# SINGLE FIBRE PULL-OUT TESTS OF POLYPROPYLENE AND GLASS FIBRES IN CEMENT-BASED MATRICES AT HIGH LOADING RATES

E. Wölfel[1], C. Scheffler[1] , I. Curosu[2] and V. Mechtcherine[2]

[1] Leibniz-Institut für Polymerforschung Dresden e.V., Department Composite Materials, Hohe Straße 6, 01069 Dresden, Germany
Email: woelfel@ipfdd.de, https://www.ipfdd.de
[2] Institute of Construction Materials, Technische Universität Dresden, Georg-Schumann-Straße 7 01187 Dresden, Germany
Email: iurie.curosu@tu-dresden.de, https://tu-dresden.de/

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**Abstract**
In this work the influence of fibre type, mechanical properties and surface modification on the failure behaviour of cement-based composites under dynamic loading is analysed. For this fundamental research different alkali resistant glass (AR) and polypropylene (PP) fibres with varying properties regarding surface modification and drawing were produced using lab-scale spinning equipment. The single fibres were embedded in cementitious matrices and tested using the single fibre pull-out test. Contact angle measurements, single fibre tension tests and scanning electron microscopy were used to characterize the fibres and clarify the failure mechanisms. The special importance of the sizing on the fibre-matrix interaction was demonstrated by applying a PP sizing on polar and nonpolar fibres. For AR glass it led to a force level drop in the dynamic pull-out tests due to a worsened chemical fibre-matrix compatibility. For PP fibres the pull-out behaviour, which was determined by considerable plastic deformations on the fibre surface, changed to slip-hardening at a higher force level. It could be shown that in the case of smooth nonpolar fibres mainly the fibre properties influence the pull-out behaviour at increased loading rate; due to a particulary low fibre-matrix interaction, an influence of the matrix material could not be observed.

## 1. Introduction

Concrete is the most often used construction material worldwide. Despite its numerous advantages, its low tensile strength and pronounced brittleness determine a relatively low resistance of concrete structures subject to dynamic loading, such as in the case of earthquake, impact or explosion.

One possibility to improve concrete ductility is by adding short fibres. Steel fibres are most commonly used, however, glass or polymer fibres represent preferred solutions in particular cases. In order to design impact resistant fibre reinforced cementitious composites, it is essential to understand and characterize the properties of the main constitutive phases under different loading rates, namely of the matrix, fibres and of their interfacial bond. Their particular strain rate sensitivities may lead to a negative influence of increasing loading/strain rates on the composite performance, if the latter is only designed with respect to quasi-static loading conditions [1, 2]. The micromechanical investigations, such as single fibre tension and pull-out tests, proved to be essential for the understanding of the composite behaviour [1].

With respect to the influencing parameters, different investigations, reaching from the determination of the fibre properties under various strain rates to surface characterization with contact angle measurements, scanning electron microscopy and single fibre pull-out tests (SFPO) [3] are presented in the paper at hand. Emphasis was put on the influence of the fibre properties and treatments on interactions between fibre and matrix material under different loading rates.

## 2. Material and methods

All investigated AR glass and PP fibres were produced at the Leibniz-Institut für Polymerforschung Dresden e.V. (IPF). The surface treatment of the filaments was conducted directly at the online spinning process by a sizing applicator roll. Both fibre types were manufactured without using any sizing but just pure water (purified/demineralised with USF ELGA PURELAB Plus) for the series AR1 and PP1 and using a PP sizing in case of the series AR2 and PP2. The fibres PP3/4 consist of the same spin finish based on polyglycol, but differ in the mechanical properties, see Table 1. A commercial available premix (indicated as C1), a normal-strength (C2) and a high-strength (C3) cement-based matrices described in [1] were used in SFPO. The main differences between the matrices consist in the type and content of cement, water-to-binder ratio and type of pozzolanic binders (fly ash in C2 and silica fume in C3).

