

Plasma Modification of Microfiber Reinforcement to Improve Adhesion with Cement Matrix Containing Recycled Concrete

ĎUREJE Jakub^{1,a}, PROŠEK Zdeněk^{1,2,a}, TREJBAL Jan^{1,c} and TESÁREK Pavel^{1,d}

¹CTU in Prague, Faculty of Civil Engineering, Thakurova 7, 166 29 Praha 6, Czech Republic ²CTU in Prague, UCEEB, Trinecka 1024, 273 43 Bustehrad, Czech Republic ^ajakub.dureje@fsv.cvut.cz, ^bzdenek.prosek@fsv.cvut.cz, ^cjan.trejbal@fsv.cvut.cz, ^dtesarek@fsv.cvut.cz

Keywords: plasma fiber modification, fiber surface modification, oxygen plasma, polypropylene fibers, PP fibers.

Abstract. The article describes oxygen plasma surface modification of polypropylene (PP) microfibers in order to increase coherence with the cement matrix. Five different times of 30, 60, 120, 240 and 480 seconds of plasma modification were used. Changes on fiber surface were detected by SEM analysis, packed cell wettability and weight loss during modification. Selected modification times were chosen to produce test samples on which the modulus of elasticity was continuously measured, and then bending and compression tests were performed. The values were compared with the values for the reference samples.

Introduction

The major problem with cement composite materials is a fragility and low tensile or bend strength. [1] Therefore, the cement matrix should be suitably reinforced. There is possibility to reinforcing by scattered fibers. This method of reinforcement has been used for a long time. For example, straw has been used in the building industry for 3500 years ago to reinforce unfired clay bricks. The article deals with synthetic fibers that have been used since the early 1990s. [2] One of the most important properties of cement composites is cohesiveness of the fiber with the matrix. Cohesion is a property depends on an interfacial transition zone.

In order to increase (or reduce) the coherence of the fibers with the matrix, plasma modification of the fiber surface can be used. Plasma is produced by supplying sufficient energy to the gas. Therefore, plasma is referred to as the fourth state of matter. Plasma contains charged particles - positive ions and negative electrons and neutral particles. [4] For plasma fiber modification process is using low-temperature plasma because of low used material temperature resistance. Plasma acts on the surface of the fibers both mechanically and chemically. With the increasing time of plasma modification process are changes on the surfaces more significant. [5,6]

In previous works, the fibers have been modified to improve their coherence with various binders. Carbon fibers has been successfully modified for their application to the epoxy binders and polyetherimide (PEI) composites. [7,8] Polypropylene fibers have been modified for better applicability of colors and adhesives on their surface. [9] Plasma modification has successfully increased the resistance of sisal fibers in alkaline environments. [10]

Materials and Samples

Fibrofor polypropylene microfibers was chosen for plasma plasma oxygen modification. It is a common commercially reinforcement used in the building industry most often for reinforcing concrete as a secondary reinforcement. The mechanical properties of the fibers are shown in Table 1.

Tab.1 Fiber properties

Fibers	Company	Туре	Length [mm]	Diameter [µm]	Tensile strength [MPa]	Modulus of elasticity [GPa]	Density [g/cm3]
РР	Fibrofor	High Grade	19	80	400	4,9	0,91

The fibers were plasma modified in the Tesla VT 214 for 30, 60, 120, 240 or 480 seconds. The plasma modification process was in the chamber of the Tesla VT 214 from which the air was first evacuated to a pressure of 22 ± 1 Pa and subsequently filled with oxygen at a pressure of 60 ± 1 Pa and then the plasma modification process was initiated for a set period of time. The low-temperature plasma was produced by radio frequency source with 100 W input power. The temperature of the substrate in the chamber gradually increased during the plasma modification, reaching the temperature at about 50 °C for the longest time. Based on the results of the measurements, fibers with the 60s and 480s were selected for reinforcement of the test samples. After modification, the fibers were stored in sealed bags to prevent air exchange and some fibers were stored in open bags for 5 days in a laboratory environment at 22 ± 1 ° C and a relative humidity of $50 \pm 2\%$. (Table 2).

Designation	Fibers	Modification time [s]	Plasma gas	Storage
PP 60 s	PP	60	Oxygen	Sealed bags
PP 480 s	РР	480	Oxygen	Sealed bags
PP 480 s old	PP	480	Oxygen	Open bags

Tab. 2 Fibers modification

There were made four sets of samples, each set contains six testing samples. The dimensions of the test samples were $40 \times 40 \times 160$ mm. Portland cement CEM 42,5R (Radotin), micromilled concrete recyclate and plasma modified fibers were used to produce this samples. The composition of all test samples was the same, differing only by the modifications of the fiber surfaces (Table 3).

Designation	Cement [g]	Recyclate [g]	Water [g]	W/(C + R)	Fibers [%] volume	Fibers [g]
PP ref	400	100	205	0,41	2	5,13
PP 60 s	400	100	205	0,41	2	5,13
PP 480 s	400	100	205	0,41	2	5,13
PP 480 s old	400	100	205	0,41	2	5,13

Tab.3 Composition of the samples

Experimental Methods and Results

All fibers were weighed before and after their plasma modification. The fibers were weighed by Kern with accuracy 0.1 mg. The highest weight loss was 0.18% of their original weight at the modification time of 480 seconds. From the measured data it can be concluded that the loss of mechanical properties of the fibers (reduction of the fiber diameter) is almost negligible.

