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Improving the bond characteristics of AR-glass strands by microstructure modification technique

Mert Yücel Yardımcı [a,*], Ronit Tirosh [b], Pavel Larianovsky [b], Moshe Puterman [b], Arnon Bentur [b]

[a] Department of Civil Engineering, Zonguldak Karaelmas University, 67100 Zonguldak, Turkey
[b] National Building Research Institute, Faculty of Civil and Environmental Engineering, Technion – Israel Institute of Technology, Haifa 32000, Israel

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ABSTRACT

This paper introduces a fiber modification technique for enhancing the bond characteristics of AR-glass strands embedded in cement paste. AR-glass strands were modified by absorption of four different types of slurries containing sub-micron organic and inorganic particles. Two different sample preparation processes were applied prior to incorporation the modified strands in the cement paste. Bond characteristics of unmodified and modified AR-glass strands embedded in cement paste were determined by pull-out test and the failure mechanism of pulling out strands was determined by Failure Investigation by Light Transmission (FILT) test. Test results showed that the bond properties and failure type of AR-glass strands can be enhanced by the microstructure modification technique. The efficiency of the modification was highly dependent on filler type, structure and also the production method.

1. Introduction

In recent years there has been a considerable drive for innovation in construction materials, based primarily on the development of high performance fiber reinforced cementitious composites. These materials are engineered to induce a strain or deflection hardening behaviours so that the brittle cementitious matrix is turned into a composite exhibiting high tensile strength and ductility, using very small amount of fiber reinforcement [1–3]. In addition to this the use of fabric reinforcement (textile reinforced concrete) has been the subject of intense research and development effort, to produce high performance composites with controlled geometry that could enable new lightweight building systems [3–5], and also for strengthening purposes [6]. It should be noted that most of the high performance man made textile fibers are in the form of multifilament bundles (rovings), with the monofilament diameter being in the range defined as micro-fiber (less than 100 μm). The multifilament reinforcement is particularly attractive because of the wide range of fiber properties which can be produced, and the technology available for making technical fabrics which could be tailored for high performance cementitious composites, with controlled two- and tri-dimensional geometry.

The use of man made multifilament reinforcement for cements has numerous advantages, which are the drivers for their application for the development of advanced cementitious composites. As a result the reinforcing unit is a bundle with external filaments in direct contact with the matrix, rather than single filaments with each being completely embedded and engulfed by the matrix (which is the case in polymer matrix composites). This results in a reinforcing unit in which the external filaments are in intimate contact with hydration products, and the internal filaments are relatively free, and they interact and transfer stresses through surface contacts between them (e.g. Refs. [5–9]). Therefore, the inner filaments are not effectively utilized for the stress transfer.

The bundled nature of the reinforcing unit leads to different bonding mechanisms, which cannot be simply simulated by conventional models, and alternative ones have been developed for this purpose (e.g. [7,8]). More than that, this special microstructure is posing several types of limitations, such as: (i) inefficient use of the internal filaments, and (ii) a system where the nature of the bond is changing during the life time of the composite, due to slow and gradual process of deposition of hydration products in between the filaments (e.g. [3,9]). These issues pose considerable difficulties and limitations for maximizing the efficiency of the reinforcement and providing an age-stable composite. The embrittlement of glass fiber reinforced cement over time [3,9,10] and the step recommended sometimes to impregnate the strand with polymer before the production of a cementitious composites (e.g. [11]) are examples of the drawback of this microstructure.

In this study modification of the microstructure of reinforcing strands by a relatively simple process, to generate a controlled differential bonding in the reinforcing strand was studied. AR-glass strands were modified by the impregnation of glass fiber strands

* Corresponding author. Tel.: +90 372 2574010x1652; fax: +90 372 2574023.
E-mail address: myyardimci@gmail.com (M.Y. Yardımcı).

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with flowable slurries containing four different types of inorganic and polymeric sub-micron particles. Controlled telescopic bonding mechanism could be generated by introducing the sub-micron particles in between the filaments. The present paper reports the study of the pull-out of strands which were impregnated by the sub-micron particles. Extension of this study to evaluate long term durability influences will be reported in the future.

2. Experimental

2.1. Materials and sample preparation

Fig. 1 shows warp knitted AR-glass fabric and SEM picture of warp yarn filaments. Yarns for pull-out tests were obtained by unstitching yarn from a warp knitted fabric. The technical properties of AR-glass yarn are given in Table 1.

