

Interaction Enhancement between Recycled PET Macro-fiber Reinforcement and Cement Matrix via Plasma Treatment

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Abstract

Presented work deals with an interaction enhancement between recycled PET macro-fibers reinforcement and cement matrix via plasma treatment (oxygen low-pressure inductively coupled plasma). Wettability between demineralized water / treated fibers and adhesion between fibers / cement matrix were studied in order to assess the treatment effect. The wettability was measured using an optical goniometer, while the adhesion was examined by means of indirect mechanical testing – displacement controlled four-point bending tests of cement samples reinforced with reference and 30 s treated fibers. Consequently, an impact of the treatment on fiber tensile strength was tested. It was shown, that the plasma modification increased the fiber wettability by the water almost three-times already after 5 s treatment, while fiber tensile strength stayed almost unaffected. Bending tests revealed that the prismatic cement samples reinforced with treated fibers reached to higher toughness in elastic response, to higher residual-strength in post-cracking phase and to lower maximal limit of elasticity in comparison with reference samples.

Introduction

Steel fibers used as reinforcement in concrete mixtures can be replaced with polymeric-based macro fibers, if certain circumstances respected [1, 2]. The usage of the polymeric fibers is regulated by ČSN EN 14889-2 and EN 14845-2. According to the second cited standard, the residual strength of fiber reinforced concrete (amount of fibers 4 kg/m³, measured if CMOD is equal to 3.5 mm) obtained during bending test has to be higher than 1.4 MPa. Unfortunately, in civil engineering praxis, a compliance of mentioned minimal residual strength is difficult to achieve because of adverse polymeric fiber surface properties. These properties can be described as smooth and chemically inert in relation to other concrete mixture phases [3-5]. As a consequent of these drawbacks, both strong chemical as well as physical interaction between fiber surfaces and surrounded matrix is not guaranteed [3, 5]. In order to eliminate this phenomenon and to achieve stronger interaction between the two materials (bond), some modifications should be applied. In contrast with mechanical (surface roughening) and chemical (wet oxidation, polymeric chain opening) modification methods, a plasma treatment seems to be a good alternative combining both the physical (ion bombardment) and the

chemical (surface activation by functional groups implementation) modification. Moreover, compared to the other mentioned methods, plasma treatment modifies the fiber surfaces gently – fiber mechanical properties stay with no decrease [3, 6]. For these purposes, we used oxygen plasma treatment known as dry oxidation modification in this study.

Materials and Methods

Fibers. Recycled PET (polyethylene terephthalate) macro-fibers having diameter equal to 260 μm and length 20 mm made by Spokar (Czech Republic manufacturer) were treated by plasma treatment in order to change their surface properties such as wettability by water and surface free energy.

Cement samples. Fiber reinforced cement prismatic samples having dimension equal to 40 \times 40 \times 160 mm was made from CEM 42.5 II R. The water to cement ratio was equal to 0.4. The curing and hardening of mixtures conducted for 28 days after water admixing. An amount of fibers was 2 % wt.

Plasma treatment. Oxygen plasma treatment in inductively coupled low-pressure cool plasma system was done (power 100 W, O₂ flow 50 sccm). The treatment process differed only in exposition time (from 5 to 60 seconds).

Wettability measurements. Wettability of both, the reference and plasma treated fibers were examined through contact angle measurement using a direct optical method, according to [7]. Fibers were wetted by demineralized water.

Mechanical tests. Both the fiber tensile strength (force needed to fiber break) of single fiber and bending strength tests (four-point bending test) of fibers-reinforced cement samples were conducted using Web Tiv Ravestain FP100 loading frame. The experiment was displacement controlled at constant rate of 0.6 mm/min.

Result and Discussion

The contact angles between plasma treated fibers and demineralized water observed using direct optical method decreased more than three-times after 5 s plasma treatment in comparison with reference ones, as captured in Fig. 1. Based on these findings, it can be clearly said that the plasma treatment duration lasting longer than 5 s is no effective in terms of wettability. On the other hand, to attain desirable physical changes (roughening) onto fiber surfaces by ion bombardment, the treatment time should be longer lasting. We estimated that the most effective treatment time is approximately 30 s. This finding is consistent with our earlier published researches, for example with [8].

Tensile strength tests of single fibers showed that their tensile strength (more precisely said force needed to fiber break) stayed almost unaffected regardless to the treatment time. A slight decrease revealed (max 15 %) obtained during testing can be attributed to surface damage caused by ion bombardment. The results are summarized in Fig. 2.

The four-point bending tests showed that the samples reinforced with 30 s plasma treated fibers were characterized by steeper strength increase in the liner-elastic response compared to reference samples containing no treated fibers. This phenomenon can be probably explained by an elimination of shrinkage cracks caused by stronger interaction between the two materials. Significant changes were observed in the post-cracking phase of samples response. Residual bending strength of samples reinforced with treated fibers was considerably higher in comparison with reference ones. Force needed to attain mid-span deflection of 1 mm oscillated about 4 N and 3 N in the case of samples containing modified and reference fibers, respectively. This behavior clearly pointed out to the desirable interphase interaction increase between fiber surfaces and cement matrix due to plasma treatment. Force-displacement diagram captured during bending test is shown in Fig. 3.

