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# Scanning Electron Microscopy Study of Fiber Reinforced Polymeric Nanocomposites

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## 1. Introduction

The nanoscale materials offer the opportunity to explore new behavior beyond those established in conventional materials. It has been established that mechanical and thermal properties, moisture barrier, and flame resistance of polymeric composites can be improved by adding a small amount of nanoparticles as filler particles without compromising the density, toughness, storage life, weight or processability of the composite [Sandler et al., 2002; Bruzaud & Bourmaud, 2007]. The higher surface area is one of the most promising characteristics of nanoparticles due to their ability in creating a good interface in a composite. The dispersion of nanoparticles in the matrix is one of the most important parameters in fabricating nanophased composites. It depends on processing techniques such as solution blending, shear mixing, in-situ polymerization, ultrasonic cavitation, and high pressure mixing [Giannelis, 1998; Yasmin et al., 2003; Vaia et al., 1996]. Nanomaterials have enhanced various characteristics in a given polymer. However, these enhancements have limitations at higher loadings due to increased agglomeration causing premature failure.

During service life, composite structures might encounter high stresses resulting in crack propagation through fiber matrix interfaces. Therefore, stronger adhesion between fiber and matrix, higher strength, and higher toughened matrix are desired. Improvement of flexural strength by addition of nanofillers into a matrix is expected to be observed for several reasons. Young's modulus of the second phase dispersed particles is higher than that of the matrix and thus stress transfer from the matrix to the particles will take place. As a result, the strength of the composites is increased. Strong interfacial bonding between the fiber and matrix also contributes to higher flexural strength. Dispersed filler particles act as a mechanical interlocking between the fiber and matrix which creates a high friction coefficient. Finally, a mixed mode of fracture (flexural and shear) occurs under bending-load conditions. After an initial failure of fibers at the tensile side of the specimen, cracks are deflected parallel to the fibers and also to the applied load direction. The stress-strain curve shows a sharp increment with increasing load before reaching the maximum stress and then irregularities and staggered decrease in stress were observed for both conventional and nanophased composites [M.K. Hossain et al., 2011]. However, the initial load and the crack arrest area are higher in nanophased composites which lead to high energy absorbing mechanisms [Hussain et al., 1996].

The morphological study of the nanoparticle dispersion in the CNF-loaded polyester nanocomposites using various processing techniques, including mechanical mixing, magnetic stirring, and sonication is evaluated through SEM studies in this article. Nanoparticles facilitate proper wetting out fibers with resin, void reduction, enhanced crosslinking, and increased friction co-efficient. Sometimes it also works as a nucleating agent in a fiber reinforced polymeric composite. Morphology of the glass/polyester-CNF composites manufactured by the VARTM process has also been studied using the SEM for various applications, including civil infrastructure, automotive, aerospace, sporting, and marine industries. Quantitative enhancement in properties is characterized through the flexural test in this article. Qualitative and visual analyses are important to support the resultant experimental quantitative data and fracture morphology evaluation of different tested specimens using SEM is inevitable in this matter. Therefore, the fracture morphology of all types of tested specimens has been evaluated in this article.

## **2. Research methodology**

### **2.1 Neat and CNF-loaded polyester sample fabrication**

B-440 premium polyester resin, styrene, and heat treated PR-24 CNF were used as matrix, thinner, and nanoparticle, respectively. Polyester resin contains two parts: part-A (polyester resin) and part-B (MEKP- methyl ethyl ketone peroxide) as crosslinking agent. Sonication was performed in a glass beaker using a high intensity ultrasonic irradiation (Ti-horn, 20 kHz Sonics Vibra Cell, Sonics Mandmaterials, Inc, USA) for 60, 90, and 120 minutes, respectively, adding 0.1-0.4 wt.% CNFs to polyester resin while adding 10 wt.% styrene. The mixing process was carried out in a pulse mode of 30 sec. on and 15 sec. off at an amplitude of 50%. To study other types of mixing methods, CNFs were mixed using mechanical mixing and magnetic stirring methods. The mechanical mixer was run for 90 minutes at 300 rpm at room temperature. The magnetic stirring was carried out for 5 hours at 500 rpm at room temperature. To lessen the void formation, vacuum was applied using Brand Tech Vacuum system for about 90-120 minutes and 0.7 wt.% catalyst was then added to the mixer using a high-speed mechanical stirrer for about 2-3 minutes and vacuum was again applied for about 6-8 minutes to degasify the bubbles produced during the catalyst mixing. The as-prepared resin was poured into the mold and kept at room temperature for 12-15 hours. Controlled polyester samples were fabricated to compare with the nanophased samples. All samples were kept in a mechanical convection oven at 110 °C for 3 hours for post curing [M.E. Hossain et al., 2011].

### **2.2 Conventional and nanophased fiber reinforced composite sample fabrication**

Both conventional and nanophased E-glass/polyester-CNF composites were manufactured by the VARTM process. Vacuum was maintained until the end of cure to remove any volatiles generated during the polymerization process. The panels were cured for about 12-15 hours at room temperature and then thermally post cured at 110 °C for 3 hours in a mechanical convection oven. The fiber volume fraction for the nanophased glass reinforced polyester composites fabricated by VARTM was found to be around 56%. The void content (3-4%) was also within a reasonable limit in these composites [M.K. Hossain et al., 2011]. The overall sample fabrication procedure is presented in Figure 1.