Slurries of inorganic and polymeric materials were used for the treatment of the reinforcement (yarns). Each of the slurries contains about 50% of solids, which were of four types:

SFS – silica fume with particles of 0.05 μm
SFL – silica fume with particles of 0.2 μm
PSP – polystyrene polymer with glass transition temperature, T_g, of 110 °C, and particle size of 0.2 μm.
SAF – styrene acrylic polymer with glass transition temperature, T_g, of −6 °C, and particle size of 0.2 μm.

The inorganic slurries of silica fume differ in their particle size, one being smaller (SFS) and the other bigger (SFL), while the polymeric slurries differ in their film forming capacities, the SAF with the lower T_g being film forming, and the PSP having the T_g higher than room temperature is non film forming.

The treatment was simply done by immersing the yarns in the slurry for several seconds during which the slurry could be absorbed into the spaces between the filaments. Before the incorporation of the modified yarns into the paste, two types of sample preparation processes, namely wet and dry, were applied. In the wet process, modified yarns were incorporated into the paste just after the modification. In the dry process the modified yarns was dried for 24 h in the laboratory conditions (60% RH, 20 °C) before incorporation into the paste.

For characterizing the pull-out behaviour of unmodified and modified glass fibers, glass fiber yarns were embedded in a paste matrix of 0.40 water to cement ratio. The cement used in this study was CEM-II 42.5 N/B-LL blended Portland cement with a limestone content of 21% by weight. Compressive strength of 28-day paste was 56.6 MPa. Pull-out specimen preparation was based on special flexible silicon molds which were developed for this purpose (Fig. 2a and b).

Table 1
Properties of AR-glass yarn unstitched from the fabric (the value in bracket shows standard deviation).

Tex number[a]	1200
Density (g/cc)	2.678
Cross section area of the bundle (mm^2)	0.4481
Average tensile strength (MPa)	1028 (104.31)
Modulus of elasticity (GPa)	65.592

[a] Tex is defined as the fineness of the yarn given by the weight per length ratio, in grams per 1000 m of the yarn.

Thin slits on both sides of the silicon mould were cut to enable to position the yarn in the center and keep it straight and stretched. The reinforcing yarn is embedded on one side in the paste matrix and in the other in a polyester matrix which is gripped for pulling out of the yarn from the paste matrix.

Twelve samples were prepared for each type of treatment and the results presented in this study are the average of these twelve samples.

2.2. Pull-out test

Pull-out test setup and the sample are shown in Fig. 3a and b, respectively.

The pull-out test was performed in a Zwick 1445 testing machine. Loading rate was 0.8 mm/s. The pull-out specimen is positioned on a moving bridge with a slit for threading the polyester base. A thin rubber piece was used between the sample and steel moving bridge (Fig. 3b) for eliminating fiber inclination in order to ensure homogeneous stress transfer to filaments. Therefore it should be kept in mind that the pull-out displacement values which were recorded in this pull-out system contain also the rubber displacement as an additional displacement to the slip displacement of the filaments pulling out from the matrix. Once the sample is placed, the polyester grip is firmly held by metal plates tightened by screws. Two LVDTs are attached on both sides of the polyester base, to record its movement relative to the bridge and thus measure the displacement (slip) of the yarn as it is being pulled from the matrix. The use of two LVDTs is also intended for monitoring the alignment during the pull-out. The load and the displacement signals are fed continuously into a computerized control system and the load – pull-out displacement curve was recorded. The area under the load–displacement curve up to the displacement level of twofold the displacement at the maximum load was calculated (total energy) and the post peak portion of the total

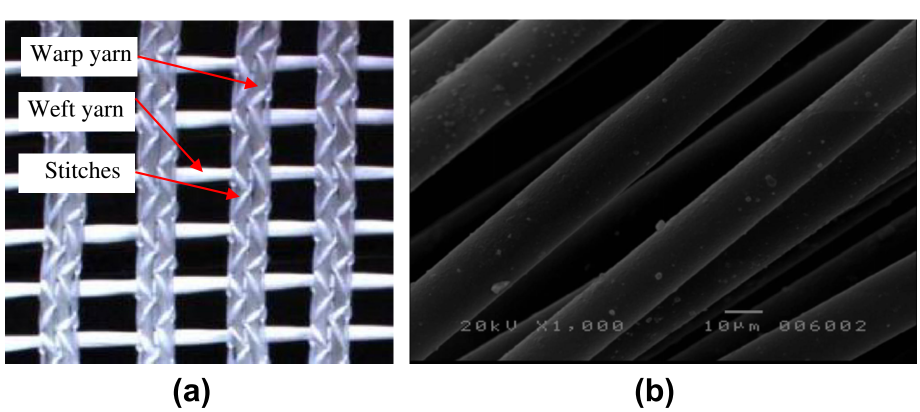

Fig. 1. Warp knitted AR-glass fabric (a) and SEM picture of warp yarn filaments (b).

