10th International Conference on Fracture Mechanics of Concrete and Concrete Structures
FraMCoS-X
G. Pijaudier-Cabot, P. Grassl and C. La Borderie (Eds)
https://doi.org/10.21012/FC10.235602

INTERPHASES IN POLYPROPYLENE AND GLASS FIBER REINFORCED CEMENTITIOUS MODEL COMPOSITES UNDER DYNAMIC LOADING

ENRICO WÖLFEL* AND CHRISTINA SCHEFFLER

Leibniz-Institut für Polymerforschung Dresden e.V.
Hohe Straße 6, 01069 Dresden, Germany
e-mail: woelfel@ipfdd.de, scheffler@ipfdd.de, www.ipfdd.de

Key words: Glass fibers, Single fiber pull-out, Dynamic loading, Cementitious matrix

Abstract: In the work at hand the influence of the mechanical properties as well as the fiber shape by means of polypropylene (PP) fibers, and the influence of the surface modification of alkali resistant (AR) glass fibers on the failure behavior of cementitious single fiber model composites were investigated. The specific fibers were produced without sizing on lab spinning equipment and the surface modification of the AR glass fibers was done via dip coating afterwards. The fibers were embedded into a high strength matrix and tested under dynamic and quasi-static loading with the single fiber pull-out (SFPO) test. Additionally, single fiber tensile tests, contact angle measurements and scanning electron microscopy before and after the pull-out were performed to clear up the failure mechanisms. Increased mechanical properties as well as the change of the cross sectional shape of PP fibers from circular to trilobal, lead to changes of the force level and shape of the measured force displacement curves. In case of the surface modified AR glass fibers the shape and force level depends on the chemical composition and mechanical properties of the applied polyurethane based sizings. The aimed modification of the interphase properties due to crosslinking in the sizing resulted in a changed failure behavior. These findings are the basis for further investigations aiming at tailored sizings to improve the energy absorption under dynamic loading.

1 INTRODUCTION

To overcome the pronounced brittleness and low tensile strength of concrete under dynamic loading situations like impact, earthquake or explosion, the reinforcement with short fibers is one of the possible solutions to improve the ductility. Beside steel fibers which are most commonly used, glass or polymer fibers could be a more appropriate solution in specific cases. Anyway, for the development of impact resistant cementitious composites it is necessary to consider the change of the mechanical properties of the involved materials when the loading rate moved from quasi-static to high dynamic [1]. The overall performance is not only determined by the type, shape and length of the reinforcement fibers, the surface properties and therefore the interfacial bond due to various interactions [2] to the cementitious matrix play a crucial role. A common way to influence the adhesion or rather interactions between fiber and matrix is the application of a fiber sizing, which allows a systematic modification of the chemical composition of the interphase. For the investigation of the occurring effects concerning the interphase behavior in dependence of the loading rate, a well suited method is the single fiber pull-out test (SFPO) [3]. Micromechanical investigations are confirmed to be essential for the understanding of the macroscopic composite behavior [1] and form the basis for the aimed development of impact resistant

cementitious composites. In this work, the influence of the fiber properties and surface treatment on the fracture behavior and interactions between the fiber and matrix material under different loading rates was highlighted.

2 EXPERIMENTAL METHODS

2.1 Materials

The fibers used for the investigations were produced at lab scale spinning devices at the Leibniz-Institut für Polymerforschung Dresden (IPF). Polypropylene fibers (homopolymer Borealis HG475FB) with a diameter of about 20 µm and a circular (PP-C1/2) and trilobal (PP-T) cross section shape were spun completely without sizing to study the influence of the shape and mechanical fiber properties only. The alkali resistant (AR) glass fibers were produced [4] without sizing (AR-0) and the surface modification was done afterwards with a dip coating process. For this purpose, the fibers were separated, fixed on a special frame and dipped into the various sizings. All used polyurethane (PU) based sizings consisted, beside the different film forming agents, of 3-aminopropyl-triethoxysilane as adhesion promoter and a nonionic surfactant to adjust the wetting. As film forming agents a polyether-polycarbonate PU (PU1), and a system consisting of a polyether PU (PU2) and an isocyanate based crosslinking agent to adjust the properties of the formed polymer film (NC non crosslinked, C crosslinked).