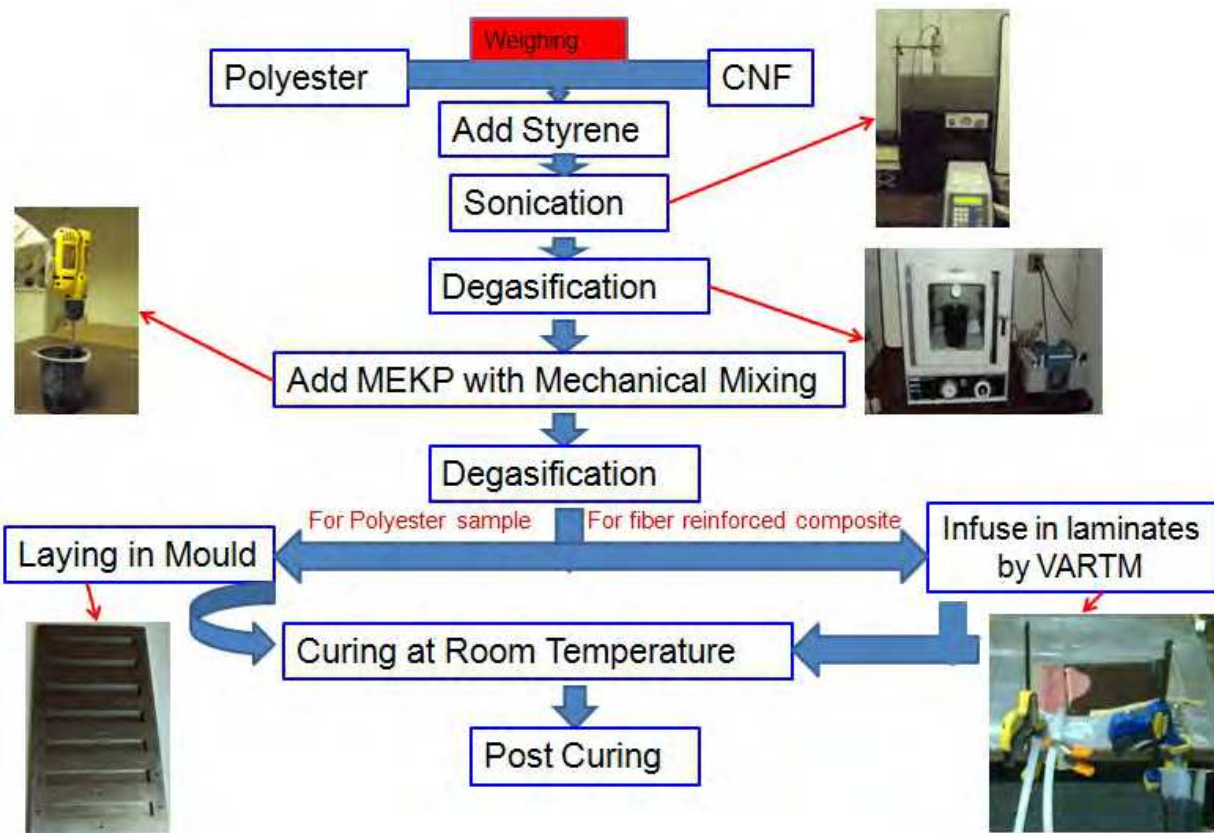


Fig. 1. Flow chart of sample fabrication

### 2.3 Flexural test

Flexural tests under three-point bend configuration were performed using a Zwick Roell testing unit according to the ASTM D790-02 standard to evaluate flexural modulus and strength of each of the material systems of the polymer nanocomposites and its laminates [M.E. Hossain et al., 2011; M.K. Hossain et al., 2011]. The machines were run under displacement control mode at a crosshead speed of 2.0 mm/min and tests were performed at room temperature. The span to depth ratio was maintained at 16:1. The maximum stress at failure on the tension side of a flexural specimen was considered as the flexural strength of the material. Flexural modulus was calculated from the slope of the stress-strain plot. Five samples of each type were tested. The average values and standard deviation of flexural strength and modulus were determined.

### 2.4 Scanning Electron Microscopy (SEM)

SEM studies were carried out to examine change in the microstructure due to the addition of CNFs using a JEOL JSM 5800 microscope. SEM also facilitates to monitor the failure approach at micro level. Failed samples from the three point flexure test were examined to distinguish the changes in the failure mode. The samples were cut through the cross-section of the failed region. The samples were positioned on a sample holder with a silver paint and coated with gold to prevent charge build-up by the electron absorbed by the specimen. A 15 kilovolt accelerating voltage was applied to achieve desired magnification [M.E. Hossain et al., 2011; M.K. Hossain et al., 2011].

## 2.5 SEM sample preparation

SEM samples must have an appropriate size to fit in the specimen chamber and is generally mounted rigidly on a specimen holder. Specimens must be electrically conductive, specially the surface, for imaging, and electrically grounded to prevent the accumulation of electrostatic charge at the surface of the specimen during electron irradiation [Suzuki, 2002]. Metal objects require little special preparation for SEM except for cleaning and mounting on the specimen holder. Nonconductive specimens tend to charge when scanned by the electron beam, especially in the secondary electron imaging mode. This causes scanning faults and other image artifacts. They are therefore usually coated with an ultrathin electrically-conducting material, commonly gold, deposited on the sample either using a low vacuum sputtering machine or a high vacuum evaporation unit.

## 3. Results and discussion

### 3.1 Flexural test results

Typical stress-strain curves of neat and nanophased polyester samples as well as their fiber reinforced laminates generated from flexural tests are illustrated in Figures 2 and 3. Flexural strength, modulus, and the strain at maximum strength for all CNF-loaded samples were larger than those of the neat samples. CNF has high aspect ratio which can prevent crack generation and propagation in the polyester matrix (Figure 2). In all cases, the samples failed rapidly after experiencing the maximum load showing induced brittle nature of failure due to the addition of CNFs. The 0.2 wt.% CNFs loading and 90 minutes sonication time were observed to be the optimal condition for this nanocomposite system. The 0.2 wt.% CNF-loaded samples enhanced the flexural strength and modulus by about 88% and 16%, respectively, compared to the neat ones. The failure strain also increased significantly with the addition of CNFs into the system. Flexural properties were slightly decreased at higher CNF content. It might be due to the creation of micro aggregates of CNFs in various regions of the polymer matrix, which act as areas of weakness [M.E. Hossain et al., 2011].

Typical stress-strain curves of conventional and nanophased glass/polyester composites presented in Figure 3 demonstrated significant improvement in the mechanical properties up to the 0.2 wt.% of CNFs loading, beyond which there was a decreasing trend. These curves showed considerable nonlinear deformation before reaching the maximum stress. This was attributed to the random fiber breakage during loading. However, more or less ductility was observed in each type of laminate sample and cracking noise was heard while the individual fiber broke or the inter-layer delaminated. No obvious yield point was found. From the resultant data, it was concluded that the 0.2 wt.% CNF was the optimum amount for this material system to achieve the maximum flexural modulus and strength. These specimens showed approximately 49% and 31% increase in the flexural strength and modulus, respectively. There are several reasons for the better mechanical properties observed in the CNF-infused glass fabric reinforced polyester laminates. First, CNFs increase the strength and modulus of the polyester matrix, which was observed in the CNF-loaded polyester in this study. Second, the presence of CNFs increases the crack propagation resistance and prevents crack generation by bridging effect at the interface region of the long glass fiber, CNF, and polyester matrix. Moreover, CNF has high aspect ratio, which improves the strength and modulus [M.K. Hossain et al., 2011].