The single fibre tension tests were performed using the FAVIGRAPH (Textechno, Germany). The clamping length was 10 mm and the strain rate was 0.5 to 10 $min^{-1}$. The tests aimed at the mechanical properties of the different fibres depending on drawing ratio and applied sizing.

Contact angle measurements were conducted using a tensiometer DCAT 21 (DataPhysics Instruments GmbH, Germany) to investigate the wetting behaviour of the fibres. Single fibres were prepared and dipped into pure water. The advancing contact angle was determined by means of the Wilhelmy method.

The optical studies of the fibre surfaces before and after micromechanical testing were performed with scanning electron microscopy using the SE2 detector of an ULTRA PLUS (Carl Zeiss Microscopy GmbH, Germany) microscope.

The preparation of the single fibre model composites and the pull-out tests were done with equipment designed and constructed at the IPF [3, 4]. After mixing of the cementitious matrices they were transferred to sample holders and the fibre ends were embedded to a depth of 1 mm using a computer assisted device. After storing the model composites for 28 days in a humid atmosphere they were transferred to the quasi-static and dynamic pull-out devices and the fibre was fixed at the mandrel in such a way that the fibre free length was minimized (<0.15 mm). The quasi-static pull-out tests were carried out at a displacement rate of about 0.001 mm/s and the dynamic tests at approximately 10 mm/s and the force displacement curves were recorded.

## 3. Results and discussion

To evaluate the nature of fibre surface in terms of roughness and the distribution of the sizing, SEM images were taken at different locations of individual fibres. A comparison of all fibres used in the work at hand can be seen in Figure 1. The unsized fibres AR1 and PP1 show a smooth surface. On the surface of the fibres AR2 and PP2 the PP-sizing can be clearly observed. There are also some accumulations of sizing, which increase the roughness of the surfaces. In comparison, the fibre finish on fibres PP3 and PP4 seems to be very homogenously distributed. Both fibre types show a nearly smooth surface comparable to that of PP1.