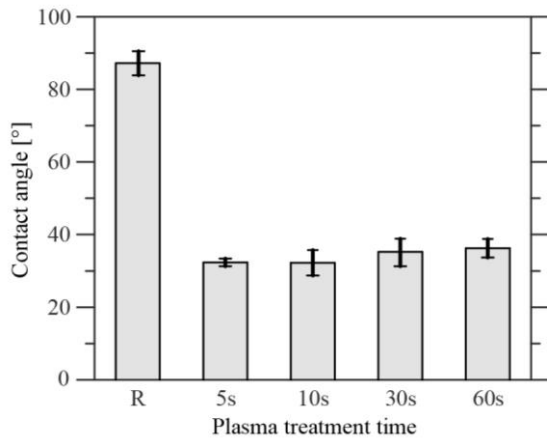


Fig. 1: Contact angle sizes depending on plasma treatment time obtained during contact angles measurements.

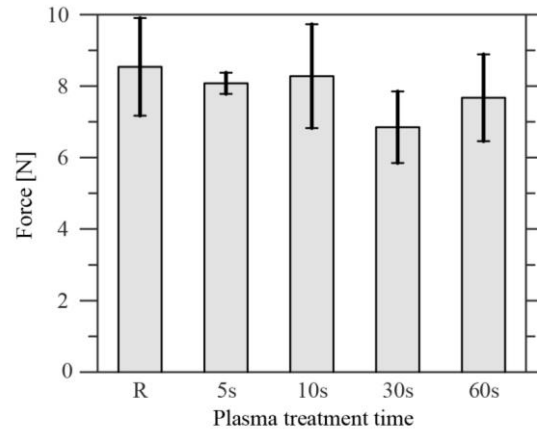


Fig. 2: Force needed to break the fibers depending on treatment time.

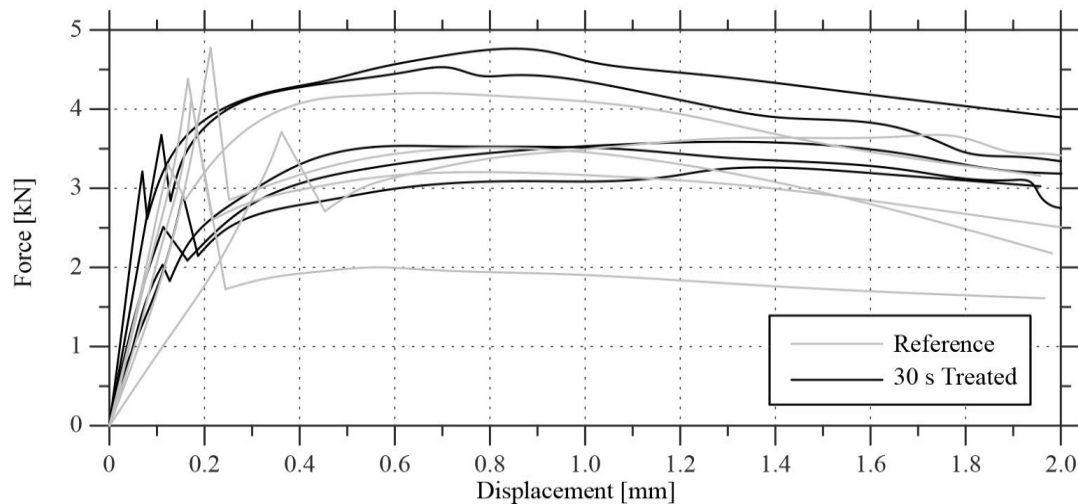


Fig. 3: Loading force depending on specimen mid-span displacement obtained during four-point bending tests.

Conclusion

The aim of this work was to eliminate general polymeric fiber drawbacks such as smooth and inert surface and to attain their bond increase with cement matrix. Polymeric macro-fibers (polyethylene terephthalate) having 260 μm in diameter and 20 mm length were surface modified in order to increase their wettability and roughness. To achieve desirable changes, low-pressure cool inductively coupled oxygen plasma treatment was done. The wettability of modified fibers by distilled water was examined using a direct horizontal goniometer. The impact of ion bombardment on fiber mechanical properties was investigated by means of tensile strength testing. The adhesion increase between the two materials was examined indirectly by bending tests of cement samples reinforced with reference and modified fibers. It was shown that the plasma treatment increased fiber wettability by water almost three times already after 5 s of treatment duration, while fiber tensile strength stayed nearly unchanged. Significant changes of bending response were found out during four-point bending test of prismatic cement samples reinforced with reference and modified fibers. Samples containing

modified fibers exhibited a higher residual strength in the post-cracking phase compared with reference ones. This bending response plainly pointed out to an increase of mechanical cohesion between modified fibers and cement matrix due to ion bombardment causing fiber surface roughness increase.

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