STUDYING BEHAVIOR OF FIBER REINFORCED COMPOSITES USING SCANNING ELECTRON MICROSCOPY ANALYSIS - A REVIEW

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Abstract- The microscope has been a versatile tool in the study of cementitious composites since the development of these materials. The scanning electron microscope (SEM) is one of the most developmental instruments available for the analysis and examination of morphological characteristics of fiber and its matrixes. This paper gives a brief review of the accuracy of scanning electron microscopy method for understanding performance of fiber reinforced composites and prediction of behavior of fibrous concrete. It also focuses on the scope of the scanning electron microscopy (SEM) method in the near future in fields fiber reinforced concrete research work. Selected researches of SEM usage in FRC have also been discussed. The electron microscopy is used as an investigation instrument in understanding the potential of the varying behavior of different fibrous composites at different conditions, an investigation mechanism in making suitable matrix, and a diagnosis equipment on problems like cracking. The paper also investigated how microstructural analysis reveal the expected mechanical behavior.

Keywords- FRC, performance of fibrous composites, scanning electron microscopy.

1 INTRODUCTION

The use of SEM studies in recent years has become increasingly popular in concrete studies, similarly this approach remains important in the study of fibrous composites for many civil engineers. The importance of the versatile method is further incorporated as an electron microscopy [1]. An electron microscope equipped with an X-ray spectrometer for diffusion energy can entertain very useful interpretations in analyzing concrete problems with high resolution images such as alkali-aggregate reactions in aggregates [2]. The performance of high-performance concrete cannot be adequately evaluated without examining the features of interfacial microlevel analysis of high-performance concrete, as seen under SEM [3]. As the use of electronic microscopy is becoming more expensive, there is a chance that the process will be widely used and understood [4]. This understanding of the underlying mechanism, the opportunities it provides for understanding the behavior of fibrous composites and analyzing integrated fiber problems, and strives for a recommended opportunity in experimental programs where intensive studies can make good output from SEM.

Microscale surface morphology analysis of the tested FRC samples can be easily revealed by SEM observations. To have reliable information about the microstructure, a large number of images were collected and analyzed for various microscale studies using a scanning microscope of mixed fibrous concrete containing fly ash and bottom ash [5]. It was concluded from the SEM experiment that when mixing the concrete, this light, C-S-H gel, covered the top surface of the concrete. This confirms that the C-S-H gel acts as a major handle on the concrete core to achieve the required strength, which is in consistent with the results in previous studies. [6]. The morphological analysis such as interfacial bonding, dilation of the matrix, fiber pulling or fracture between the fiber and matrix in treated and pretreated condition was studies in the composites are examined and analyzed using SEM analysis [7]. The investigation of thermal properties hybrid fibrous composites and its effects of chemical treatment was found using Thermogravimetric analysis (TGA), Dynamic Mechanical Analysis (DMA) and differential scanning calorimetry (DSC) and the correlation of change in fiber morphology was verified by SEM analysis [8].

In early 80s, SEM image analysis was used to study the surface morphology and fracture pattern during cracking. The separation of cracks, branching and the debonding of interface provide energy absorbing capacity of the steel fiber composite. This energy absorption was verified as additional effect to fiber pull out [9]. SEM analysis of broken specimens of shear and compression shows a considerable difference in the fiber-cement transition. For un-aged fibrous composites, the transition zone around the fibers was open and having gaps and microcracks, and this was accompanied by a greater fiber pull out in contrast to fiber exposed to natural aging and carbonation. [10]. SEM analysis of Chemically Treated and untreated coir fiber micromorphology have shown different characteristics i.e. the former has partially split unit cells due to removal of wax and larger pits and the latter have uniform unit cell and shallow cavities due to lignin and fatty substances. and mechanical results are verified by the evidence of SEM observations. [11]. Carbon nano fiber and nano materials significantly increase the mechanical performance of concrete. [12] observation of cracked surface in glass fiber reinforced cement revealed the effect of aging on the pull out of fiber from the matric during fractures. The images obtained showed how the surface were modified and increased pull out of fiber from the matrix. [13] The plain-strain fracture test was conducted by applying load on the composite specimen on the y-axis in the direction of loading and causing the propagation of the crack and the ZX plane. Carbon fibers are likely to be attacked along the bead printing direction because of its behavioral characteristics. Therefore, in perpendicular samples, the associated fibers may not contribute much to the stiffness. Because of this, the inter-bead contributed negligently to the cracking conditions. In contrast, where the beads are aligned with the fracture plane and have contributed to the fracture elasticity. The fibers within the conventional overlap size should provide surface resistance So, a large amount of fiber resistance and uneven topography are expected from the surface of the perpendicular beam fractures. In the case of 45 oblique samples, both cracks contributed equally to the crack process during load application [14]. SEM analysis is crucial in fiber reinforced composites and to the best of author knowledge no research is done on revealing FRC behavior using SEM method. A systematic literature review of SEM analysis on durability of natural, synthetic and steel fiber will be helpful in the application of the fibers.