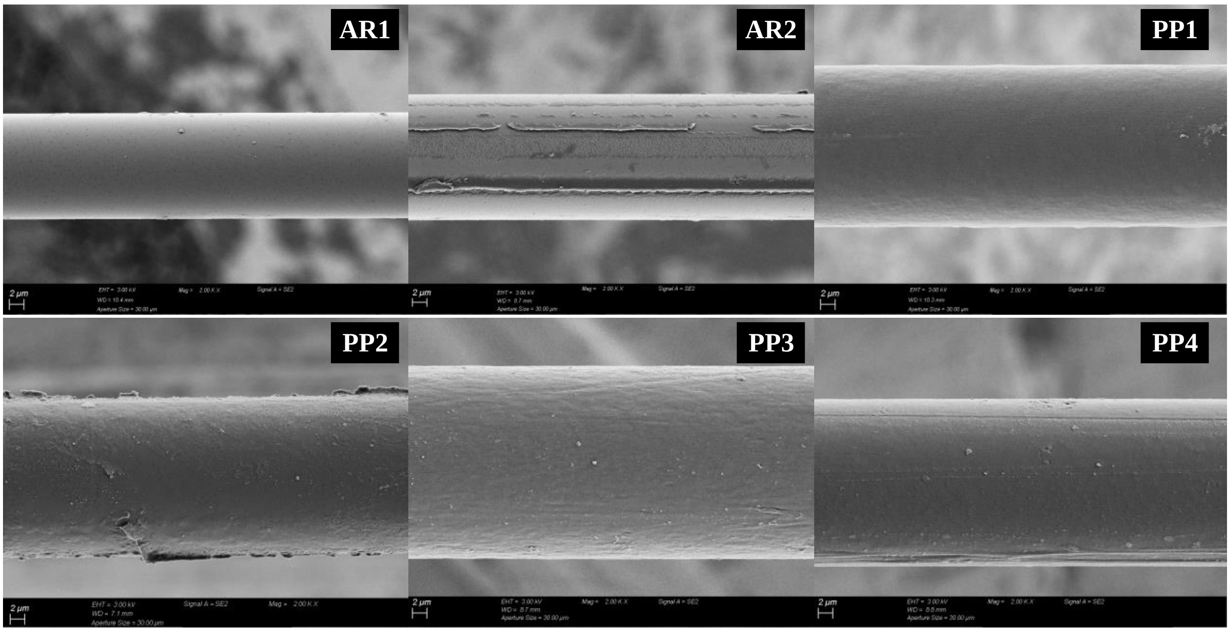


**Figure 1.** SEM images of the surface of all investigated fibres before the pull-out test: AR1 and PP1 without sizing, AR2/PP2-PP4 sized

The mechanical properties of the investigated fibres vary strongly depending on the type, surface treatment and production conditions, see Table 1. As expected, the AR glass fibres show higher tensile strength and Young’s modulus and less elongation capacity than the PP fibres. In direct comparison the sized AR2 fibres exhibit higher values than AR1 fibres due to the healing effect of surface flaws of the applied sizing [5]. In case of the different PP fibres, tensile strength, Young’s modulus and ultimate strain mainly depend on the drawing conditions during the production process. A higher degree of stretching leads to a higher tensile strength and Young’s modulus and less strain capacity due to the alignment of the polymer chains in fibre direction.

**Table 1.** Results of the tension tests (clamping length 10 mm, strain rate 0.5 $min^{-1}$) and contact angle measurements on the fibres under investigation.

| *fibre* | *diameter* (µm) | *strain* (%) | *tensile strength* (MPa) | *Young's modulus (0,5-1%)* (GPa) | *contact angle* (°) |
|---|---|---|---|---|---|
| AR1 | 13.7 ± 1.3 | 2.6 ± 0.9 | 1424.1 ± 472.9 | 56.23 ± 1.84 | 46.3 ± 10.9 |
| AR2 | 16.1 ± 2.1 | 4.4 ± 1.3 | 2443.5 ± 683.2 | 61.67 ± 1.10 | 67.1 ± 3.8 |
| PP1 | 20.8 ± 2.1 | 326.4 ± 102.0 | 125.7 ± 24.6 | 1.06 ± 0.29 | 95.2 ± 7.2 |
| PP2 | 22.6 ± 4.8 | 313.7 ± 76.5 | 136.0 ± 24.1 | 1.21 ± 0.35 | 80.4 ± 3.7 |
| PP3 | 27.4 ± 1.0 | 52.3 ± 13.9 | 287.4 ± 22.6 | 2.43 ± 0.22 | 86.6 ± 6.4 |
| PP4 | 24.7 ± 0.8 | 160.6 ± 30.5 | 159.8 ± 18.1 | 1.55 ± 0.22 | 80.3 ± 2.8 |

In terms of micromechanical investigations, the mechanical properties of the fibre are mandatory for the analysis of the measured force displacement curves concerning the interfacial parameters depending on the evaluation models used [6]. To understand how the mechanical properties of the fibres change with increasing strain rate tension tests were conducted on fibre PP3 (see Figure 2) because it revealed the best performance in the dynamic pull-out tests (see Figure 4). It can be noted that tensile strength and Young’s modulus increase strongly by increasing the strain rate from 0.5 to 2 min$^{-1}$ followed by only slightly when going from 2 to 10 min$^{-1}$. The reverse behaviour is revealed by the measured strain. The maximum strain rate is limited by the equipment at 10 min$^{-1}$, but the tendency suggest a steady increase in tensile strength and Young's modulus with increasing strain rate. Comparable findings concerning the influence of the velocity were found for HDPE and PVA fibres in [1].