The cementitious matrix system used for the preparation of the single fiber composites for the micromechanical testing was a high strength system described in [1].

2.2 Surface and mechanical characterization

In order to gather information about the wetting behavior of the fiber surface, contact angle measurements were performed with a tensiometer DCAT 21 (DataPhysics Instruments GmbH, Germany). Single fibers were dipped into pure water, and the advancing angle was determined via Wilhelmy method.

The optical characterization of the fiber surfaces before and after the micromechanical testing was done with scanning electron microscopy. As microscope an ULTRA PLUS (Carl Zeiss Microscopy GmbH, Germany) equipped with a SE2 detector was used.

Single fiber tensile tests were performed using a FAVIMAT+ (Textechno, Germany). The tests were realized at 50 single filaments of each fiber type with a clamping length of 10 mm and a velocity of 5 mm/min.

2.3 Micromechanical testing

The production of the pull-out specimens and the single fiber pull-out (SFPO) were performed with devices designed and constructed at the Leibniz-Institut für Polymerforschung Dresden [5], [6]. The cementitious matrix was prepared with a speed mixer and transferred to a sample holder. Subsequently, the fiber was end embedded computer assisted to a depth of about 500 µm for AR glass and 1000 µm for PP, and the specimens were stored for 28 days at humid atmosphere until testing. The single fiber composites were put into the quasi-static and dynamic SFPO devices and the fiber end was fixed at the mandrel with minimized free fiber length. The measurements were performed with a velocity of about 0.001 mm/s for the quasi-static (QSFPO) and 10 mm/s for the dynamic (DSFPO) case and the force displacement curves were recorded.

3 RESULTS AND DISCUSSION

The characterization of the mechanical and surface properties of the fiber material were mandatory to clear up the failure effects in micromechanical tests, which are used to assess the fiber matrix interactions.

In order to investigate the influence of the mechanical properties and the cross-sectional shape of PP fibers on the pull-out behavior, fibers with various mechanical properties were spun (see Table 1). Due to a higher stretching during the fiber drawing process, PP-C1 showed with 4.6 GPa and about 427 MPa

tensile strength at 76 % strain the best mechanical properties of the used PP fibers. In comparison the PP-T with trilobal cross-section shape showed slightly better values in terms of Young's modulus and residual strain compared to PP-C2 but less tensile strength.

Table 1: Mechanical properties of the produced PP and AR-glas fibers (clamping length 10 mm, strain rate 0.5 min^{-1})

Fiber	Diameter [µm]	Young's modulus [GPa]	Tensile strength [MPa]	Strain [%]
PP-C1	19.4 ± 1.0	4.6 ± 0.2	427.1 ± 14.3	76.3 ± 11.7
PP-C2	21.7 ± 1.7	2.1 ± 0.4	208.8 ± 26.7	238.7 ± 29.4
PP-T	20.7 ± 1.6	2.6 ± 0.1	183.5 ± 14.7	215.8 ± 20.5
AR-0	13.7 ± 1.3	56.2 ± 1.8	1424.1 ± 471.9	2.6 ± 0.9

It must be noted that all PP fibers were spun only with pure water to exclude surface finish influences, which limits the usable spinning parameters and therefore the maximum reachable mechanical performance. As expected, the alkali resistant glass fibers (AR-0) showed the highest mechanical properties, which directly affected the possible load transfer or rather capacity in micro- and macromechanical tests.