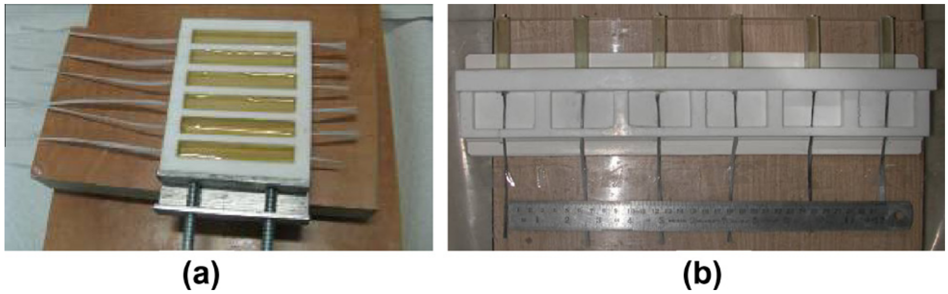

Fig. 2. Special moulds for polyester grip (a) and cement part of pull-out sample (b).

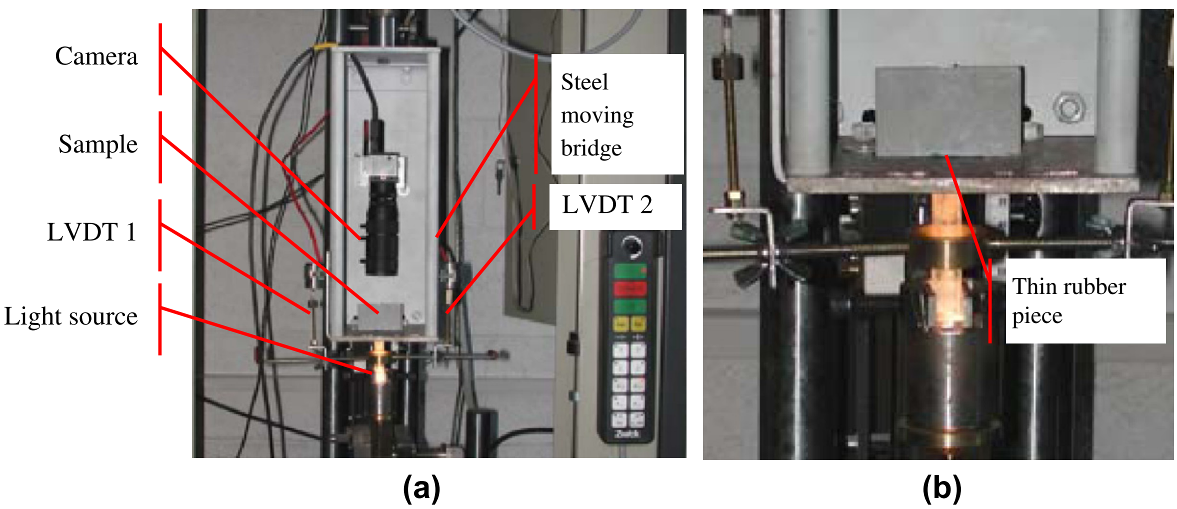

Fig. 3. Pull-out test setup (a) and pull-out sample (b).

energy was divided by the elastic part (area up to the displacement at the maximum load level) to obtain the relative post peak energy. The scheme and the details of this pull-out and fiber failure monitoring system are based on a system developed by Banholzer in Aachen [7].

2.3. Imaging of filaments during pull-out

Imaging of filaments during pullout was obtained using the scheme developed by Banholzer [7] in which light is passed through the filaments from a light source which is placed underneath the pull-out specimen. The light going through the filaments is captured by a CCD camera positioned above the specimen which enables to make a continuous observation of the filaments and record it in a video system (Fig. 3a). The video record by CCD camera was continuous and the video record consisted of three frames per second. Synchronizing the image, force and displacement data have been carried out by Lab-VIEW software. Broken filaments during the pullout cannot transmit the light and so they can not be seen any more by the camera. Thus a video image showing darkening of points indicates the locations of the broken filaments. A digital image analysis technique was used to determine filament failure locations.

The first step of the image analysis was the selection of appropriate frames, which is corresponding to analyzing load step of each pull-out test video record. For this purpose computer software named Ulead Video Studio was used. Desired frames (instant photo-images of a strand) of the video record were captured by using this software. The image processing step of the image analysis was carried out using Matlab R2007b.

Captured photo-images which correspond to analyzing load levels were converted to 8-bit grey-scale picture and then the threshold was adjusted by considering the grey-scale histogram of the picture, automatically. In this grey-scale picture the pixels under the selected threshold level correspond to matrix, voids, light-traces and broken filaments which cannot transmit light any more. Thresholded 8-bit grey scale image were converted to a binarized image, which consists of white and black pixels only.