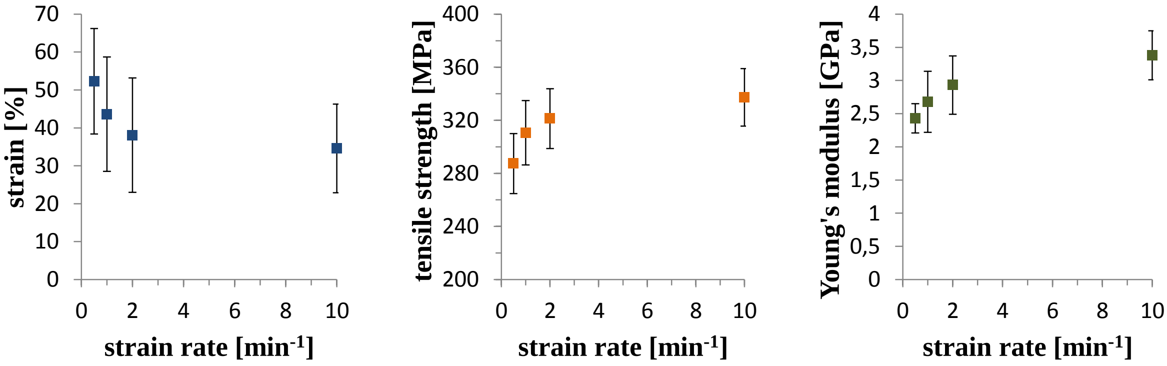


**Figure 2.** Strain, tensile strength and Young's modulus of PP3 fibre in dependence of the strain rate

The chemical compatibility between fibre surface and matrix material influences the possible interactions. With regard to that, wetting measurements can provide helpful information. To investigate the wetting between fibres and polar cementitious matrices, pure water was used in the contact angle measurements. The smallest advancing contact angle with about 46.3° and therefore best wetting towards water was observed for the unsized AR glass fibre, which was polar, see Table 1. The worst wetting angle of 95.2° was revealed by the unsized and nonpolar PP1 fibre. The sizing of AR2 which contain a PP film former leads to an increase in the contact angle to circa 67.1°. In case of the PP2 fibre the PP sizing improved the wetting probably due to the included surfactants. The fibres PP3 and PP4 also showed a better wetting because of the fibre finish. Due to the smaller sizing content of the fibre PP3 the improvement was less pronounced.

The dynamic pull-out test was used to investigate the effect of type, surface modification and mechanical properties of the fibre on the interaction with cement-based matrix and the failure behaviour under high loading rates. The highest maximum values of the pull-out force were attained for the AR1 glass fibres, see Figure 3. The maximum forces were reached at low displacements followed by slip softening until the test was stopped at the maximal displacement of 0.3 mm (limited by the test device; embedding fibre length was $l_e$=1 mm). This strong interactions between the fibre and matrix C1 were supported by the low contact angle measured. In comparison to AR1 the force displacement curves measured for AR2 reveal the same shape but on a lower force level, which corresponds to the higher contact angle values and displays the influence of the sizing on the interactions between fibre and matrix material.
The influence of the fibre type (glass fibre: high modulus/polar surface, PP fibre: low modulus/nonpolar surface) can be directly seen by the great difference in the shape of the force-displacement curves of AR1 and PP1, see Figure 3. The maximum forces for PP1 are reached at higher displacements followed by slightly accending curves until the end of measurement. But, despite of the bigger fibre diameter, the pull-out resistance (in terms of pull-out force) is more than ten times lower than that of the AR1 fibres because of the nonpolar nature of PP1 fibres. In contrast to the treatment of AR glass fibres, the application of the PP sizing leads to an increase in the maximum forces in case of the PP2 fibres and a larger area under the curve which corresponds to higher pull-out work. Furthermore, the force-displacement curves reveal slip-hardening, which is desired for the goal of enhanced energy absorption. The good wetting behaviour induced by the sizing in combination with an increased surface roughness (see Figure 1) seems to be an important factor to adjust the failure behavior in a way that slip-hardening – caused by the plastic deformation of the fibre surface – occurs.