Table 2: Advancing contact angle of used fibers (*exemplary also for PP-C1 and PP-T)

Fiber	Contact angle [°]
PP-C2*	104.5 ± 7.6
AR-0	56.0 ± 8.7
AR-PU1	74.0 ± 3.4
AR-PU2-NC	61.2 ± 4.7
AR-PU2-C	58.5 ± 2.5

The material itself or more specifically the chemical composition of the surface strongly influences the possible interactions between fiber and matrix material. Therefore, a good wetting of the fiber with the matrix material was the first step to obtain a tough composite. The measured contact angles of the different used fibers against pure water shown in Table 2 give information about the polarity of the fiber and the wetting behavior against the water containing cementitious matrix during production of the model composites. The very high contact angles of the PP compared to AR glass fibers show the nonpolar character respectively hydrophobicity and thus expectable worse chemical compatibility and wettability. For AR glass fibers the contact angle depends on the chemical composition of the applied sizing. While the untreated AR glass showed a low contact angle of about 56°, which indicates a good chemical compatibility and wettability, the applied polyether-polycarbonate PU based sizing PU1 increases the hydrophobicity and deteriorated the wetting. In contrast the sizing PU2 revealed comparable contact angle values referred to the untreated fiber in non-crosslinked and crosslinked state.

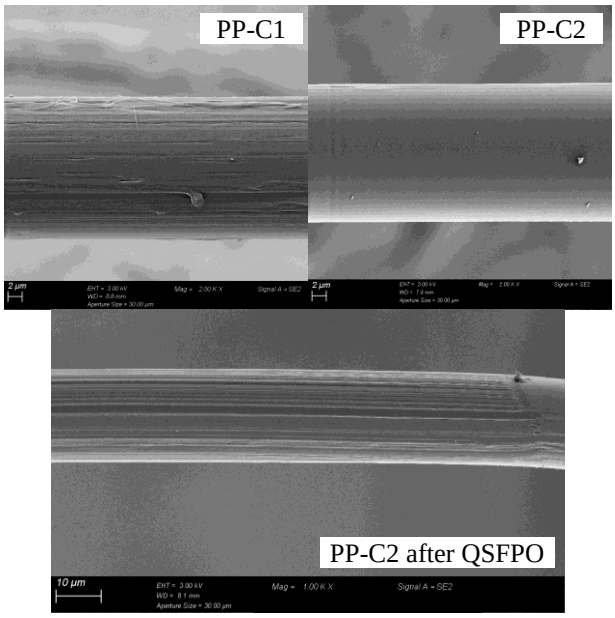

Figure 1: SEM images of the PP fibers, PP-C1/C2: comparison of the Surface structure in the initial state (before pull-out); PP-C2 after QSFPO: scratches due to mechanical abrasion during pull-out

Beside the chemical interactions also mechanical interlocking between fiber surface and matrix plays an important role for the resulting single fiber pull-out behavior. To investigate the topography of the fiber surfaces, scanning electron microscopy was done on the initial fibers. In the upper part of Figure 1 grooves and an overall rougher

surface of PP-C1 can be seen in comparison to the very smooth surface of the fiber PP-C2. The fiber PP-T also predominantly had a smooth surface (not shown).

The force-displacement-curves of the dynamic single fiber pull-out of the PP fiber types were displayed in Figure 2 to Figure 4 and show big differences between the maximal force values and shape of the force displacement curves. It can clearly be seen that the overall force level of PP-C1 was higher in comparison to PP-C2. Taking into account that both fibers made of the same material and thus also have the same chemical composition of the surface, the differences can be traced back mainly to the higher mechanical properties and to some extend on the higher surface roughness of PP-C1.

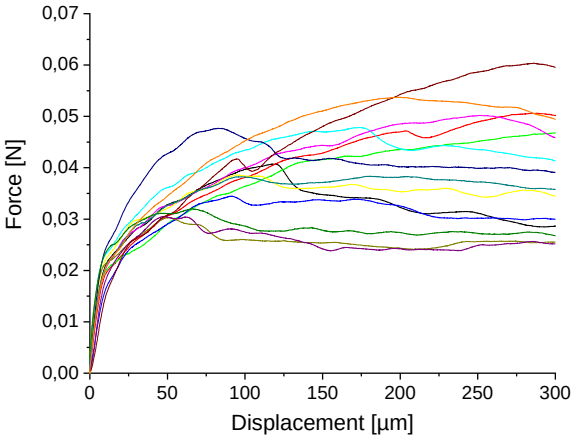

Figure 2: Dynamic SFPO - PP-C1 (circular shape)