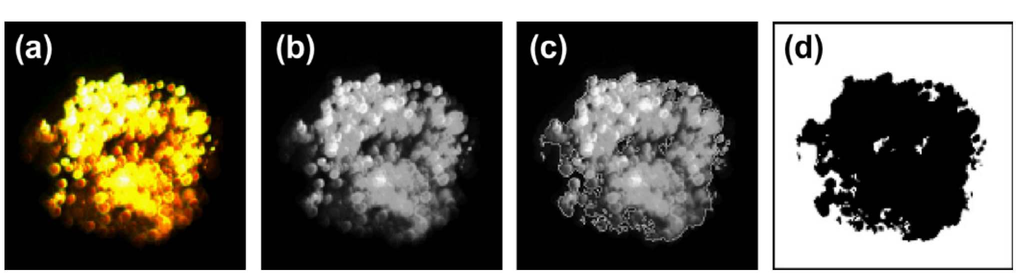

Fig. 4. Captured photo-image (a), 8-bit grey-scale of the image (b), thresholded image (c) and binary image (d).

Fig. 4 shows an example of captured photo-image, 8-bit grey-scale form of the image, thresholded image and binarized image. In this binarized image (Fig. 4d) the black pixels show active filaments which are remaining unbroken at the observed stage of loading. For each picture, the total area of these black pixels, which are showing the active (unbroken) filaments during the pull-out test, was calculated and regarded as active filament area at this loading level.

The active (unbroken) filament area should remain constant as long as the increased load does not result in any fiber breakage; the active filament area should start to decrease when reaching the load level causing fiber damage. At each load level the active filament area was normalized by dividing it by the maximum active filament area and thus, the parameter of active filament ratio was obtained. The active filament ratio is a parameter varying between 1 and 0 (Value of 1 shows unbroken yarn and 0 shows a yarn where all the filaments were broken) and it represents the ratio of unbroken filaments at any displacement and/or loading level.

3. Results

3.1. Microstructure

The microstructure of the yarn after treatment and drying is shown in Fig. 5. In the SFS, SFL and PSP treatments the presence of the particles absorbed from the slurry can be clearly seen. In the case of the SAF treatment the formation of a film around the filaments is evident.

3.2. Mechanical properties

Typical pull-out load – displacement curves of dry and wet-modified yarns are presented in Fig. 6.

The maximum pull-out load levels of the different systems in the wet and dry processes are presented in Fig. 7. The overall load carrying capacities of the various modifications in the dry process were similar except for the film forming SAF system, which shows a much lower load bearing capacity. In the wet process, however, all modified systems showed considerably low load bearing capacity when compared to the unmodified (reference) yarn.

The reduced bond in the wet systems might be accounted for by the formation of a water reach zone around the treated yarn, which results in an effectively high *w*/*c* ratio matrix just around the yarn, leading to the lower bond compared to the dry treatment, where the water is removed before embedding the treated yarn in the matrix. An exception is the SAF in the dry treatment which upon drying leads to the formation of a film, which probably separates between the filaments and the matrix, leading to a reduced bond.

The pullout energies of unmodified and wet and dry modified yarns are presented in Fig. 8. As can be seen the figure, wet treatment seems less effective in enhancing the total energy due to the low bond capacity of wet-treated yarns. On the other hand, the treatments seemed to be effective in enhancing the post peak and relative post peak energy in all the dry treated systems and the wet treated SFS and SFL. In the case of the wet SFS, SFL and the dry SAF significant enhancement in relative post peak energy might be related to the lower bond which can enhance slippage. However, in the dry treated SFL the higher energy cannot be related to lower bond, and perhaps some other mechanism is responsible for this behaviour.

Fig. 9 shows the effect of the modification method (dry and wet processes) on pull-out load and the energies. Drying the treated yarns for 24 h in laboratory conditions has resulted in improved bond capacities (dry to wet ratio greater than 1) for the inorganic slurries (SFS and SFL) and the polymeric material which is not film forming (PSP). However the reverse trend was observed for polymeric material which is in the film form (SAF). The latter trend can be explained by the formation of the film upon drying, which reduces bonding.