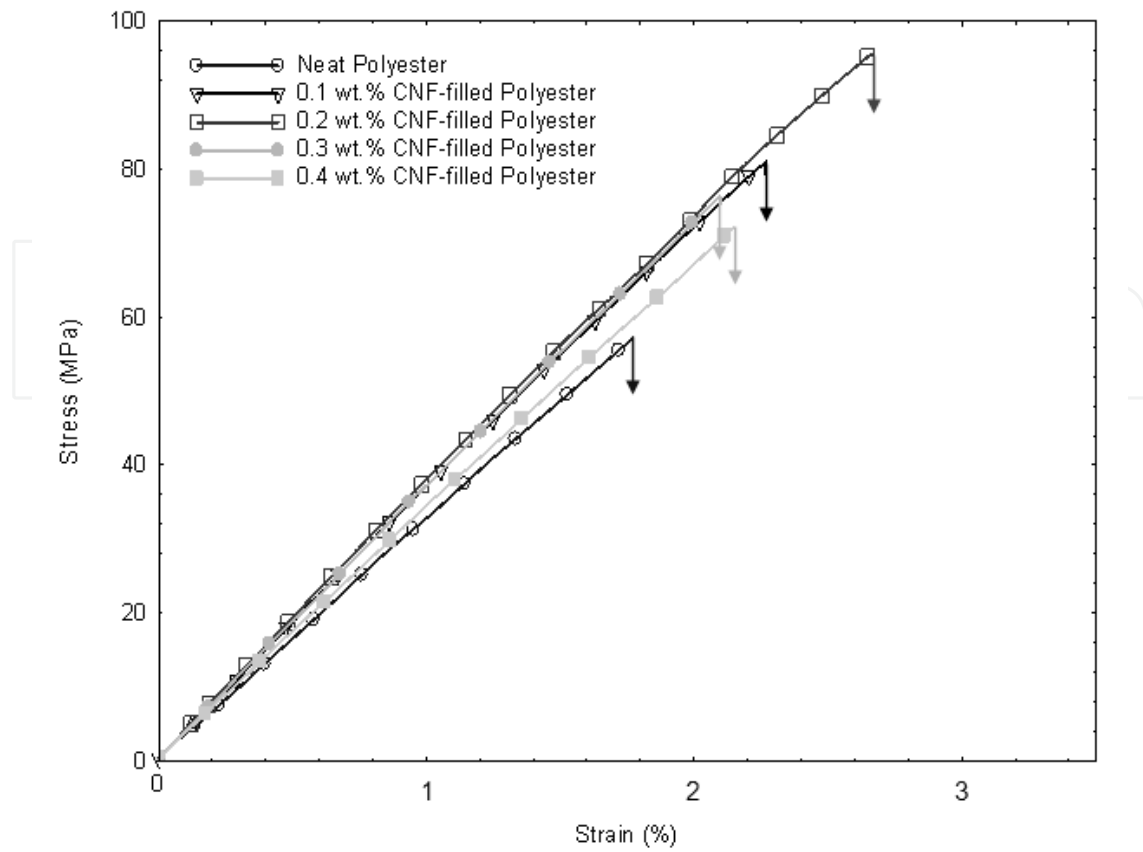


Fig. 2. Flexural stress-strain plot of polyester samples with different wt.% of CNFs.

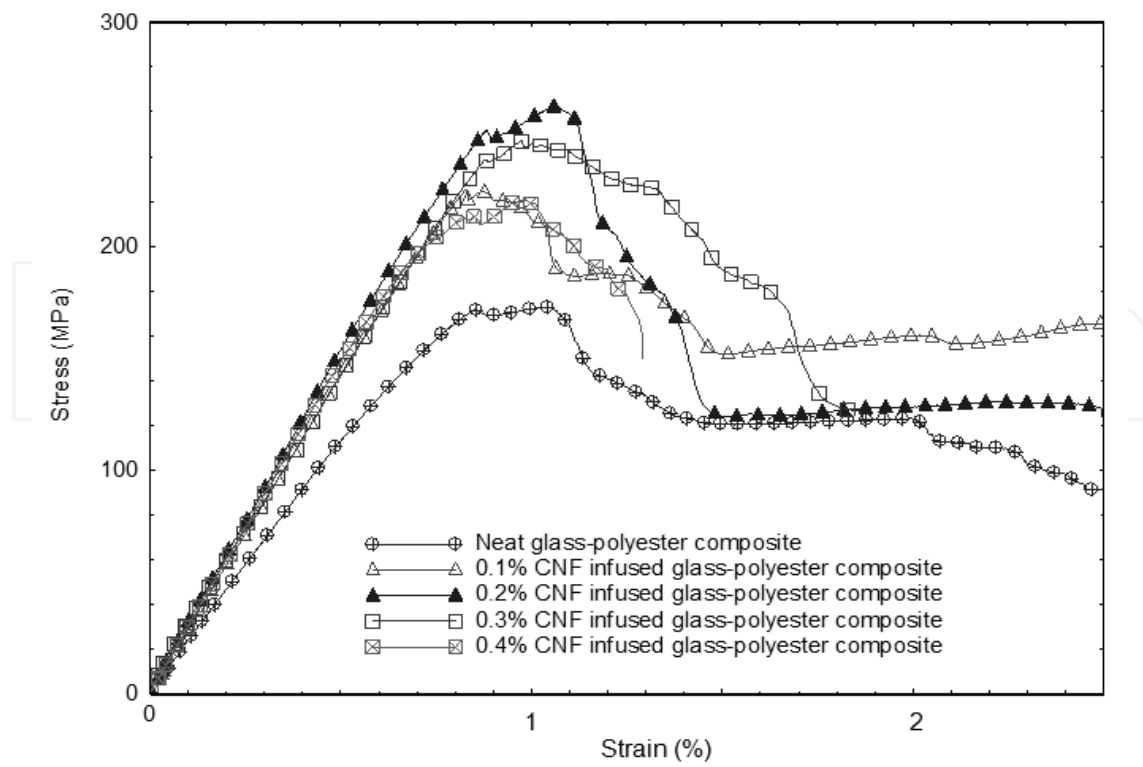


Fig. 3. Flexural stress- strain plot of GRPC laminates with different wt.% of CNF.