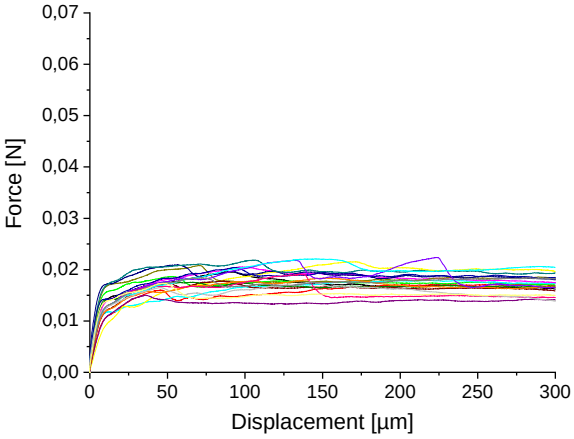

Figure 3: Dynamic SFPO - PP-C2 (circular shape)

Differences in the roughness lead also to the variety of curve shapes of PP-C1. While a higher roughness favored mechanical interlocking and higher forces, smooth surfaces lead to slip out of the fiber with a nearly constant force level until full pull-out. When comparing the measured force displacement curves of PP-C2 and PP-T the curve shape was totally different. While the PP-C2 curves show an onset on a slightly higher force level but then remain constant, the PP-T fibers mainly show a continuous rise until the end of measurement. Under consideration of the small differences in the mechanical properties between PP-C2 and PP-T, a possible reason for the different behavior is probably the more complex cross-section shape and thus surface area, which lead to an improved anchorage in the cementitious matrix and therefore higher stretching of the fiber associated with increased energy absorption. Especially the deformation of the fiber itself because of stretching and/or the surface because of interlocking e.g. scratches (see Figure 1, lower image) play a crucial role in terms of energy absorption of polymer fibers.

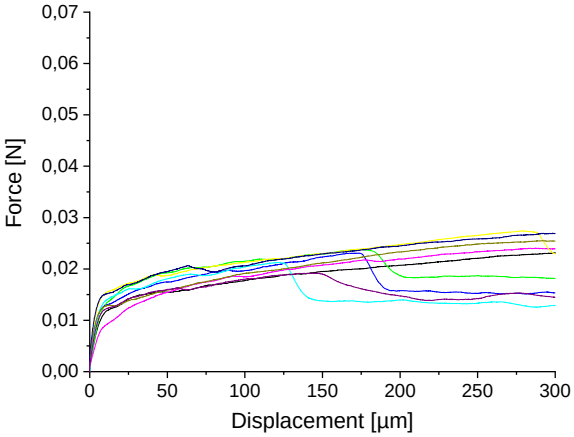

Figure 4: Dynamic SFPO - PP-T (trilobal shape)

In comparison to the dynamic measurements the differences in the force level between PP-C1 (Figure 5) and PP-C2 (Figure 6) are not that pronounced in the quasi-static measurements. While PP-C1 reveal a comparable behavior and force level in dynamic and quasi-static loading (compare Figure 2 and Figure 5), PP-C2 shows nearly

continuously a higher force level (reaching partly PP-C1 level) under quasi-static loading. It seems like the influence of the mechanical properties is less in the quasi-static case.