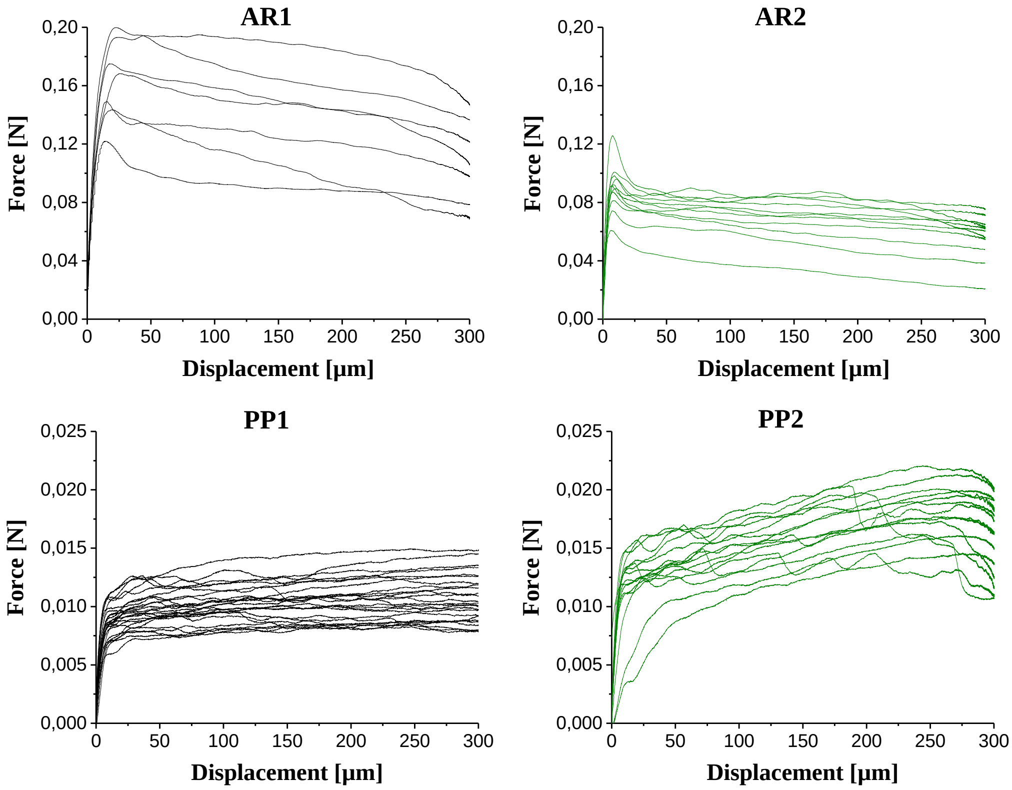


**Figure 3.** Force-displacement curves obtained by the dynamic pull-out tests: AR1 and PP1 fibres without sizing embedded in C1 (black), AR2 and PP2 fibres with PP sizing embedded in C1 (green)