### 3.2 Microstructure and morphological analyses

The SEM micrographs of as-received PR-24 CNF and the neat polyester matrix are shown in Figures 4 (a) and 4 (b), respectively. To investigate the dispersion properties of CNFs in polyester, drops of concentrated  $\text{HNO}_3$  acid were added on the cleavage surfaces to partly unveil the CNFs formerly covered by the polyester. From the micrograph of 0.2 wt.% CNF-filled polyester, excellent dispersion of CNFs was found (Figure 5). Only broken ends of CNFs were observed near the surface. Some CNFs broke in a brittle manner and some were pulled out.

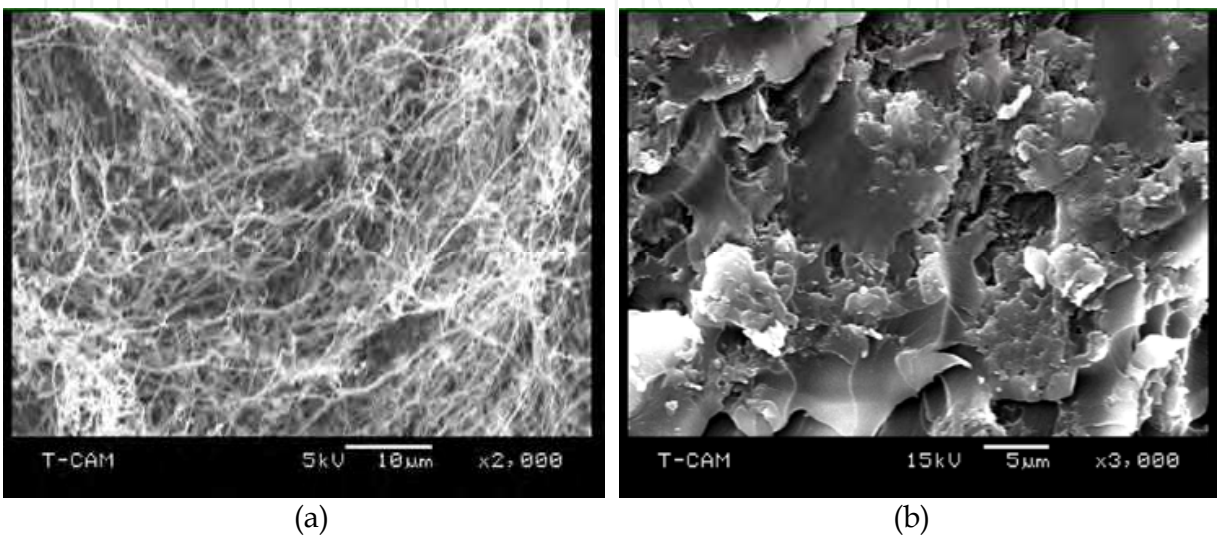


Fig. 4. SEM micrographs of (a) as-received PR-24 CNF and (b) neat polyester matrix.

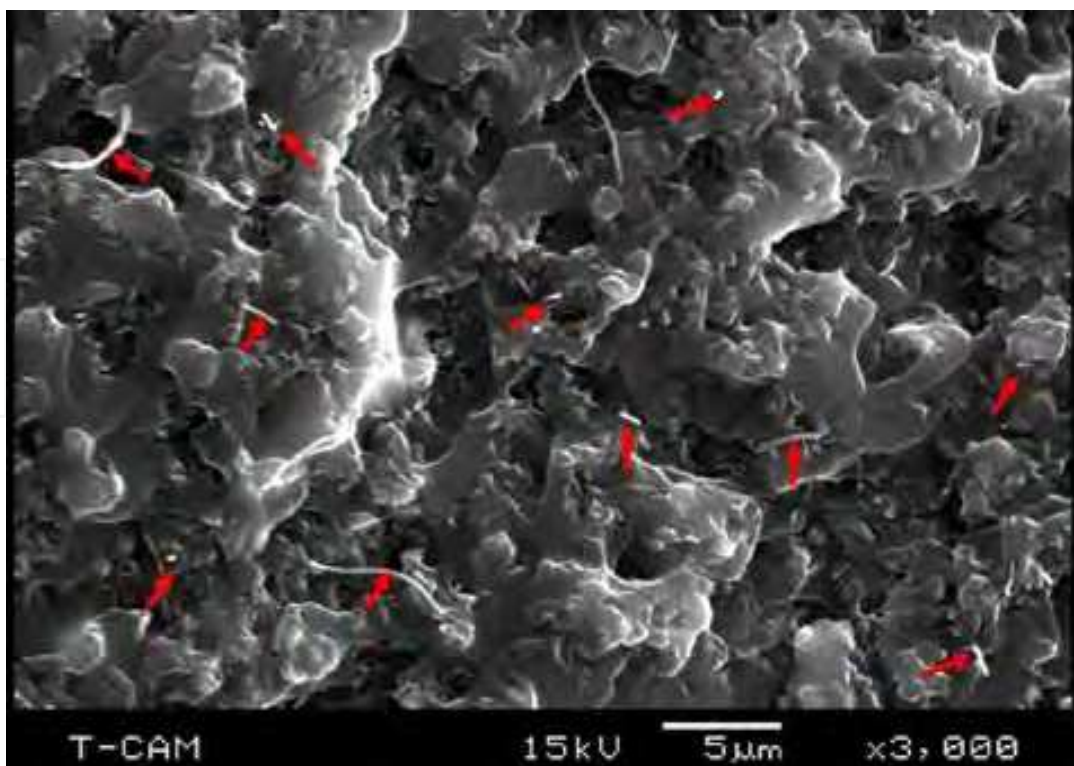


Fig. 5. SEM micrograph of acid-etched 0.2 wt.% CNF-loaded polyester at 3000X.

Agglomerations in the polyester matrix were observed from the micrographs (Figure 6) of 0.2 wt.% infused polyester samples prepared through mechanical mixing and magnetic stirring methods, respectively. These agglomerates area create stress concentration zones which might act as a crack initiator.

