A study on fiber/matrix contour and interface/interphase integrity by SEM and AFM techniques

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Abstract

The characterization of fractographic features associated with fibers, matrix and also interface/interphase has been emphasized here to analyze the surface topographical contours across the glass fiber and epoxy matrix. Atomic force microscope (AFM) and Scanning electron microscope (SEM) were performed to characterize the micro-failure features (like micro-voids, small matrix and interfacial cracks) and structural homogeneity/integrity of composites. AFM study showed that thermal conditioning has resulted non-homogeneous degree of cross-linking reflected by the height-scan images. The uneven post-curing may adversely affect the stress transmissibility integrity of interphase region, which may eventually lead to changes in thermophysical, mechanical and chemical characteristic of fibrous composite. Scanning micrograph indicates the increase in cusp thickness due to increase in plasticity of resin matrix.

Key words: Polymer composites, Cross-linking, SEM, Cusp, AFM

1: Introduction

Polymer composite technology is based on taking advantage of the stiffness and strength of high performance fibers by dispersing them in a matrix, which acts as a binder and transfers forces to the fibers across the fiber/matrix interface. Severe environmental exposure affects physical and mechanical properties of polymer composite materials resulting in an undesirable degradation (1). An interfacial reaction may impart various morphological modifications to the matrix microstructural in proximity to the fiber surface (2). The interfacial adhesion and mechanical properties can be improved by increasing the fiber surface roughness particularly on the nanometer scale (3). As a result, extensive research has been devoted to the study of microstructural assessment of GFRP composite. The work represented here has identified both surface and interphase properties interms of topography, fractography, adhesion and stiffness characterized by AFM, SEM the nanometer scale, which are essentially for the understanding of the macromechanical response to fracture. Special emphasis is placed on the local characterization of the fiber surface topography, interphase mechanical properties and adhesion features. The nanoscale crosslinking process of the epoxy resins near fiber surface influenced by the thermally conditioning treatment is analyzed by AFM and SEM.