2 SCANNING ELECTRON MICROSCOPY

The SEM is constructed of a high-resolution image with an electron optical strand that produces and concentrates the electron beam above the visible surface. An evenly sized plate eliminates the energy of the electrons that strike through a series of certain scattering events. In cement and concrete attachments this volume can be 2-3 microns in depth and width [15]. Scanning low-power electron from the upper parts of the chamber are reflected on the surface layer of the specimen. The complex comes from the applied industry and is a subtle signal that can be detected such as back electrons, secondary electrons and X-rays. The unchanged electrons have high potential as they are dispersed in the process depth and are measured by detectors and converted into high resolution images. Comparison of concentrated electronics is very efficient in detecting basic differences, often in material that is distorted, and topographical distortion can still be seen if the difference in the standard range is large [1]. The basic comparisons, in the case of compounds, are equal to the atomic number of substituted elements depending on their relative behavior [16].

2.1 Sample preparation

Optimizing the SEM view set in a separated and soft spot is easy by comparison. Paste at the beginning of 2 hours can be optimized for SEM observation [17]. High resolution images without polishing allow the examination and identification of crystals in cracked, aggregated surfaces that can be composed of polished plates [18]. Backscattered electron images in the molded area were particularly sensitive to unwanted changes in the sample in preparation. The sample structure can be stabilized by adding a low viscosity epoxy due to the Vacuum installation method, which is why the unwanted change can be reduced [19]. Some investigators consider that the invisible and open wire circuits of both scattered electrons and scattered back electrons give sufficient results when compared to embedded and polished epoxy samples [20]). For the preparation of both compacted and molded samples for SEM testing including pre-test suspension, this procedure may alter the microlevel coating surface and results in smaller cracks.

2.2 Image analysis

Backscattered electron Images make provision of examining the specific gravities of different phases at microlevel analysis directly. The characteristics of hitting of electrons scattered from a specimen locality on the sample surface solely depends on the structure and atomic/compound number of the material at that position. It was established to possibly categorized four stages: grains of un-hydrated cement takes shape as bright, pores black, other hydrated compounds darker grey and calcium hydroxide light grey [19]. The Backscattered electron Images examination process is developing and is mostly used together with other imaging investigation tools. SEM method has made elaboration to the systematic analysis of the different stages in cement composites with expanding refinements [21]. Voids in fibrous cement composites can be distinguished, with the help of high-resolution images in software analysis, with respect to absorption and porosity, grain sizes, distribution and their inter connectivity. [15]. Now, there is important progress on the hardware side, particularly in the environmental scanning electron microscope. Optimistically, this will mitigate the issues related to damages in sample during electron microscopy observations [22].

2.3 Usage of SEM

SEM have been used by many researchers in the field of concrete and cement composites, the detailed literature of the various applications is given below.