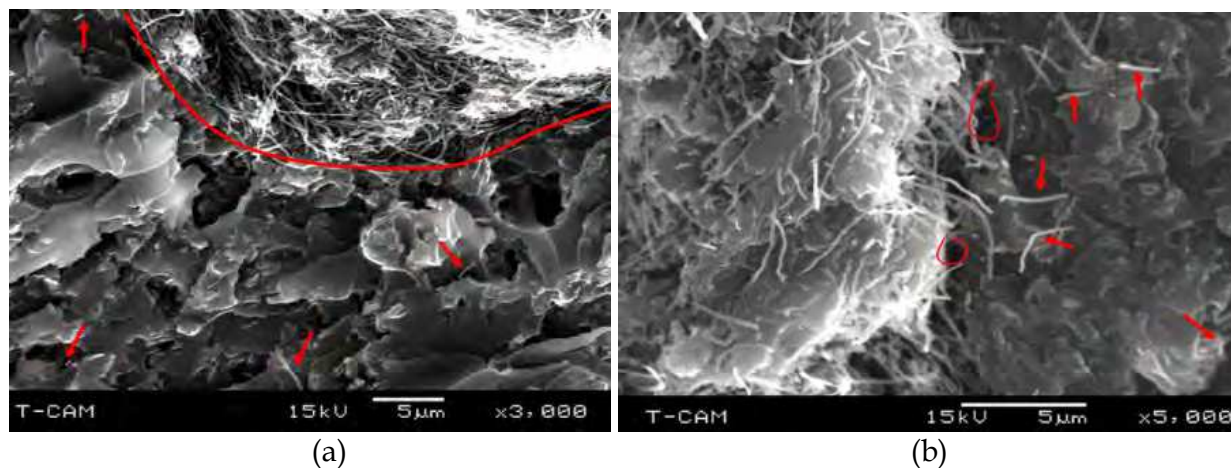


Fig. 6. Micrographs of acid-etched 0.2 wt.% CNF-loaded polyester: (a) Mechanical stirring and (b) Magnetic stirring

Strong attractive fiber van der Waals forces cause CNFs to agglomerate, which reduces the strength of the nanocomposite by stress concentration effect. Agglomerates of CNFs, called nanoropes, are difficult to separate and infiltrate with matrix. They entangle and form nest-like structures due to their curvature and high aspect ratios. Both disagglomeration and dispersion in resins depend on the relative van der Waals forces, curvature, and on the relative surface energy of CNFs versus that of the resin. To overcome attractive forces, researchers have been extensively using mechanical energy, intense ultrasonication, and high speed shearing. Some rebundling of the aggregates is possible even after discontinuation of the external force [Yoonessi et al., 2008]. However, optimal loading and uniform dispersion of CNFs in matrix are the key parameters to promote better nanofiber-matrix interface properties to reach an efficient load transfer between two constituents of the nanocomposite [Kozey et al., 1995; Ma et al., 2003].

Uniform dispersion of 0.2 wt.% CNFs into the polyester resin was achieved using the sonication mixing method for 90 minutes. High magnification SEM micrograph in Figure 7 clearly exhibits that CNFs are well separated and uniformly embedded in the 0.2% polyester resin system. It can also be easily observed that the interfacial bonding between the CNF and matrix was very compact which would allow CNFs to be anchored in the embedding matrix. In essence, these CNFs are likely to interlock and entangle with the polymer chains in the matrix [Li et al., 2008]. Thus, addition of CNFs enhanced the crosslinking between polymer chains and provided better interfacial bonding.

Figures 8 (a) and 8 (b) show the woven glass reinforced polyester laminates with 0.2 wt.% CNFs. It was found that the resin was distributed uniformly over the fabric, and the interfacial bonding between matrix and fiber was very good. Resin flow and impregnation of the glass fibers can be observed in the SEM micrographs. Clear resin matrix adhesion is present in these micrographs, and glass fibers are observed to be embedded within the



matrix. Good matrix-fiber wetting was achieved and resin is also visible in between the glass fiber filaments. It appears that better interfacial bonding between the nanophased polymer matrix and glass fiber is present due to the presence of CNFs [Green et al., 2009]. The fiber volume fraction as determined from matrix digestion method for the nanophased glass reinforced polyester composites fabricated by the VARTM process was found to be around 56%.