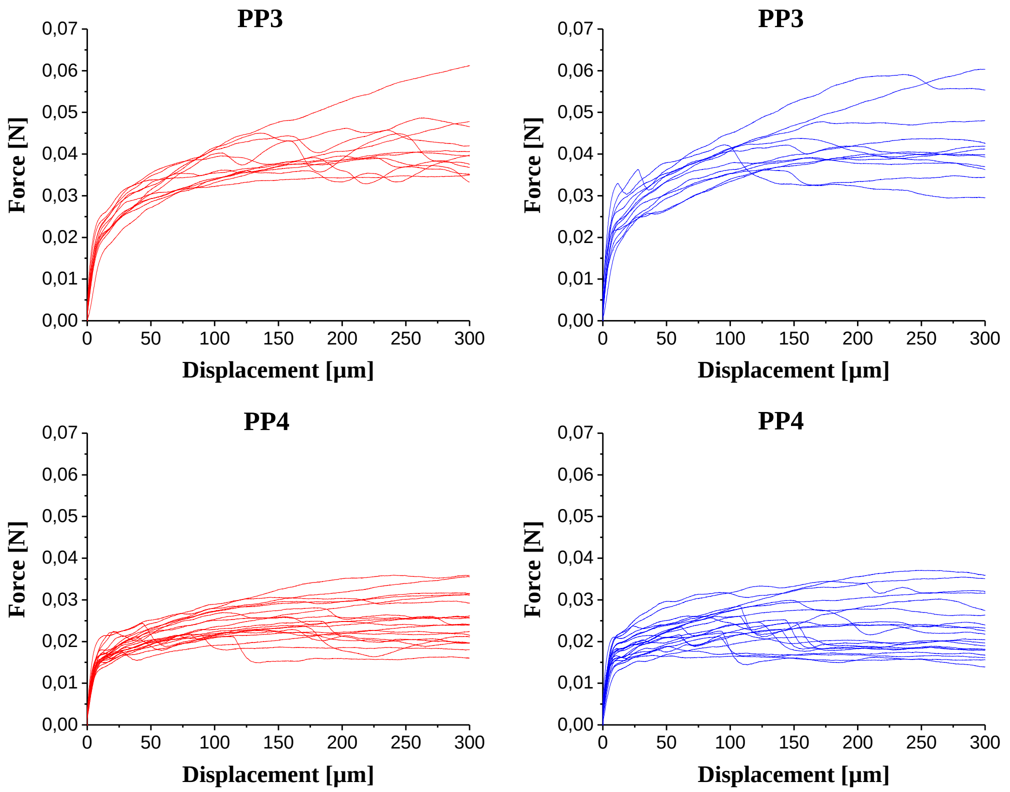


**Figure 4.** Force-displacement curves of the dynamic pull-out tests: PP3 and PP4 fibres embedded in C2 (red), PP3 and PP4 fibres embedded in C3 (blue)

The dynamic pull-out behaviour of nearly nonpolar PP fibres with different mechanical properties and the influence of the matrix type are shown in Figure 4. By comparing the force-displacement curves for PP3 fibres embedded in normal-strength matrix C2 and high-strength matrix C3 it can be seen, that the shape of the curves and the level of the forces are nearly identical in both cases. The same finding was revealed by testing PP4 fibres embedded in C2 and C3. It seems that the matrix plays a subordinated role in case of a smooth fibre with little interaction to the matrix material. In contrast to this finding, there is a great difference between the force level of the curves obtained for PP3 fibres and PP4 fibres embedded in the matrices C2 and also C3. The higher tensile strength, Young's modulus and lower strain capacity of the fibre PP3 lead to higher forces in the dynamic pull-out test, while force-displacement curve shape remains almost unchanged. This indicates that PP fibres should have low residual strain and high tensile strength and Young's modulus values to maximize the energy absorption capacity in composites with cementitious matrices.