2.3.1 The aggregate/paste interface

The two-dimensional system of adhesion and bonding to concrete can be better understood by microstructure studies using SEM. The model for the relationship of porosity and tensile strength was also considered as a system for both coating and bonded surfaces [6]. The delicate center of the joint and the adhesive shown by its magnitude, have been considered as a weak concrete phase for some time. This is a sensitive, compound-bonded, repeatable C-S-H film [23]). The SEM studies of this crucial bonding in concrete are easily understood using high resolution images. The space between the adhesive particles in the concrete is almost up to 100 microns, while the average compaction area and the adhesive are about 50 microns. A thorough examination of this guide and how it can be modified or installed has greatly assisted in the construction of the concrete for the strength and desirable properties. [6].

2.3.2 Effects of various Admixtures on the microstructure and properties

The installation of various admixtures has always played critical role in the manufacturing of desirable concrete these days to enhance its efficiency, strength, durability, decrease in dampness and cracking. Upgraded and new admixtures are still required for desirable properties. Mineralogical admixtures such as fly ash and silica fume, are widely used in advanced and local concrete. The small effects of the admixtures structure cannot be ignored in different studies [16]. The incorporation of various mineral admixtures alters the properties of the surfaces through immersion pores and grains [24] .A convincing conclusion was drawn from research on the related effect of silica fume on concrete and adhesive, the supplement did not improve the adhesion strength, but the synthetic concrete was stronger than the adhesive [15].

2.3.3 Many sites practice

Higher elevated temperatures due to cement delivery, materials use, different handling or concrete than curing and concrete vibration techniques have an impact on concrete properties. [25] Redon and jean used SEM and other analyzes to determine the effect of the therapeutic regime on the concrete microstructure. It was confirmed that pre-treatment of the joints with cement-lubricant or sliding off of the fume reduced the absorption and durability of the adhesive and adhesive linings. Many studies have shown the improvement of the paste/aggregate surface by pretreatment of concrete constituent.

2.3.4 Concrete durability

Various investigators have studied the contribution and effects of other factors on the performance and durability of concrete, through SEM testing. This includes cracking results similar to those found in concrete with steam treatment [26]. Tightening the bonding and its strength at paste/aggregate zoning, and the importance of paste/ fine-aggregate interface instead of paste and coarse aggregate [27].

2.4 Output of SEM

SEM is important for developing our learning of the inner properties of concrete. Cement hydrated products are not only complex but also have structural importance [26]. Structurally, the paste contains crystalline, quasia-morphous and amorphous phases. In terms of particle size, the ratio is about the size of a micrometer. The particle assembly to form an adhesive micostorganist, on a micro-morphological scale, is in the tens or hundreds of micrometers. SEM has good solutions to these calculations and has been widely used in systematic studies. There was an increasing interest in concrete research in the 1970s. Cement concrete studies under SEM revealed "internal product" (cement hydration by the incorporation of water molecules into a waterproof concrete), and "external product" (made from finishing and burning without concrete)[28].

3 SCANNING ELECTRON MICROSCOPY IN FRC

The multidirectional divided and oblique cracks were initially studied in Steel fiber reinforced cement in SEM observation. The acetone and trichloroethane fibers of a smooth and clean surface were inspected under SEM chamber prior to loading [9]. The cracking pattern was also observed after the loading of the preferred behavior of the reaction coupled with the extreme adhesion of the cement paste to solid and solid steel. Internal recognition of the interface at the bottom of the frame indicates that the fracture mechanism in the matrix around the fiber is catastrophic, and in many cases, characterized by branches, sometimes terminated by the formation of small microcracks around the larger one. The effects of the chemical treatment on surface polypropylene fibers (PPF) and the relative strength of PPF reinforced concrete have been investigated by N. loavat et al. There were three treatment groups in this study, all containing polypropylene fibers, and a control group. The first and second treatments were performed by adding PPF to a solution of mild aleic acid and an alkaline solution of the B-H group respectively. Some failed samples were randomly selected and examined using the Scanning Electron Microscopy (SEM) method. This was used to investigate the characteristics of the optical interface between the concrete tray and the fiber surface. The polypropylene fibers failed to be deposited and gold was synthesized prior to SEM examination. Acid carbon atoms are attached to the surface of the PPF, forming an incomplete bond. This will render the earth inactive and in the surrounding matrix of the cement and consequently allow crystalline growth of hydrated materials in the form of Carbon-Hydrogen crystals on the fiber surface [29].