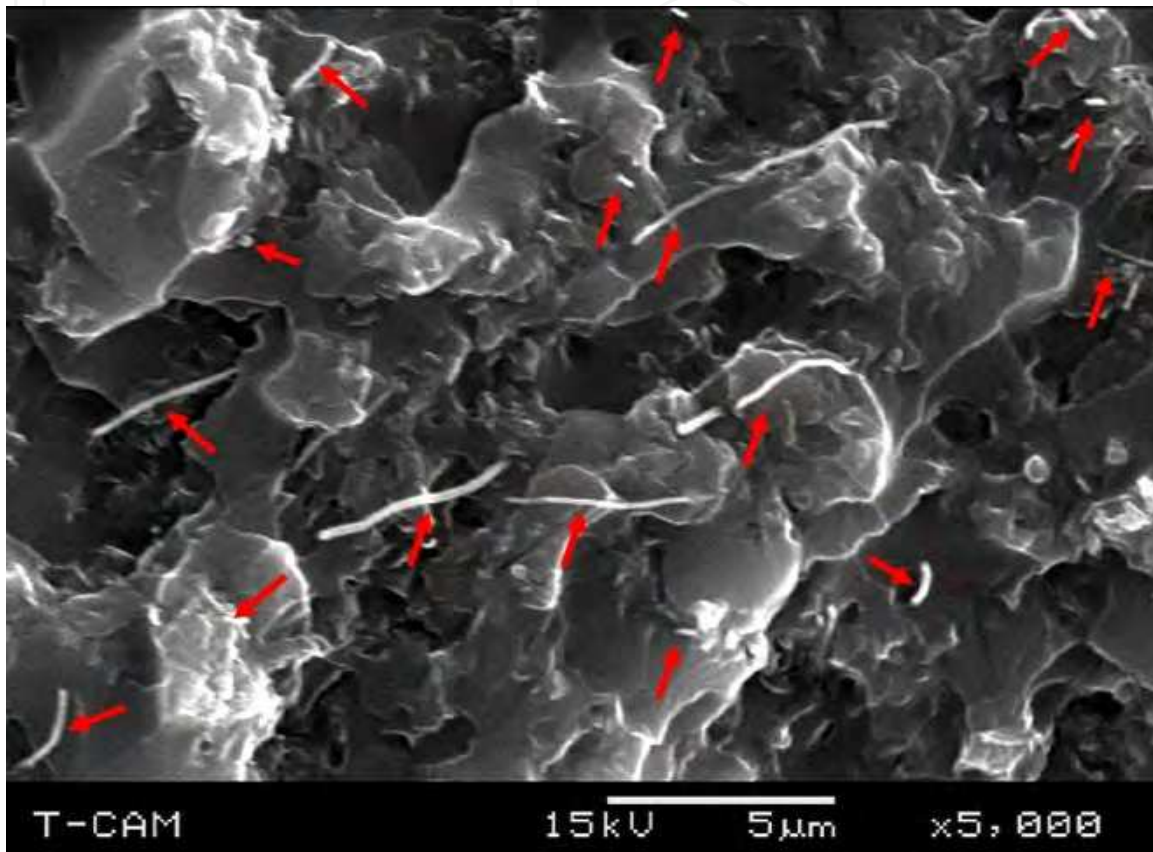
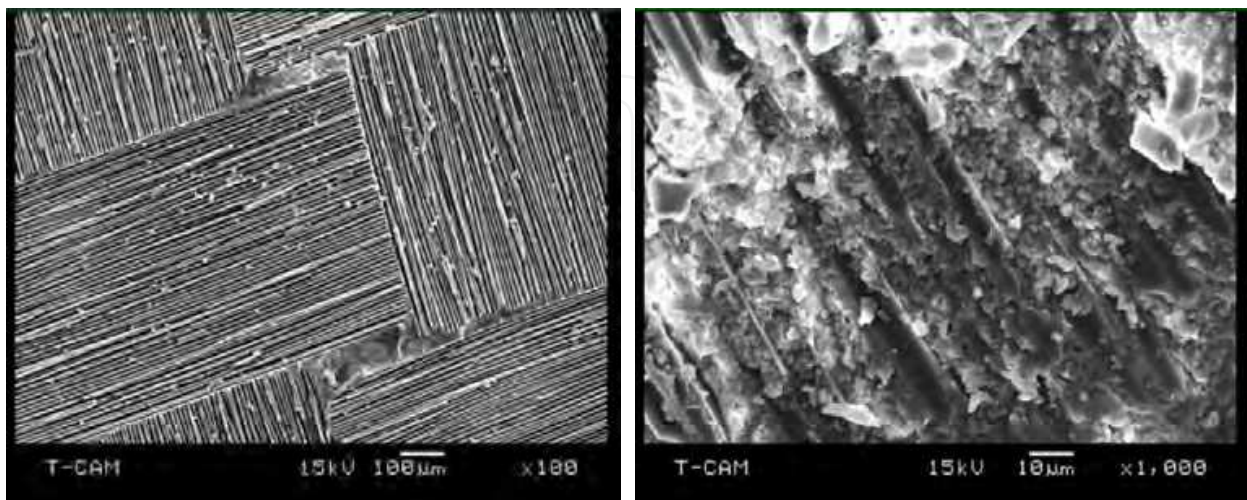


Fig. 7. SEM micrograph of acid-etched 0.2 wt.% CNF-loaded polyester at 5000X



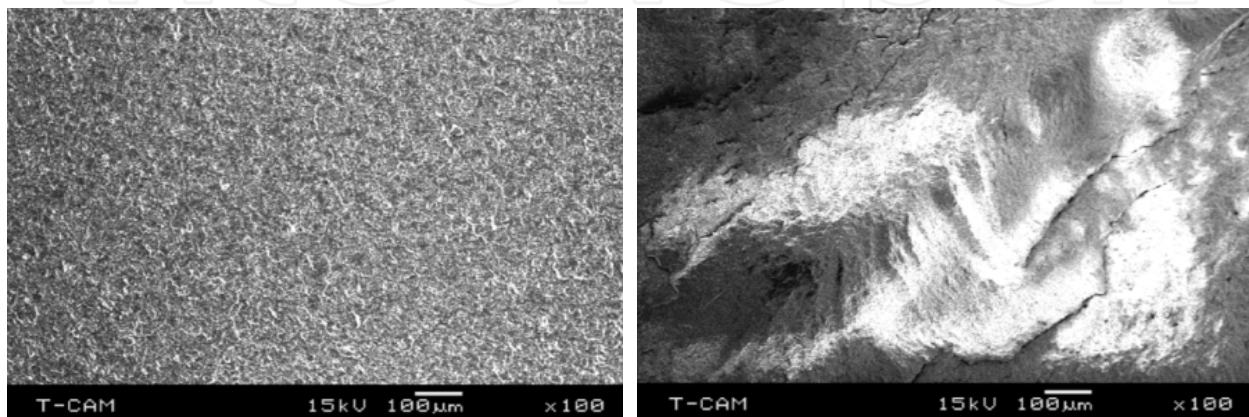
(a)

(b)

Fig. 8. 0.2 wt.% CNF-loaded GRPC laminates (a and b).

### 3.3 Fracture morphology analysis

Results from the SEM study substantiate the quantitative results obtained through flexural test [M.E. Hossain et al., 2011; M.K. Hossain et al., 2011]. SEM performed on the fractured samples of flexure tests revealed rough and smooth fracture surfaces in 0.2 CNF-loaded sample and neat sample, respectively (Figure 9). The bonding between polyester and CNF was seen to be strong and attributed to cause deviation in the path of crack front as it propagated, thus requiring more energy to fracture the samples. This has resulted in increased strength and stiffness of samples. The effect was most pronounced at 0.2 wt.% loading of CNFs.