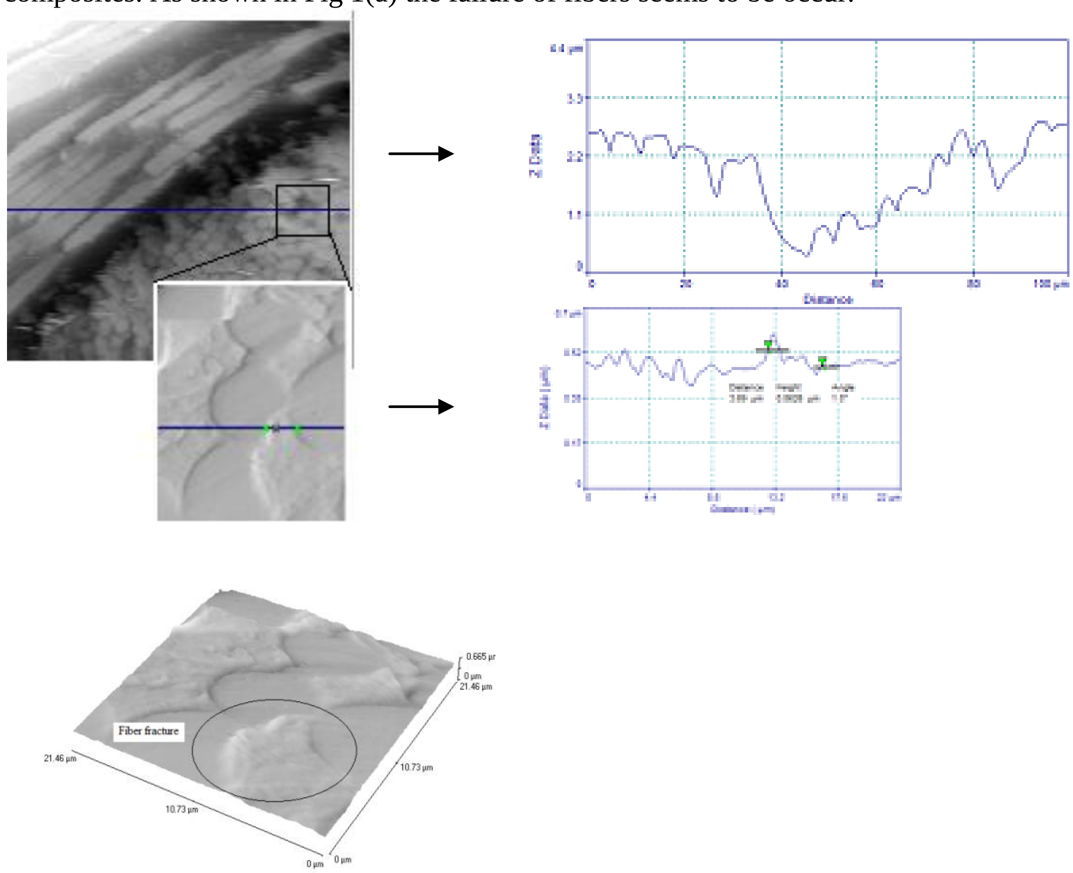
2: Experimental

The fracture surfaces of advanced polymer composites fibers were studied. The composites were treated to above glass transition temperature subjected to 3-point bend test until failure for a certain period of time. Then they were characterized with SEM and AFM techniques. E-glass reinforced epoxy polymer composite was fabricated by hand lay-up method. The composite panels were cut into small pieces which were made into a plug with the cross section exposed for polishing. Polishing steps covers all the grinding papers followed by cloth polishing and then 1μm alumina particles for micropolishing subjected to 30 min for each samples. Thermal conditioning was conducted by placing the specimen as prepared at

+60°C for 1 hr in oven. After treatment the specimens were taken out and scanned by AFM. AFM demonstrates resolution of fraction of a nanometer by feeling the surface with a mechanical probe. These images in contact mode with a conducting P(n) doped silicon tip were obtained with a SPMLab programmed Veecodilnnova multimode Scanning Probe Microscope. The scans were taken at scan rates of 1 Hz. Images are taken to analyze the surface topography in micro and sub-micron levels. However, 3D micrographs can be obtained from the analysis. SEM

3: Results and Discussion

The AFM height –images scans of untreated glass fiber reinforced composite are shown in Fig 1 (a) and (b). Remarkable failure modes can be observed between the untreated and thermal conditioning GFRP composites. As shown in Fig 1(a) the failure of fibers seems to be occur.

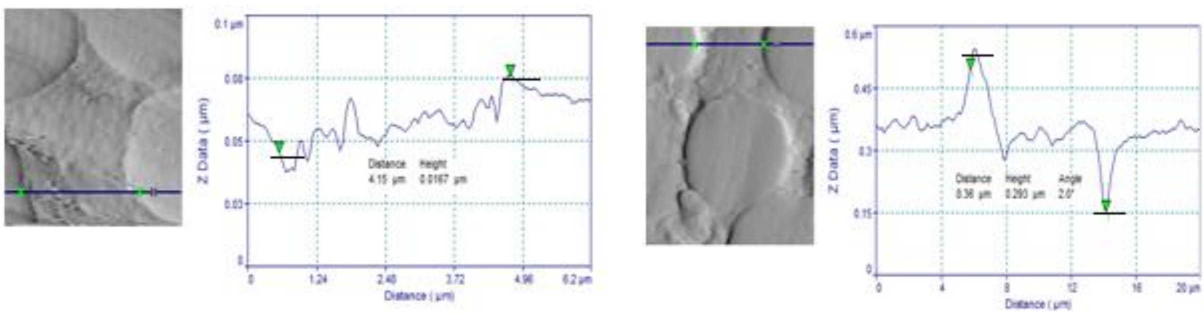


(a) (b)

Fig 1: AFM topography images of untreated GFRP composites (a) line analysis of image (b) 3D image analysis

In untreated sample, the failure of fibers may be due to either by over loading or extensive strain. In PMC, the fibers have greater strain-to-failure. Stress concentrations from matrix cracks and fiber/matrix debonding can also promote fiber breakage which shown in Fig 1(a). If a fiber fracture occurs, the matrix translates the load to the neighboring fibers in the composite. The stress concentration at the broken fiber ends, unless dissipated properly, may induce failure in the adjacent fibers and tends to catastrophic failure. High stress concentration is expected to develop in the matrix near the fiber ends and the voids created by the broken fiber (4). Because of this the fiber/matrix interactions in the vicinity of fiber fracture also pompous. The three-dimensional morphological characteristics of fiber fracture are shown in fig 1(b).

After thermal conditioning at below glass transition temperature (+60°C) for 1hr, the interface/interphase region is likely to affect which shown in height image analysis (Fig 2). The height distance between two peaks was 4.15µm. The sharp fall of graph near the peak shows degradation of interface/interphase region. Between these two peaks the matrix plays dominate role for failure. As the non uniform degree of cross linking of matrix increases, the chain network as a whole becomes like to uneven. This should shift the tendency from a more localized shear banding mode to a more homogeneous diffuse shear yielding mode. The thermal misfit strain can also result in debonding effects at the interface region (5).