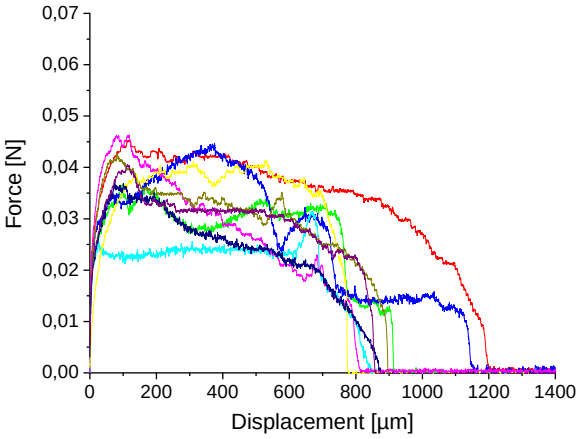

Figure 5: Quasi-static SFPO - PP-C1 (circular shape)

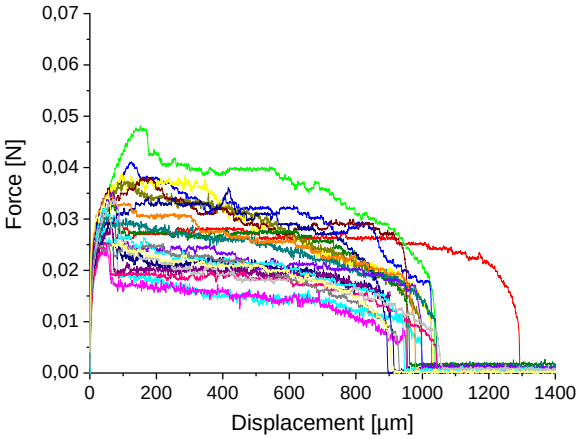

Figure 6: Quasi-static SFPO - PP-C2 (circular shape)

The dynamic and quasi-static measurements show that the mechanical properties, cross-sectional shape and the surface roughness are essential to influence the failure behavior and energy absorption capacity of nonpolar polymer fibers.

In order to investigate the influence of the sizing properties on the fracture behavior under dynamic and quasi-static loading, the surface of the initial AR glass fibers (AR-0) were modified with a polyether-polycarbonate PU (AR-PU1) and a polyether PU/crosslinker sizing (AR-PU2-NC/-C). Besides the chemical composition, also the change of the mechanical properties of the sizing due to crosslinking was studied.

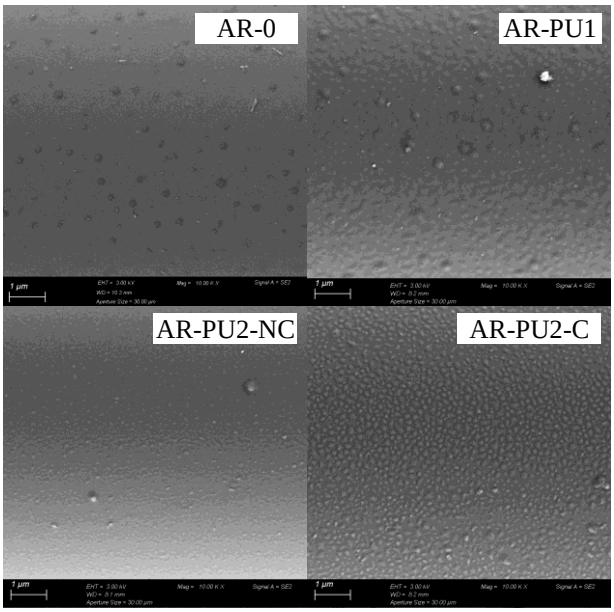

Figure 7: SEM images of the various used AR-glass fibers (surface before pull-out)

Figure 7 shows the surface structure of the untreated and differently sized fiber surfaces. For every type of sizing the surface roughness was only slightly increased compared to AR-0, but the structure of the surface strongly depends on the chemical composition and degree of crosslinking. Because of the better chemical compatibility of all AR glass fibers (see Table 2) to the matrix and the higher mechanical properties, the resulting force level under dynamic and quasi-static loading is much higher than for the PP fibers.