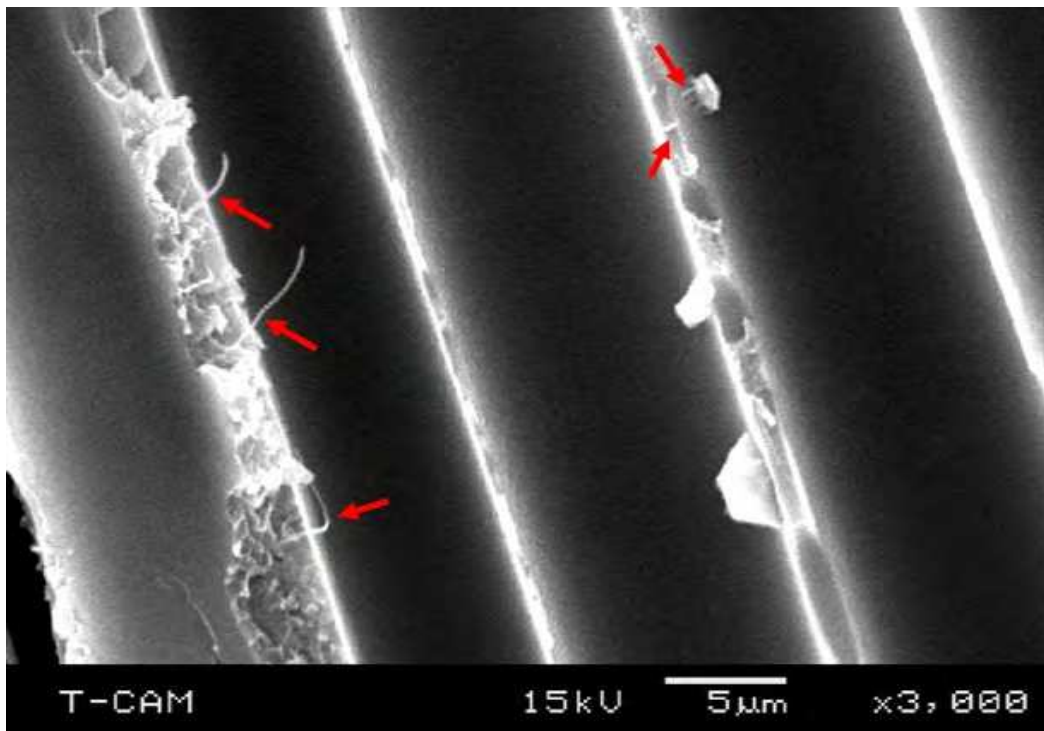


(a) Controlled polyester samples

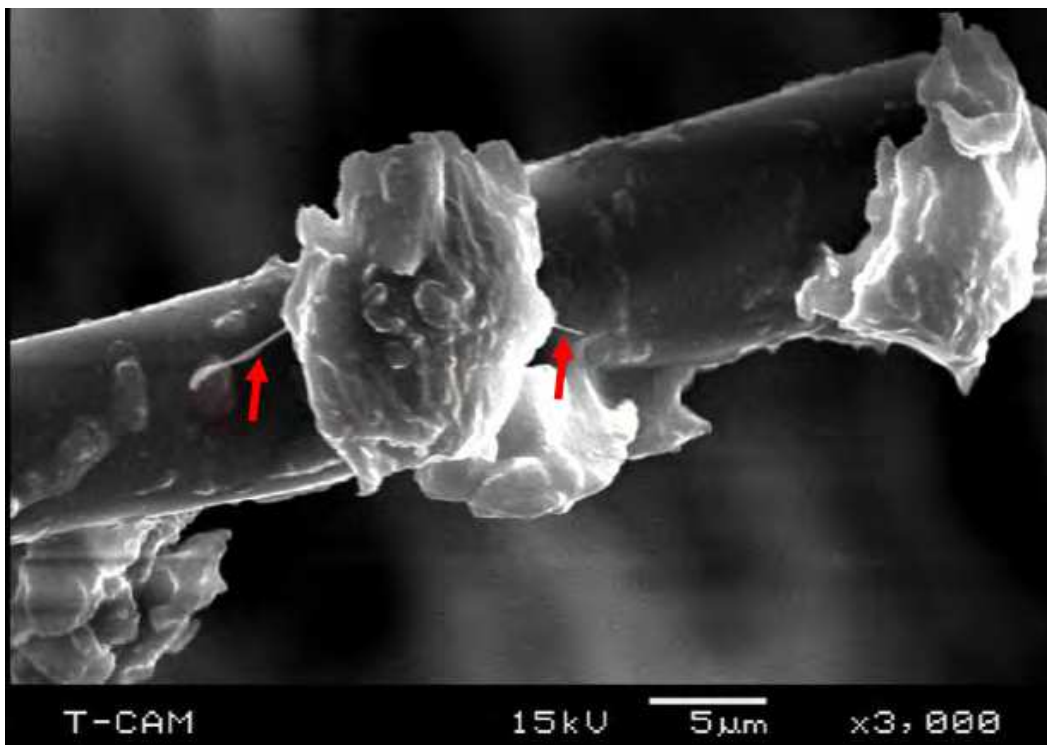
(b) 0.2 CNF loaded polyester samples

Fig. 9. SEM micrographs of fracture surface (a & b) after flexural test

From the SEM micrograph taken at higher magnification as shown in Figure 10 (a), excellent bridging effect in the interfacial region of the long glass fiber, CNF, and matrix was observed. CNF has high aspect ratio which can prevent crack propagation and crack generation resulting in improved performance. Some resin was stacked on the fractured glass fiber as shown in Figure 10 (b), which represents better adhesion due to the addition of CNFs. The presence of polyester adhering to the fiber surface also suggests that interfacial adhesion is stronger than matrix strength in nanophased composites [Hussain et al., 1996]. Thus, it is evident from these micrographs that CNFs are anchored with both resin and fiber tightly that promotes a better interfacial bonding between the matrix and fiber. Better fiber-matrix interfacial bonding, and CNFs' crack generation and propagation resistance result in higher strength in nanocomposites. On the other hand, the addition of the CNFs led to an improvement in the modulus of elasticity of the nanophased composites. This is attributed to the stiffening of the matrix of these composites (Figure 2). The interfacial area between the resin matrix and CNFs was increased because of the high aspect ratio of the CNFs, which in turn led to better mechanical properties [Tsantzalidis et al., 2007]. The nanoparticles also act as reinforcing element and bear the load in the composite material system [Jawahar et al., 2006]. Again, both CNFs and fibers are stronger than matrix. Thus, when load is applied to the composite structures, matrix starts to crack first and stress is then transferred from the lower modulus matrix to the CNFs to the long fiber by bridging effect and ultimately, the composites' properties enhance.



(a)



(b)

Fig. 10. (a) Bridging effect at the interface region of the long glass fiber, CNF and the resin and (b) 0.2 wt.% CNF-loaded polyester matrix stacked with glass fabric after fractured laminate.