The trends for the dry/wet ratio are different for the energies. The film formation of the SAF dry system can account for its high dry/wet ratio for the post peak and relative post peak energy values. In the systems which retain their particulate nature (SFS, SFL and PSP) the SFS systems shows a low dry/wet ratio for relative post peak energy value while the SFL and PSP retain a ratio close and greater than 1. Although the maximum pull-out load levels of SFS and SFL systems in the dry process are similar to each other (Fig. 7), drying considerably decreased the relative post peak energy of SFS modified yarn (Fig. 8). This exception of the SFS system might be due to its smaller particle size leading perhaps to more

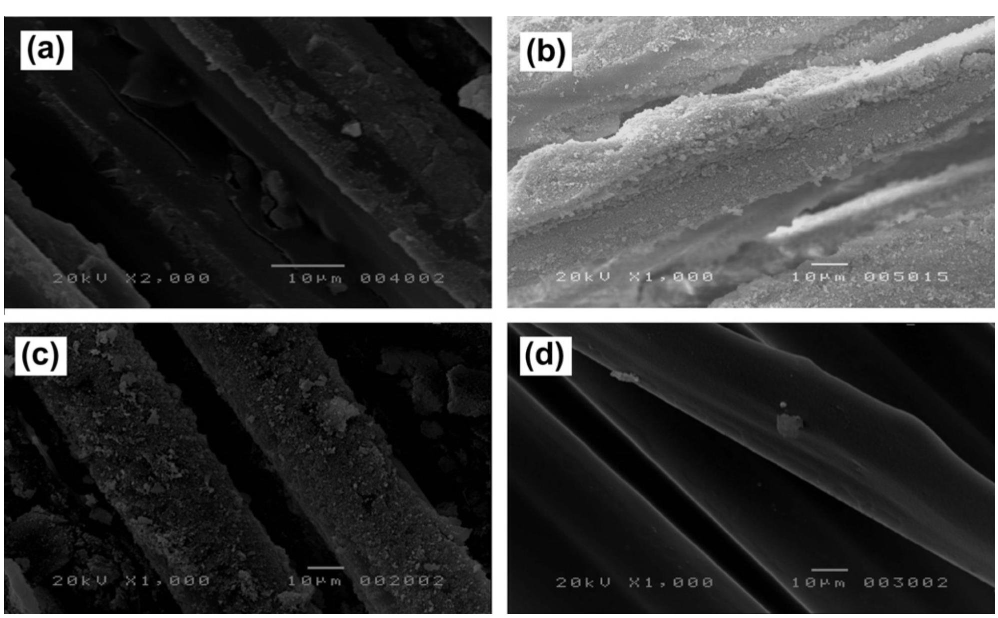

Fig. 5. Filaments in SFS (a), SFL (b), PSP (c) and SAF (d) modified yarns (dry treatment).

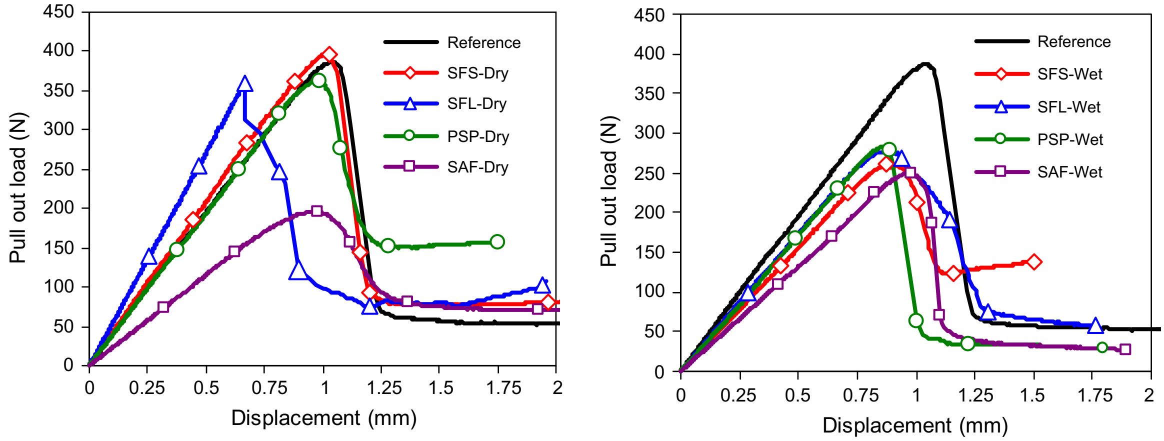

Fig. 6. Typical pull-out load – displacement relationships of unmodified (reference) and modified AR-glass strands in dry and wet processes.

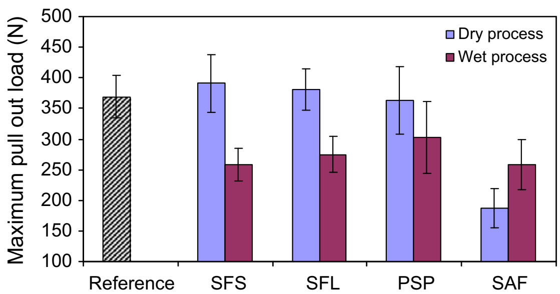

Fig. 7. Maximum pull-out load values of unmodified and modified fibers (the error bars show ± standard deviation).