The effect of microstructures and the interface on cementitious materials made with steel slag were studied by yue et al. cotton-based resin and cotton-formaldehyde resin were added with iron slag and cement at 2: 3: 5. The strength, flexibility and durability was assessed after being immersed in a mixing water for 24 hours. Microscopic composition of the hydration products was observed with SEM. The analyzed images show that the insoluble fiber surface was smooth and the formaldehyde-coated surface was well integrated with a gel that sealed the gaps in the matrix, the mechanical strength increased by 18%, and the water resistance significantly improved, that is well in line with the physical characteristics of the microstructures and the study results show that urea formaldehyde resin and fiber treated have good adhesion properties[30]. Attempts were made by [31] geng and leon to connect the extracting the fiber from the matrix to the microstructure of the material and the matrix interface. The microstructural characteristics of fiber (steel, nylon and polypropylene) / matrix interface were examined during the fiber pull out and debonding process. Because the fiber output was found to be weak for subsequent compression, microlevel studies were performed on the frictional frames and lateral compression. SEM and energy-dispersive X-ray (EDX) analysis was performed in four different phases: (a) before debonding; (b) immediately after debonding; (c) smaller sliding distance; and (d) the greater sliding distance. It was found that the molding area was placed under tension, while the steel surface was placed under plastic deformation. As discussed in the SEM study, when applying lateral pressure to the mortar during pullout of fiber, the effect of abrasion has been much greater on the steel fibers.

Lu Shang et al [32] studied glass fiber reinforced cement composites (GFRCC) with Portland cement and high fly ash content as a matrix. AR glass fiber 12 mm long with 2% volume content was used as reinforcement. The effect of ash content, treatment duration, and accelerated aging on GFRCC were investigated. The addition of fly ash reduced the initial capacity in term of early strength of the GFRCC. The higher the fly ash content, the greater the reduction in strength from 7 to 28 days, flexural strength improved again, and continued to increase after 28 days. A very poorly damaged AR glass fiber in the Portland cement matrix without being filled with ash after 1 day of rapid acceleration in 80 ° C was observed using SEM. Glass fibers from a matrix containing 60% ash showed more smooth AR glass after 1 day of rapid aging. D.A. Silva [33] investigated the durability of reusable PET fibers with the material having properties as shown in Table No. 1, that were embedded in Portland cement materials. Two separate testing methods were approved to study the degradation of fibers in cement matrix. For the first time, the fragments were observed by infrared spectroscopy and scanning electron microscopy (SEM) after their exposure to solutions of alkaline substance. In the second process, a fiber-reinforced mortar model was created and tested to find other mechanical properties. Fibers are immersed in Ca (OH) saturated solution with a Lawrence solution for 150 days at 5, 25, and 50 C and then performed microscopy, revealing details on the materials used to produce the mud. Mud samples were molded and stored in lime water for two weeks prior to the laboratory description and at 42, 104, and 164 days of age, mortar indicators were evaluated for compression and tensile strength. The thicknesses of the matrix fragments obtained from the split tensile test were observed by scanning electron microscopy (SEM), in order to detect any evidence of deformation in the cement paste. the results were in line with previous findings on cement materials that used low volume PET fiber in cementitious composites. Figure 1 shows the micrograph inside the mix after 164 days. As can be seen, the surface of the fibers appears to be very hard, indicating that the fibers are attacked by something from the cement paste matrix. SEM micrographs allowed the interpretations of the rate of degradation of fibers within the membranes after 164 days. All the analyzed fibers showed some apparent corrosion. In some regions, complete destruction of the pieces of fiber may occur. The adaptive performance of cement mixtures involving low frequency cold plasma treated polypropylene (PP) was investigated in this study. The Potential physical changes in surface layers of the fiber were detected by scanning electron microscope (SEM) on micrometer scales. The experimental results have shown that normal plasma treatment conditions lead to improvement of toughness of PP fibers in the composites. From the SEM observations and measurements of wettability, it was argued that the main mechanism for improving performance was the result of an increase in adhesion between the PP and the matrix [34]. A. A. Ramezanianpour et all studied the effect of polypropylene fibers on permeability in concrete. Water penetration, ultrasonic, and sorptivity tests were performed in order to signify the effects of PPF on durability. The study of microstructures and interfacial transition zone reveal that PPF can possible minimize permeability and porosity by pore blocking [35]