For a better understanding of the fracture process, fracture morphology of samples was studied using higher magnification SEM micrographs. The SEM micrographs of the fractured surfaces of the conventional and 0.2 wt.% CNF-loaded GRPC are illustrated in Figure 11. For conventional composite shown in Figure 11 (a), the surface of the fiber was clean, and no matrix adhered to the fiber. The fracture surface of the matrix was flat, and some cracks were seen in the matrix side near the fiber-matrix interface. Resin appears not to protrude from the surface of fibers. These results indicate that the interfacial bonding between the fiber and matrix was weak. The fracture surface of the nanophased composite (Figure 11 (b)) shows that the surface of the matrix was rougher than that of neat composite. CNFs were observed to be randomly but uniformly distributed in the matrix. The resin appears to cling to fibers well. The strengthened matrix held the glass fabrics together. The protrusion of the resin from the surface of the fibers accounts for the increase in fracture toughness of the samples. Moreover, the resin appears to be sticking to the fiber surface giving rise to a significant plastic deformation [Xu & Hoa, 2008]. The plastic deformation enhances mechanical properties significantly in the nanophased composites (Figure 3) [M.K. Hossain et al., 2011].

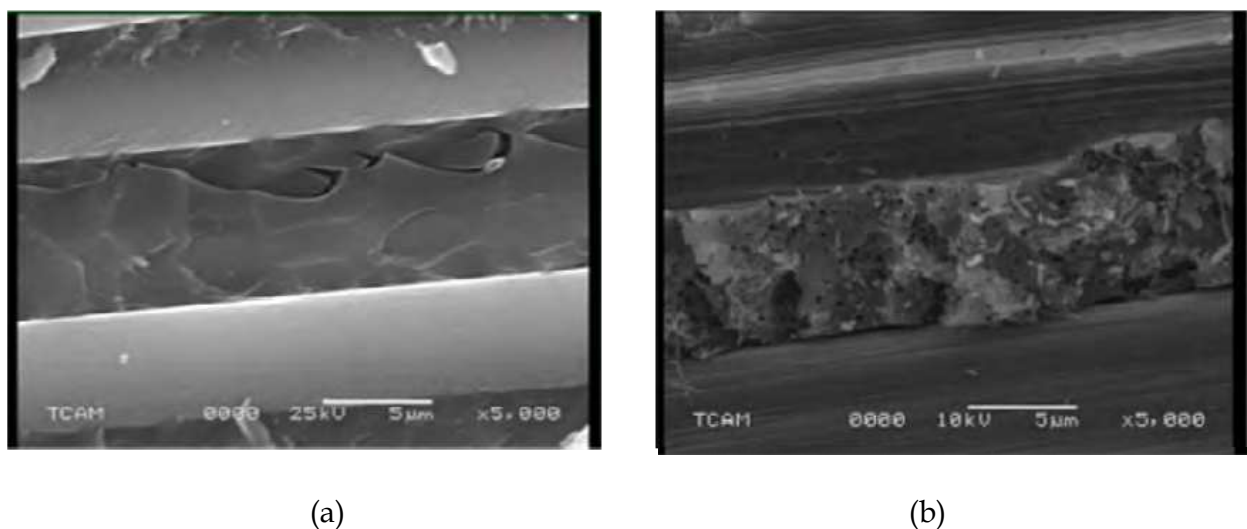


Fig. 11. Fracture morphology of (a) conventional, and (b) 0.2 wt.% CNF-loaded GRPC.

#### 4. Conclusion

Sonication, mechanical mixing, and magnetic stirring were performed to infuse 0.1-0.4 wt.% carbon nanofibers (CNFs) into the polyester resin. CNFs were used as nanoparticle fillers in woven glass fiber-reinforced polyester composites. Better dispersion of CNFs was observed in the 0.2 wt.% CNF-loaded polyester resin while CNFs were mixed using the sonication. The fiber volume fraction for the nanophased GRPC fabricated by the VARTM process was found around 56%. The void content was also within a reasonable limit in these composites. CNFs infusion even at quite low concentrations enhanced the mechanical properties of the system. This SEM investigation visually demonstrated that CNFs can be used without difficulty to modify the conventional fiber reinforced composite materials. Thus, SEM micrographs confirm that uniform dispersion and optimal loading of nanoparticles improve the mechanical properties of composites with the following outcomes:



- SEM micrographs revealed better dispersion of CNFs in the 0.2 wt.% CNF-loaded polyester prepared by sonication mixing and agglomeration in samples prepared by mechanical and magnetic mixing methods.
- Uniform resin flow and proper impregnation of the glass fibers were observed in the SEM micrographs due to the presence of CNFs.
- Uniform resin flow and proper impregnation of the glass fibers appeared to result in a better interaction between the fiber and matrix that aided to an efficient stress transfer from the continuous polymer matrix to the dispersed fiber reinforcement through the mechanical interlocking of the CNFs with the fibers.
- Excellent bridging effect in the interfacial region of the long glass fiber, CNF, and matrix was observed in SEM micrographs of nanophased GRPC.
- SEM micrographs exhibited rougher fracture surface in the CNF-loaded polyester sample compared to the neat sample due to the presence of well dispersed and well separated CNFs.
- SEM micrographs revealed the flat and clean fracture surface of the matrix with some cracks in the matrix side of the fiber-matrix interface in the conventional GRPC whereas nanophased GRPC showed rougher fracture of the matrix with randomly but uniformly distributed CNFs throughout the matrix that appeared to cling to fibers well.

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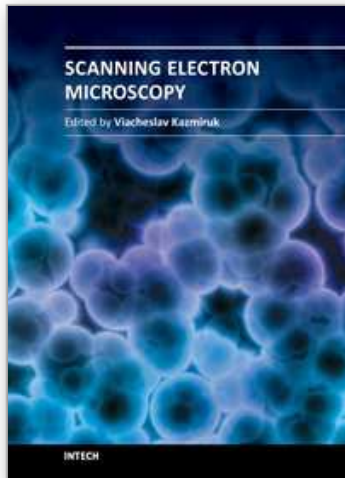
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Today, an individual would be hard-pressed to find any science field that does not employ methods and instruments based on the use of fine focused electron and ion beams. Well instrumented and supplemented with advanced methods and techniques, SEMs provide possibilities not only of surface imaging but quantitative measurement of object topologies, local electrophysical characteristics of semiconductor structures and performing elemental analysis. Moreover, a fine focused e-beam is widely used for the creation of micro and nanostructures. The book's approach covers both theoretical and practical issues related to scanning electron microscopy. The book has 41 chapters, divided into six sections: Instrumentation, Methodology, Biology, Medicine, Material Science, Nanostructured Materials for Electronic Industry, Thin Films, Membranes, Ceramic, Geoscience, and Mineralogy. Each chapter, written by different authors, is a complete work which presupposes that readers have some background knowledge on the subject.

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