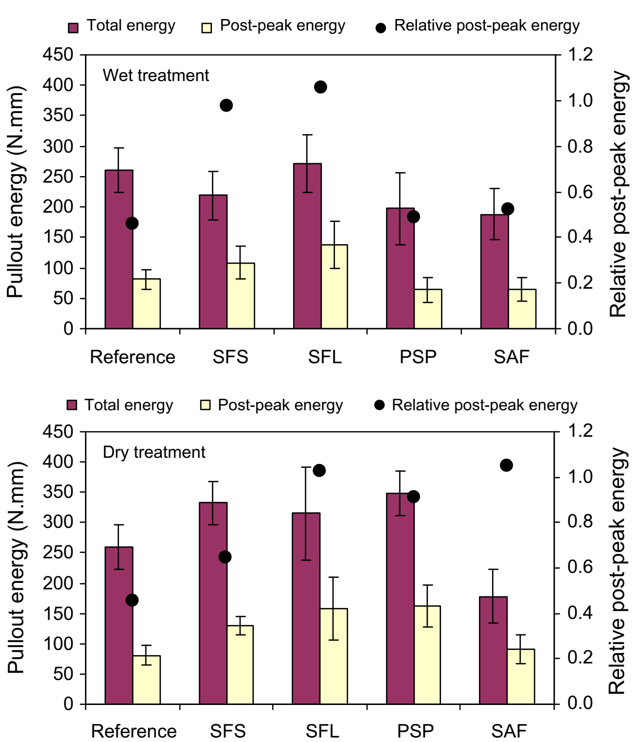

Fig. 8. Pullout energies of unmodified and wet and dry modified yarns (the error bars show ± standard deviation).

effective penetration in between the filaments in the yarn, causing a high internal friction.

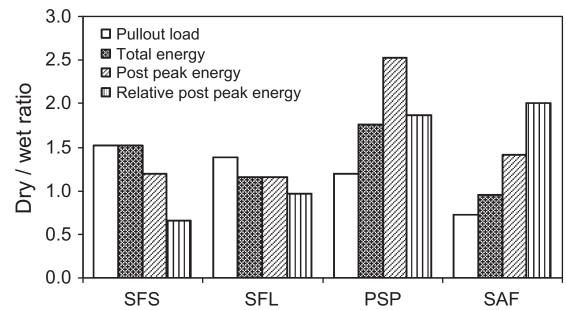

Fig. 9. Dry to wet ratio on pull-out load, total energy, post peak energy and relative post peak energy absorption capacities.

3.3. Failure mechanism of pulled out yarns

Failure mechanism of unmodified and modified yarns has been investigated by determining the active (unbroken) filament ratio during pull-out by the FILT test. Typical pull-out behaviour of unmodified yarn along with the unbroken filament ratio and binarized images of unbroken filaments at different loading stages are presented in Fig. 10. As shown in the figure, considerable amount of filaments (max. 10%) break down in tension before the maximum load is reached. It can be expected that some of the external filaments, which are surrounded by matrix, may break under tensile stress rather than pullout, since the external filaments of a strand might be strongly bonded to the surrounding matrix. The displacement level at which the fiber breakage appears in the FILT test, is also in agreement with the displacement level at which the pull-out curve deviates from the straight line in the ascending part of the pull-out load – displacement curve. Sudden filament breakages and corresponding load drop have been observed (e.g. displacement levels and corresponding pictures marked as c–e in Fig. 10) just after exceeding the maximum load level in the unmodified yarn. This observation reveals that the general failure character of the unmodified AR-glass yarn embedded in a cement matrix is relatively brittle and exhibits sudden breakage of filaments rather than gradual slipping out from the matrix. It was found that just 17% of the filaments remained unbroken at the displacement level of 1.75 mm.