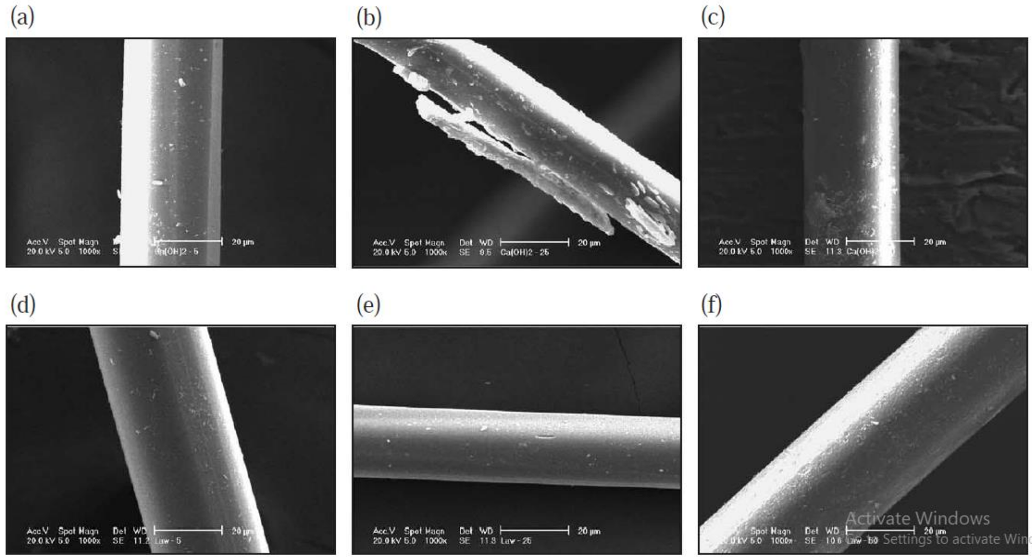

Figure 1. Micrographs showing the aspect of the PET fibers after 150 days of immersion in Ca(OH)2 solution at (a) 5C, (b) 25C, and (c) 50C, and in Lawrence solution at (d) 5C, (e) 25C, and (f) 50C [33].

F. Elgabas et al, experimentally studied that the physical, mechanical, and durability characteristics of basalt fiber-reinforced polymer (BFRP) bars. Three types of BFRP bars were investigated and Analyzed results postulated that the BFRP bars were of satisfactory performance and can be classified in the same general class as grade II and grade III GFRP (according to modulus of elasticity). The results confirm that the basalt fibers and resins used in this study were not affected by this condition. The deterioration of the strength seen in the BFRP bars was included in the fiber-matrix, which confirmed the poor bond between the resin and basalt fibers, as confirmed by SEM [36].

Table No 1. Characteristics of PET fibers[33]

Physical properties	Melting (C)	252.8
determined by DSC	Crystallization (C)	95
Mechanical properties	Strength (MPa)	323.5
	Elongation (%)	70.7
	Yield stress (MPa)	196
	Elongation (%)	7.18
	E (MPa)	41.0
	Toughness (MPa)	17279.0