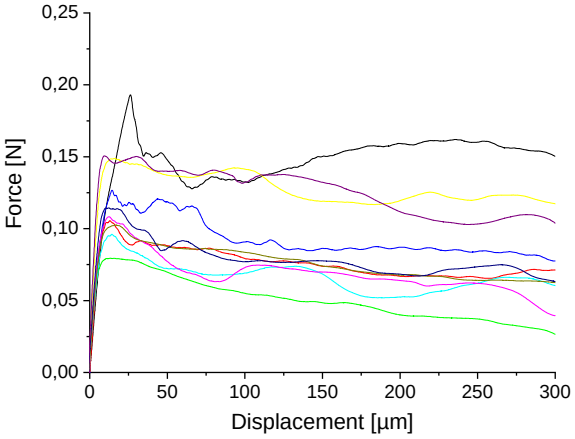

Figure 8: Dynamic SFPO - AR-0

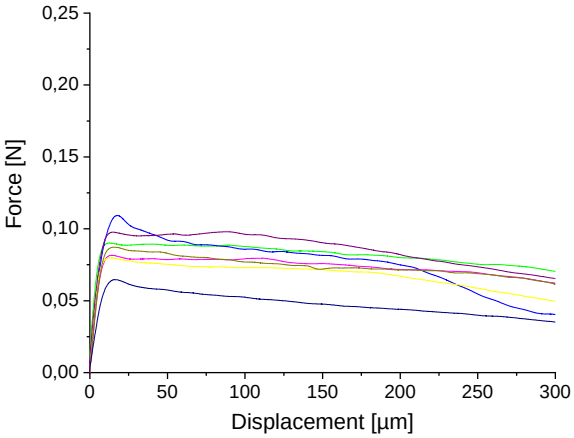

Figure 9: Dynamic SFPO - AR-PU1

The unsized fibers (Figure 8) show a wide spreading between, and also jagged curves because of the brittle behavior of fiber and matrix material. In contrast, the polyether-polycarbonate sized fibers AR-PU1 (see Figure 9) reveal smooth and less scattered curves on a slightly lower force level. In comparison the non-crosslinked AR-PU2-NC sizing (see Figure 11) lead to higher forces at the onset of the curves followed by a drop to comparable values after a displacement of about 75 µm. The changes of the chemical and mechanical properties due to crosslinking have a direct influence on the force displacement curve shape (Figure 12). Compared to the fibers AR-PU2-NC the maximum onset force level of the curves is slightly less, but instead of a significant drop directly after reaching the maximum, the force remains nearly constant for a longer displacement (up to 100 µm). Furthermore, the visible oscillations also indicate a change in the failure behavior.

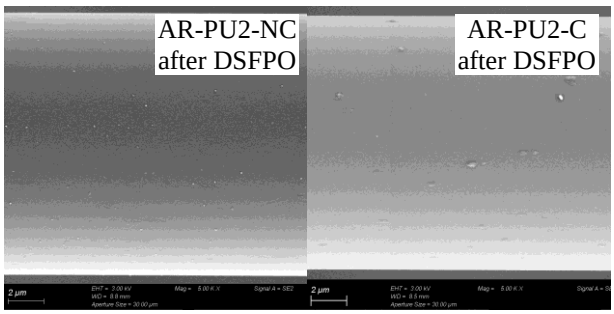

Figure 10: SEM images of AR-PU2-NC (left) and AR-PU2-C (right) surface after DSFPO