Sudden filament breakage rather than slipping out from the matrix may indicate considerable penetration of hydration products in between the filaments resulting in high internal bonding. This is more likely to occur in the untreated control sample, whereas in the treated ones the spaces are filled with less reactive or

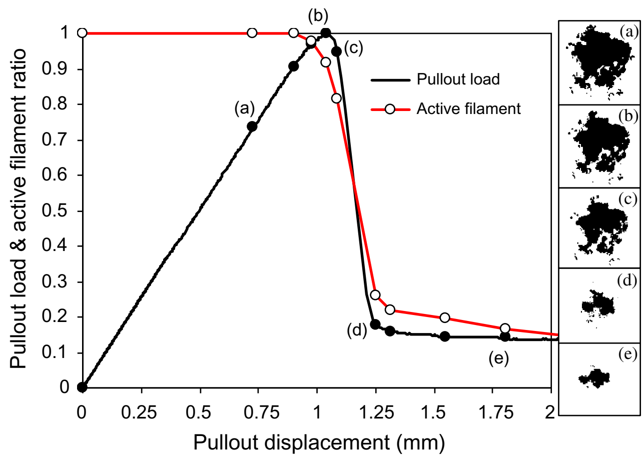

Fig. 10. Typical normalized pull-out load and active (unbroken) filament ratio – displacement relationship together with images of unbroken filaments at different loading stages of unmodified yarn.

non-reactive material which is in a spherical particulate nature preventing deposition of hydration products as well as providing some mechanism to maintain lower internal friction between the individual filaments. Modifying the microstructure of glass strands by sub-micron inorganic particles changes the failure character of the strand, as well as the bond properties. On the other hand, it was also found that the particle size of the modifying material is another important parameter with regards to failure character. Fig. 11 shows the characteristics of pull-out load and unbroken fiber ratio vs. displacement relationship of the yarns modified with the slurries containing SFS and SFL.

As can be seen from the comparison of Figs. 10 and 11, the failure character of the yarn modified with SFS-Dry was similar to that of the unmodified yarn showing sudden filament breakage. However, the gradual filament breakage was achieved by microstructural modification in the system of SFL-Dry. The gradual reduction of active filament ratio against increasing displacement reveals the gradual strand failure, layer by layer from the outer to inner filaments, characteristic of telescopic mode of failure in

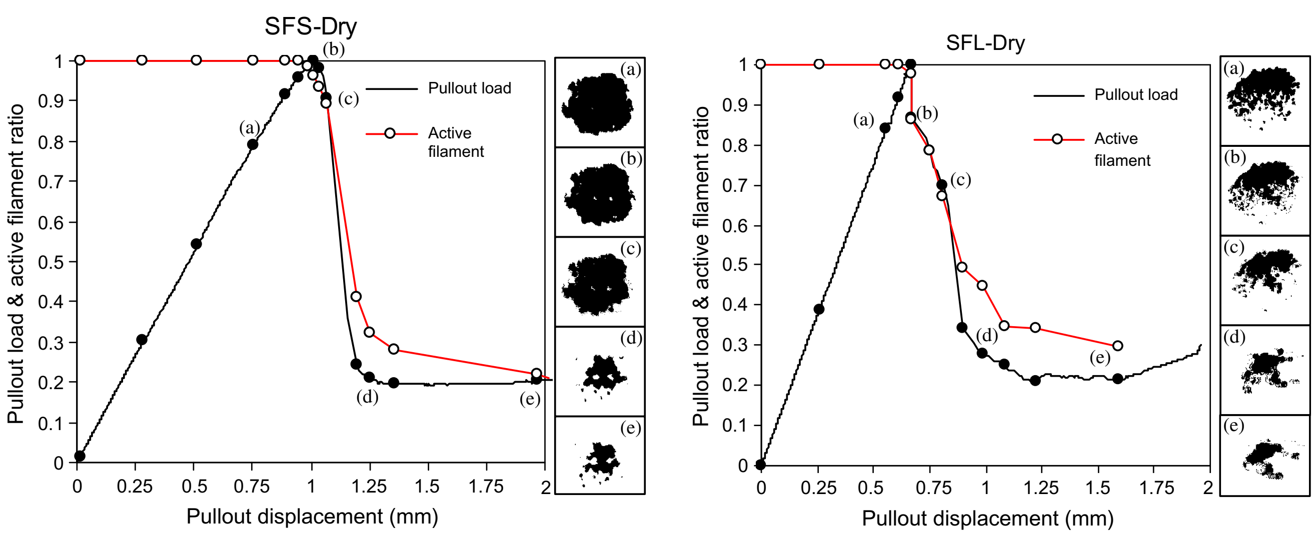

Fig. 11. Typical pull-out load and active (unbroken) filament ratio (normalized) – displacement relationships of dry-modified SFS and SFL yarns (Binarized images represent unbroken filaments at different loading stages).