Analysis of the morphological features before and after splitting tensile test was studied using scanning electron microscopy (SEM) technique to follow the failure examination in carbon fiber reinforced polymer (CFRP) and epoxy resins. SEM Micrograph analysis of the CFRP plate prior to the split test shows the presence of some manufacturing complexity and faults, which can alter the properties of the materials. Micrograph analysis after rigorous examination shows that the propagation of the crack begins in the zones nearest to production defects, which results in the pullout of carbon fiber rather than fracture. Thus, cracks propagate through the contact surfaces that affect the strength of the interaction between the matrix and the carbon fiber. For epoxy materials, microscopy experiments show that even though epoxies adhere to phase separation prior to complex experiments, the failure zone is defined by fine granular particles attached to the matrix, and the material fails aggressively when the force exceeds these bonds. The mechanism of failure of each element related to mechanical properties and the behavioral characteristics of the matrix was understood by SEM observation [37]. The cross section between FRP and substrate is reported to be the weakest link to other FRP reinforced systems, such as FRP-concrete and FRP steel system. Compared to the FRP-concrete and FRP-steel, these findings confirmed that the wood substrate is a more sensitive surface than the interface of the FRP wood system. The wood adjacent of the composite material deteriorated faster than the existing connection with heat exposure. Such information can advance understanding of the fire behavior of FRP and wood, and provide the basic details of fiber design, attachments or materials for combating firewood with better fire resistance[38]. M. Usman investigated the effect of natural weathering on prestressed concrete girder made with steel fiber reinforced concrete and polypropylene fiber reinforced concrete. The specimens were exposed to natural environment for 36 months. The durability properties were investigated, and SEM analysis was done. SEM microgram of steel fibers in confined environment are shown in figure 2(a), since the fiber were not exposed to natural environment and thus fiber are in normal condition and has no effect of rusting. The high-resolution SEM micrograph of the fibrous concrete exposed to natural environmental conditions are shown in figure 2(a) and figure 2(b). The effect of natural weather was examined in the area around the steel fiber (SF) and it was concluded that steel fiber were rusted and concrete was in normal condition. The more high-resolution image in figure 2(c) verified that steel fiber corroded due to rusting and this was happened due exposure to atmospheric condition [39].

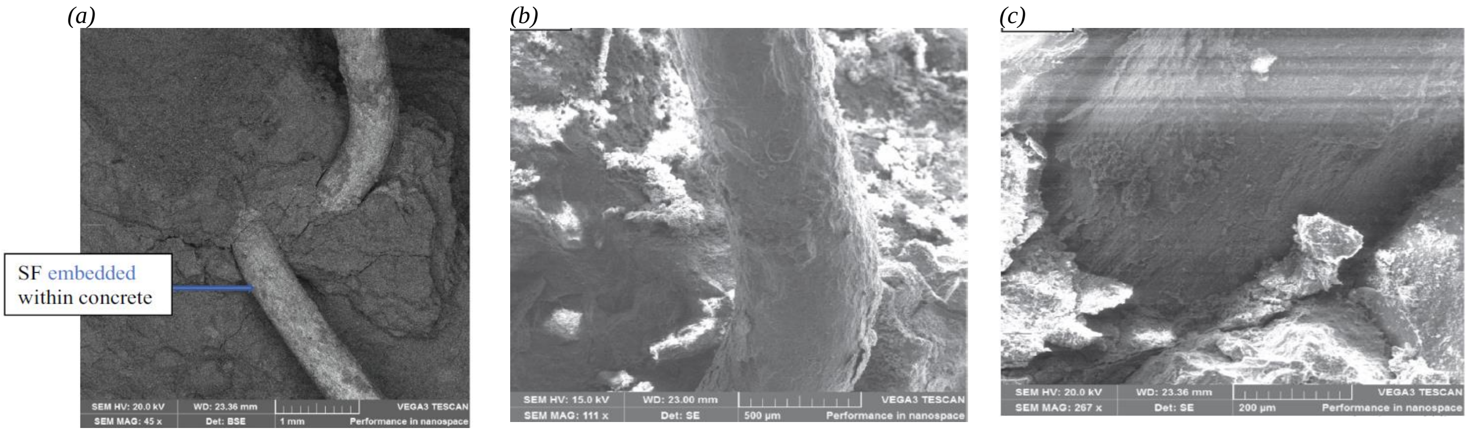

Figure 2. (a): Steel fiber embedded in concrete in confined condition, (b) and (c): Steel fiber in concrete exposed to natural weather conditions [39]

