

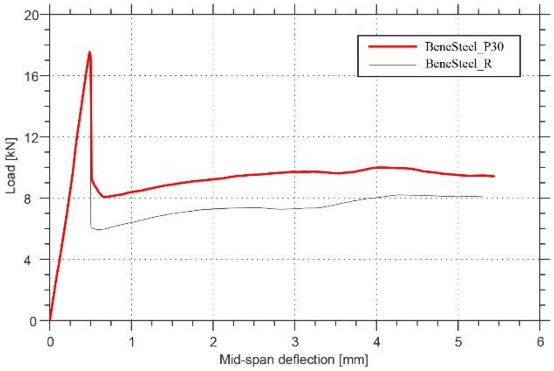

Fig. 4. Force-displacement diagram of concrete samples reinforced with reference and 30 seconds plasma treated fibers.

Conclusion

This work was focused on the influence of the plasma treatment on cohesion between the surface of fiber and cement matrix. The researched fibers were BeneSteel 55 made from a special mixture of polypropylene and polyethylene and having 480 µm diameter, 55 mm length. The surface of fibers was modificated by low-temperature oxygen plasma treatment. Seven different times of plasma treatment were studied (5, 10, 30, 60, 120, 240 and 480 seconds) together with untreated samples. Several experimental tests were applied for determination of influence of plasma treatment on cohesion between fiber surfaces and cement matrix, like electron microscopy for determining roughness of surface, determining contact angel for improving chemical interaction between fiber and cement matrix, pull-out tests for determining maximum shear stress between fiber and cement matrix and bending test for reveal the practical significance of the plasma modifications. Based on the results, it can be concluded that:

- more than 10 seconds of plasma treatment did not provide the better effect on the contact angles
- prolonged treatment time resulted in deepening of longitudinal grooves
- 480 seconds plasma treated fibers had approximately two times deeper grooves than the 240 seconds plasma treated fibers
- between 30 seconds plasma treatment and 480 seconds plasma treatment, a minimal change of the shear stress
- 30 seconds plasma treated fibers had approximately 2 times larger value of shear stress than references fibers
- post-cracking residual flexural strength of samples with the modified fiber is higher in plastic phase by about 30%.

These results are promising for usage of the plasma modified fibers as a reinforcement of cement composites in civil engineering.

Acknowledgements

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