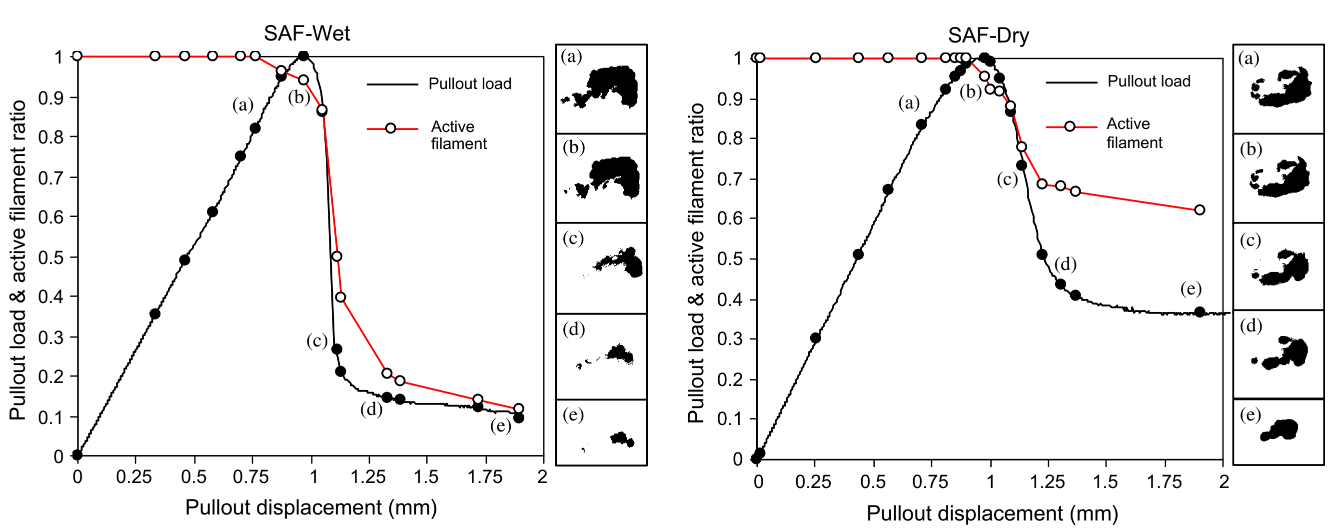

Fig. 12. Typical pull-out load and active (unbroken) filament ratio (normalized) – displacement relationships of SAF modified yarns in wet and dry processes (Binarized images represent unbroken filaments at different loading stages).

the SFL-Dry system. Relatively brittle post peak behaviour of the yarns modified with smaller silica fume particles (SFS; silica fume with smaller particles of 0.05 μm) might be explained by a dense microstructure between the filaments inducing high internal friction and hence sudden filament breakage.

An interesting result was obtained in the film forming polymeric material modification (SAF) in the wet and dry process. In the case of the film forming polymeric material (styrene acrylic polymer, SAF) modification, the load carrying capacity and also the initial stiffness (slope of the pull-out load- displacement curve) of the yarn drastically decreased in the dry process (Figs. 6 and 7). However, the drying has resulted in an increase in post peak energy (Figs. 8 and 9). Fig. 12 shows typical pull-out load and active filament ratio (normalized) – displacement relationships of the yarns modified with film forming polymeric material (styrene acrylic polymer, SAF) in wet and dry processes. The formation of a polymeric film can be obtained only in the dry conditions which facilitate the coalescence of the polymer particles, whereas their particulate nature is preserved when kept wet. The effect of the formation of the polymer film can be clearly seen in Fig. 12: its formation in the dry treatment allows the preservation of the pull-out mode of failure and eliminates fiber breakage after the peak load.

4. Conclusions

- Sub-micron particle slurries can be used to treat bundled reinforcement by introducing in between the filaments in the bundle particles which are in the 50–200 nm size range. This modification of the bundled reinforcement was found to affect the nature of bonding.
- The relatively brittle nature of failure of the unmodified glass fiber strand can be turned into a less brittle and telescopic mode of failure by fiber modification. The efficiency and magnitude of the modification is highly dependent on the filler type and the production process (wet vs. dry).
- Sub-micron particles absorbed in between filaments prevent the cementitious action within the strand and friction and rolling effects of sub-micron particles can modify the failure mechanism of AR-glass strands to become a slippage one or gradual filament breakage, layer by layer.
- The best performance with respect to load carrying capacity was obtained with SFS system in the dry process and the PSP system in wet process. In terms of post peak energy absorption capacity, the best performance was obtained with SFL in the wet and dry processes. The optimum behaviour in terms of improving both, bond and energy, was obtained with the dry treated SFL system.
- When the modification is of a film forming nature, the soft nature of the film surrounding the filaments may limit the bond capacity of the filaments with cement matrix and hence decreases the pull-out strength, but lead to enhanced slippage behaviour, as observed in the SAF dry system.
- The FILT test can be used as an effective tool for characterization of the nature of the failure mechanism of the AR-glass strands modified with different types of sub-micron particles

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