V. afroughsabet et al investigated properties of hybrid-fiber-reinforced concrete (HyFRC) constituted with expansive Type K cement and addition of fiber volume fraction of 1%. The microstructural and mechanical properties of steel double hook end (DHE), steel hook end (HE) and polyvinyl alcohol (PVA) fibrous composites were studied. The addition of type K cement improved volume stability at drying condition and enhanced pull out resistance of steel fiber by 26%. The SEM micrograph of fiber embedded in paste as shown in figure 3 were investigated. As shown in figure 3(a) on a fracture surface of matrix having 0.5% PVA fibers. A half embedded PVA fiber in matrix (solid square in red) and the other end of the fiber (red dashed square) shows large deformation, possibly this could be a fracture too. This indicated that a very well embedded PVA fiber in the mix increased resistance against cracking. In figure 3(b) a very tight bond between mix and fiber was observed and the mix was still tightly attached to steel fiber even after fracture. The SEM observation therefore confirmed that the bonding between fibers and matrix was solid and could significantly increase the fracture toughness of the mix [40].

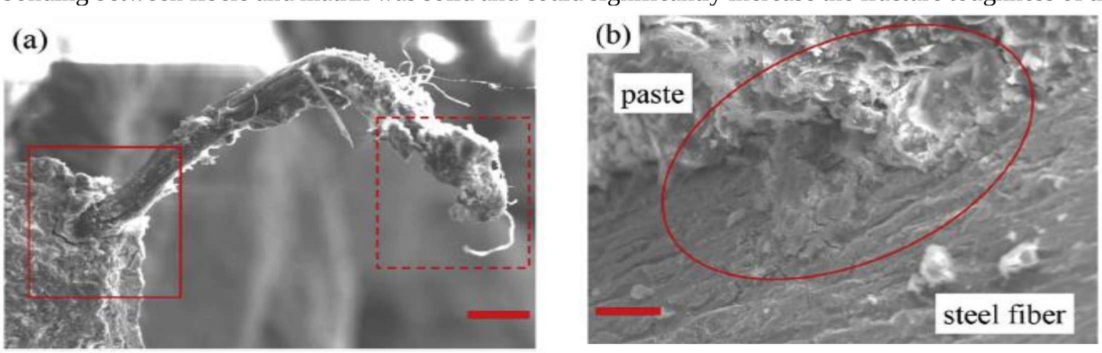

Figure 3. Microscale morphology of: (a): HE0.5 + PVA0.5 paste and (b): DHE0.5 + HE0.3 + PVA0.2 paste after 28-day curing. Scale bars are 20 µm. Red squares and circles indicate details of PVA fiber and steel fiber, respectively [40]

4 LESSONS LEARNED

Form the relevant literature review it is postulated that fiber reinforced composites enhance flexural performance, split tensile strength, toughness, ductility, microcracks resistance, corrosion resistance and reduces early age cracks. Bridging of the fiber in the matrix enhances strength and ductility. Pull out of fiber can be decrease by certain chemical treatment of fiber and surface treatment as it makes the surface rough due to removal of wax. Analyzed fiber matrix interface have elaborated different morphology of rough and serrated surface and its possible output in the fibrous composites. Continuous phase and a granular phase elaborated through BSE, verified the distribution of fibers in the fibrous matrix. Fiber continuity and fiber alignment can alter the performance in cracking pattern, more finding can conclude its effects. fiber volume fraction and resin volume fraction can be easily found through the study of high-resolution images and its outcome can be postulated with detailed studies of the matrix in different phases.

5 CONCLUSION

Following conclusions were made after extensive literature review:

- SEM analysis supported the investigations of effect of Distribution of fibers, its continuity and fiber alignment on mechanical properties.
- Surface treatment of the fiber make it rough and it can decrease the pull out of fiber from the matrix.
- SEM analysis can verify the bond between fiber surfaces and matrix and hence porosity and absorption of the fiber reinforced composites

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