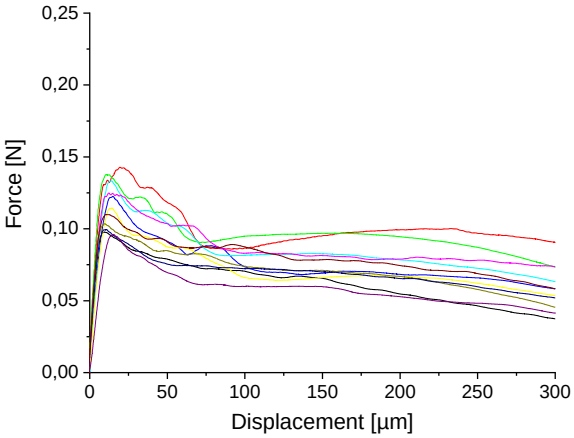

Figure 11: Dynamic SFPO - AR-PU2-NC

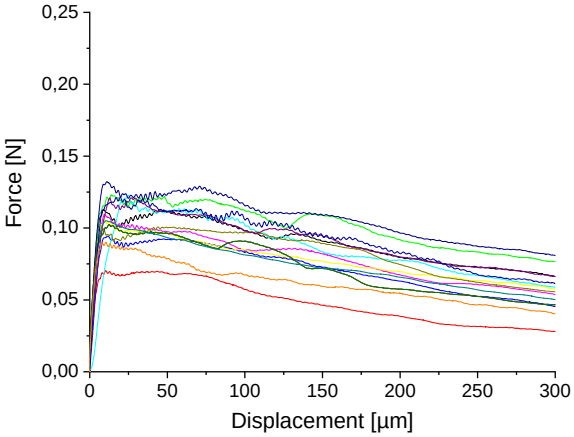

Figure 12: Dynamic SFPO - AR-PU2-C

The SEM images reveal in case of the untreated AR glass fibers, AR-PU1 and AR-PU2-NC (Figure 10) a smooth surface after the dynamic SFPO. Only the AR-PU2-C fiber shows some dots of evenly distributed residual sizing on the surface, which supports the assumption of the changed failure behavior due to the crosslinking.

The quasi-static SFPO results of the fiber AR-PU2-NC (Figure 13) and AR-PU2-C (Figure 14) confirm the DSFPO findings regarding a changed curve shape and the slightly differences in the maximal forces. In comparison to the results of the DSFPO the overall force level is higher under quasi-static loading. Especially, the force displacement curves of the AR-PU2-NC fibers are characterized by oscillations whose amplitude is decreasing with increasing pull-out. These oscillations due to the stress build-up and

release because of mechanical interlocking and slipping, can be also found for AR-PU2-C but only partly at the beginning of some curves and with a higher amplitude and less frequency. Overall the curves for the crosslinked sizing are much smoother.