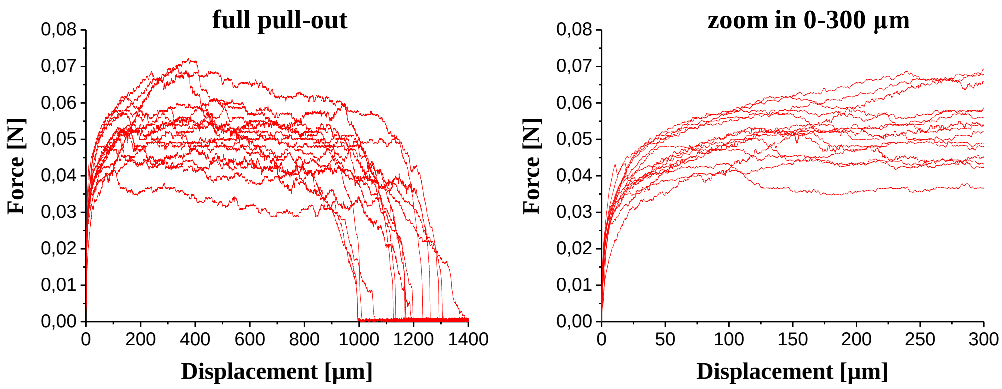


**Figure 5.** Force-displacement curves obtained in the quasi-static pull-out tests on PP3 fibre embedded in C2 matrix

The investigation of the loading rate influence was carried out on the PP3 fibres embedded in the normal strength matrix C2 by additionally performing quasi-static tests. The onset part from 0 to 300 µm of the force-displacement curves (see Figure 5) looks identical compared to the same fibre-matrix combination under dynamic loading (see Figure 4), only the force level and maximum force are higher in the quasi-static case. A higher force level in the quasi-static tests is rather untypical compared to other investigations with polymeric fibres [1]. Reasons for this behaviour could be the unsufficient compatibility to the polar matrix or lower surface roughness of the PP fibres in comparison to PVA and HDPE fibres investigated in the earlier work by the authors. Also a stretching of the fibre during the pull-out because of the relatively low Young's modulus and high plastic deformations could be a reason, which was supported by most of the curves in Figure 5 reaching zero force (full pull-out) at displacements higher than 1 mm. The shape of the measured curves for PP3 differs strongly from that of the quasi-static curves for the modified AR glass fibres, see [7]. In comparison to the slip-softening effect which can be seen in case of AR glass after reaching the maximum force, the force level for PP3 remain nearly constant only with a slight decrease followed by a strong drop to zero at the end of measurement. A reason for this different behaviour are the strong interactions between AR glass and cementitious matrix and therefore a high stress input into the fibre, in contrast to the worse adhesion and less stress input in case of the polypropylene fibre. This led in case of the AR glass to a debonding of the fibre till the maximum force, followed by the pull-out of the fully detached fibre, where the measured forces are only influenced by friction. In comparison, a stretching of the fibre and a permanent abrasion or rather plastic deformation (see Figure 6) of the fibre surface until slip-out occured for PP fibres. This failure behaviour was the preferred one with a view to solutions for high dynamic loading conditions.

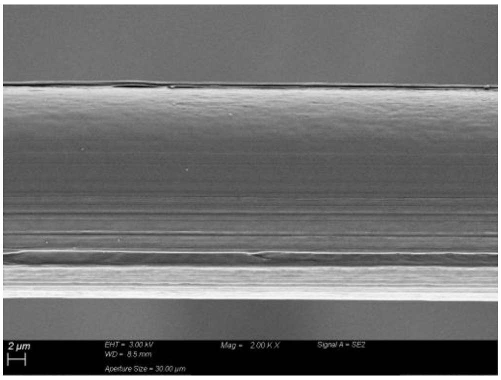


**Figure 6.** SEM image showing the plastic deformations on the surface of PP3 fibre (in comparison to the initial fibre, see Figure 1) embedded in C2 matrix after quasi-static pull-out test

In previous works different models have been used to calculate the interfacial parameters [6, 8], however, these models are based on the determination of characteristic points (debond force, maximum/peak force, initial post-debonding force) and slip-softening pull-out behaviour. These characteristic points could not be determined for the material combinations investigated in this study. A further development of the existing models or even new models are needed which describe the force displacement curves and allow the calculation of the interfacial parameters.

## 4. Conclusion

The application of a PP sizing decreases the overall force level in the dynamic single fibre pull-out tests on AR glass fibres while maintaining their slip-softening behaviour. In contrast, the same sizing improves the wetting and increase the roughness of PP fibres, which shifts the curves to a higher force level with slip-hardening behaviour caused by plastic deformations of the fibre. A higher degree of stretching of the PP fibres during the production process leads to higher force levels when these fibres are pulled out of the normal or high-strength matrix under dynamic loading; however, the matrix properties exhibit no influence on the pull-out behaviour. Under quasi-static loading the fracture behaviour of PP fibres was characterized by high plastic deformations, which led to a nearly constant force level until complete slip-out of the fibre, in contrast to the slip-softening behaviour of AR glass fibre.

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