(a) (b)

Fig 2: AFM topography image of treated GFRP interphase failure after treatment.

The SEM images of thermal conditioning of GFRP composite are shown in Fig 3. It is found that the cusps are formed with increasing manner. This may be due to, plasticity of the epoxy matrix at high temperature. The cusps resulting from the microcracks are thicker and have undergone greater deformation than the room temperature. The increase in cusp thickness implies fewer microcracks have formed because of increase plasticity (6).

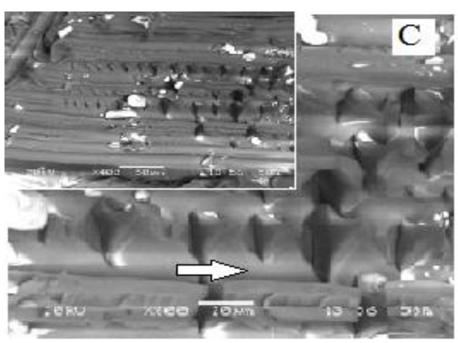
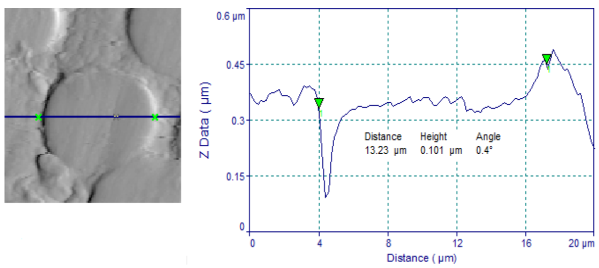
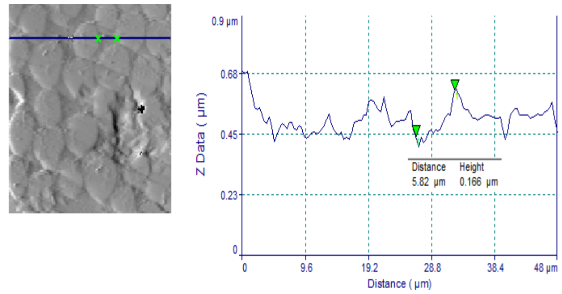


Fig 3: SEM micrograph matrix failure of GFRP composite after treatment.

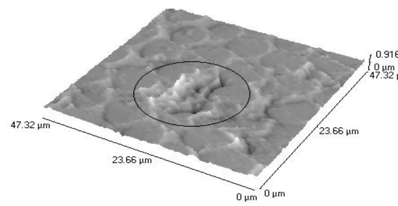
In Fig 4: shows the adhesion level at the interface region. The sharp decrease of the graph at the 1st peak shows the presence of weak interface at one side of the fiber whereas, there is increase of the 2nd peak point gives strong interface region. The failure may initiate from a weak or defective fiber/matrix interface and consequently reduce ultimate performance. The anisotropic and heterogeneous character of composites naturally results in a large possibility of failure modes (7). It was found that the improved interfacial adhesion was strongly related to the hydroxyl, ether, or aromatic groups on the fiber surface, while surface structure had little influence on the interfacial adhesion, interlaminar shear strength and the failure behavior.



(a)



(b)



(c)

Fig 4: AFM topography image of treated GFRP adhesion failure after thermal conditioning treatment.

The delamination failure mode is known to be major life-limiting failure process in a composite laminate. Delamination can induce stiffness loss, local stress concentration and local stability that can cause buckling failure. There is some deviation also occur on the fiber surface. The frail in adhesion strength relative to fiber is attributed to weak covalent bonding between silane coating of the fiber and the polar group of the matrix (8).

It is more reasonable to consider that true contact area, in nanometer/atomic scale, played a dominant role in an efficient molecular interactions which is directly associated with interface adhesion and in true interphase fracture behavior.

4. Conclusion

The study presented here has identified both surface and interphase properties in terms of topography, fractography and adhesion which are mostly characterized by AFM technique. Failure of fiber fracture due to extensive strain can be visualized by height image mode analysis. The increase in degree of cross-linking density by thermal conditioning resulted in reduction of the free volume of matrix. The resulted shrinkage of matrix because of greater cross-linking and loss of volatile matters has been indexed by the fall of height scan images of AFM. The increase in cusp thickness implies fewer microcracks have formed because of increased brittleness. The results reveal a unique view to the fractography study of GFRP composites. Potential work along this line will be able to achieve more information regarding failure and fracture of interface/interphase of polymer composite.

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