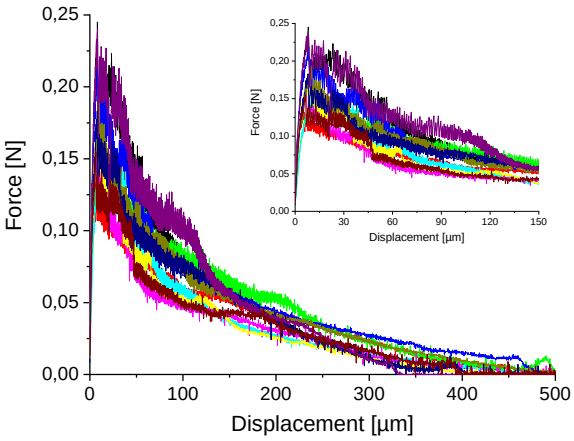

Figure 13: Quasi-static SFPO - AR-PU2-NC

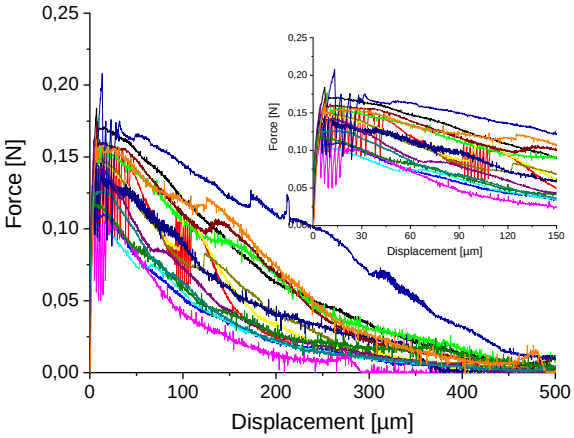

Figure 14: Quasi-static SFPO - AR-PU2-C

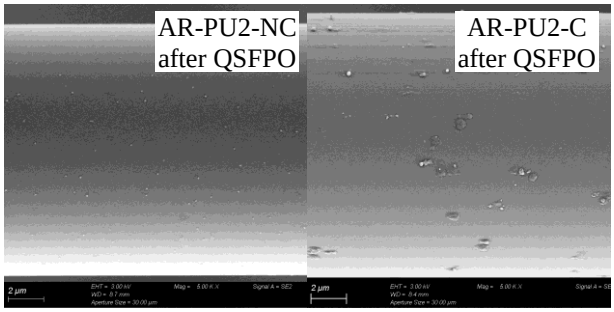

Figure 15: SEM images of AR-PU2-NC (left) and AR-PU2-C (right) after QSFPO

The SEM images of the non-crosslinked and crosslinked fiber surfaces (Figure 15) after the QSFPO look similar compared to the surfaces after DSFPO. While the surface of the fiber AR-PU2-NC is smooth, the AR-PU2-C surface show the already known, but in direct comparison higher extent of residual sizing dots.

The single fiber pull-out investigations at unmodified and surface modified AR glass fibers reveal the influence of the surface properties on the failure behavior. While in the case of an unsized fiber the interactions are dominated by mechanical interlocking and therefore a brittle failure behavior, an applied sizing introduces a softer layer which can reduce the mechanical interlocking and promote slipping. But it is also possible to add new functionalities with the help of a tailored sizing to improve the compatibility and/or adhesion between fiber and matrix material. The studies in the paper at hand show that the failure behavior under dynamic and quasi-static loading rates depends on the chemical composition and mechanical properties of the interphase, which can be specifically influenced by the fiber sizing to improve the energy absorption capacity.

4 CONCLUSION

The dynamic single fiber pull-out results of unsized PP fibers show a strong dependency from the mechanical properties of the fibers. The higher stretching during spinning and the resulting better mechanical properties lead to a higher force level and a change in the force displacement curve shape. The switching from a circular to a trilobal cross sectional shape also results in a curve shape change. Because of the better chemical compatibility to the matrix and better mechanical properties of the AR glass fibers, the force level in the SFPO under dynamic and quasi-static loading conditions was higher compared to the PP fibers. It could be shown that the force level and shape of the force displacement curves and therefore the failure behavior can be specifically influenced with the surface modification of the AR glass fibers e.g. due to

crosslinking in the sizing.

ACKNOWLEDGEMENTS

The authors thank the Deutsche Forschungsgemeinschaft (DFG) for the financial support in the frame of the GRK 2250. Moreover, we would like to thank Dr. Brünig and Mr. Smolka for the polymer fiber spinning, and Ms. Preßler and Ms. Rothe for experimental support.

REFERENCES

[1] I. Curosu, V. Mechtcherine, und O. Millon, „Effect of fiber properties and matrix composition on the tensile behavior of strain-hardening cement-based composites (SHCCs) subject to impact loading“, *Cem. Concr. Res.*, Bd. 82, Nr. Supplement C, S. 23–35, Apr. 2016.

[2] J.-K. Kim und Y.-W. Mai, *Engineered interfaces in fiber reinforced composites*. Amsterdam: Elsevier, 1998.

[3] C. Scheffler, S. Zhandarov, E. Wölfel, und E. Mäder, „Interphases in Cementitious Matrix: Effect of Fibers, Sizings, and Loading Rates“, in *International Conference on Strain-Hardening Cement-Based Composites*, 2017, S. 275–283.

[4] W. Ehrentraut, R. Plonka, E. Mäder, und S. Gao, „Pilot equipment for continuous spinning of alkaline resistant glass fibers“, *Tech. Text.*, Bd. 48, Nr. 1, S. 22–24, 2005.

[5] C. Scheffler, S. Zhandarov, und E. Mäder, „Alkali resistant glass fiber reinforced concrete: Pull-out investigation of interphase behavior under quasi-static and high rate loading“, *Cem. Concr. Compos.*, Bd. 84, Nr. Supplement C, S. 19–27, Nov. 2017.

[6] E. Mäder, K. Grundke, H.-J. Jacobasch, und G. Wachinger, „Surface, interphase and composite property relations in fibre-reinforced polymers“, *Composites*, Bd. 25, Nr. 7, S. 739–744, Jan. 1994.