The fiber surface was examined using the Merlin ZEISS electron microscope, which allows the observed object to be magnified up to 2,000,000 times. Mechanical changes by plasma modification on fiber surfaces compared to reference fibers were observed by electron microscopy at time 120, 240 and 480 second (Fig. 1).



Fig.1 Comparsion of fibre surface, reference – left, plasma modified surface at 480 seconds - right

Fiber wettability was measured by an indirect method similar to the Packed-cell method. The fibers were placed into small container with a perforated bottom and lid. Subsequently, on the fibres were placed lead weights, consequently the fibers were compressed. The whole this container was weighed by Kern with accuracy 0.1 mg and then dipped under water up to the top edge for 30 seconds. Subsequently, the container was taken out of the water and after 120 seconds weighed. The container and lead were also weighed separately. From the measured data was calculated the percentage weight of the water in the container compared to the weight of the fibers (Figure 2).



Fig.2 The weight of water between the fibers

The values of the compressive and the flexural strength of the samples were determined at the 24 days. Samples were tested in three-point bending test. They were loaded with a controlled displacement in a Heckert model FP100 hydraulic press at a rate of 0.4 mm per minute. The remaining half of the samples were tested in pressure test at a rate of 0.8 mm per second.

Flexural strength in plasma-modified fiber samples increased by 20-25% (Figure 3). In spite of the fact that the chemical effect of the plasma modification is not stable over time because of air humidity, we can conclude from the experimental results that after five days under normal conditions the chemical effect of the plasma modification almost did not degrade. Compressive strengths were nearly 75 ± 2 MPa for all sample sets.



Fig.3 Comparison of Flexural strength

Conclusions

The plasma modification may suitably modify the surface of the polypropylene (PP) fibers. Increased bending strength of cement composite material occurred at shorter times of modification and, over longer periods of time, the strength did not increase, despite the fact that mechanical changes were observed on fiber surfaces by electron microscopy over longer periods of time. As an optimal modification with a given plasma modifying device setting is oxygen plasma modification for 60 seconds.

Acknowledgement

This paper was financially supported by the GA ČR research a project under the number 17-06771S and by Czech Technical University in Prague under No. SGS project SGS16/201/OHK1/3T/11.

References

[1] KERŠNER, Zbyněk. *Křehkost a lomová mechanika cementových kompozitů*. Brno, 2005. Habilitační práce. VUT v Brně.

[2] BHARATHI, D. The history of fibre reinforcement[online]. 2015 [cit. 2017-10-31]. Dostupné z: https://www.linkedin.com/pulse/history-fibre-reinforcement-bharathi-d

[3] BENTUR, Arnon. a Sidney. MINDESS. Fibre reinforced cementitious composites. 2nd ed. New York: Taylor, 2007. ISBN 978-020-3088-722.

[4] ROSE, Margaret. Definition Plasma [online]. 2017 [cit. 2017-10-31]. Dostupné z: http://whatis.techtarget.com/definition/plasma

[5] SABREEN, Scott R. Cold Gas Plasma Surface Modification – Optimize Plastics Bonding Adhesion [online]. 2012 [cit. 2017-10-31]. Dostupné z: http://www.adhesionbonding.com/2012/04/30/cold-gas-plasma-surface-modificationoptimize-plastics-bonding-adhesion/

[6] CHRYSOSTOMOU, Demetrius. Plasma surface modification for the biomedical industry. California, 2009. Prezentace.

[7] MOOSBURGER-WILL, Judith, Matthias BAUER, Fabian SCHUBERT, et al., Methyltrimethoxysilane plasma polymerization coating of carbon fiber surfaces. Surface and Coatings Technology [online]. (2017), 223-230 [cit. 2018-01-06]. DOI:

https://doi.org/10.1016/j.surfcoat.2017.01.017. Dostupné z:

http://www.sciencedirect.com/science/article/pii/S0257897217300178?via%3Dihub

[8] EUNG-SEOK, Lee, Lee CHOONG-HYUN, Chun YOON-SOO, Han CHANG-JI a Lim DAE-SOON, Effect of hydrogen plasma-mediated surface modification of carbon fibers on the mechanical properties of carbon-fiber-reinforced polyetherimide composites. Composites Part B [online]. 2016 [cit. 2018-01-06]. DOI:

https://doi.org/10.1016/j.compositesb.2016.10.088. Dostupné z:

https://www.sciencedirect.com/science/article/pii/S1359836816312227

[9] PULÍČEK, Roman, 2016. Optimalizace procesu hydrofilizace polypropylenového textilu pomocí DBD atmosférické plazmy. Liberec. Diplomová práce. Technická univerzita v Liberci.

[10] BARRA, B.N., S.F. SANTOS, P.V.A BERGO, C. ALVES, K. GHAVAMI a H. SAVASTANO, Residual sisal fibers treated by methane cold plasma discharge for potential application in cement based material [online]. 2015 [cit. 2018-01-06]. Dostupné z: https://www.sciencedirect.com/science/article/pii/S0926669015302764#!