Micrographs of the Fracture of Vinyl Ester Composites Cured by Microwaves: Pilot Study

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Abstract: Short bar method of fracture toughness measurement [1-3] was used to perform tests to find out the difference in fracture toughness between microwave cured vinyl ester particulate composites and those cured under ambient conditions. The results show that the difference in the fracture toughness is minimal [4]. This paper presents the analysis of the fracture of the samples using a Scanning Electron Microscope (SEM). The fracture toughness of vinyl ester composites varies slightly after undergoing microwave treatment; it varies with different microwave power level and duration of exposure. The microscopic results show some important features such brittle behaviour, elongation of the fracture surface. The sample with lower fracture toughness is found to have more bubbles in the composite than those samples with higher fracture toughness. Therefore, the microwave power level and exposure time appear to correlate with the fracture toughness values obtained.

1. Introduction

There is a growing demand for composite materials in many industries due to their superior mechanical properties. These industries include the electronics, aerospace and bioengineering sectors. To meet the industry needs, the University of Southern Queensland had established the “Fibre Composite Design and Development Centre of Excellence” to facilitate research and development in this area. One of the focuses of the centre is vinyl ester particulate reinforced composite, which is an important material for building and bridge structures.

The main problem of vinyl ester resins is its shrinkage during solidification or curing. Shrinkage can be up to twelve percent, which is much higher than claimed by some researchers [5-6]. The main drawbacks of this shrinkage in a composite component are the difficulty in controlling dimensional accuracy and stresses set up internally. These stresses are usually tensile in the core of the component and compressive on the surface [7]. When these stresses act together with the applied service loads they may cause premature failure of the composite component. At the moment, the Centre solves the shrinkage problem by breaking a large composite component into smaller composite parts because smaller parts tend to have less shrinkage. The hardened smaller parts are then joined together to form the overall structure. This approach did not directly address the shrinkage issue. Instead, it tries to reduce the shrinkage indirectly through changes in design and production. The main disadvantage is that the manufacturing lead-time and costs of a composite component is significantly increased.

To address the shrinkage problem directly, researchers had looked into alternate heating processes that can reduce the curing time. Recently, industrial microwave technology for processing polymers and polymer-based composites is increasingly being applied because of its deep penetration into the material without overheating the surface [8-10]. Lewis et al. studied the morphology and fracture toughness of thermoplastic modified epoxy resin networks prepared via electromagnetic processing [11]. They demonstrated that the rate of network formation for epoxy resins was significantly accelerated with the retention of good mechanical properties by utilizing electromagnetic radiation. They also discovered that the fracture properties of epoxy resins cured in this manner were identical to conventionally cured resins. Hedrick, et

al. accelerated the curing of epoxy resins using microwaves. They discovered that the curing of epoxy resins was significantly accelerated by microwave irradiation [12]. The degree of acceleration in the curing would depend on both power and the sample utilized. The mechanical properties of the microwave-cured resin were similar to those for the thermal cured system. Boey and Yap also discovered that microwave curing is effective in reducing the overall cure time for epoxies [13]. Lin and Hawley cured a composite prepreg made of glass fibre and vinyl ester by microwave in the process of pultrusion [14]. They found that the heating and curing results justified the application of continuos microwave technology to the pultrusion process. Their work is the only research found, mentioning the curing of vinyl ester by microwaves. However, the work did not examine in detailed the factors that can affect the shrinkage. The vinyl ester composite used in this study is thirty three percent (33%) by weight of fly ash particulate reinforced vinyl ester resins [VE/FLYASH (33%)], which is exactly the same type of material used in the previous relevant study [15-18].

2. The Composite Samples

As the raw materials of the composites are liquid and ceramic hollow spheres, the short bar specimens were cast to shape. The resin is a colourless liquid and is first mixed with the colourless accelerator. After that the fly ash is added to the mixture and they are then mixed to give the uncured composite. Table 1 shows the mass in grams of resin, accelerator and fly ash required respectively to make a volume of 1000 millilitres of uncured composite (of 44% by volume of fly ash or of 33 % by weight). The uncured composite was then poured into the moulds for curing in ambient or microwaved conditions (Ku et al., 2003a). The mould was made from

hard cartoon with six pieces of short bar specimen each as depicted in Figure 1. The slots were made by inserting plastic sheets of suitable thickness. Figure 2 shows some of the VE/FLYASH (33%) short bar specimens ready for the tests.

3. Micrographs of Fractured Vinyl Ester Composites

Figure 3 illustrates the six broken samples. The fractured surfaces of the samples were analyzed using a Scanning Electron Microscope (SEM). It was anticipated that by exposing the samples to microwaves, there would be some changes of the microstructure of samples. The changes would affect the material properties like its fracture toughness and so on. It was found that some area of the chevron edge cut showed ductile failure and some displayed brittle cleavage. There were five critical points of the chevron facture to be analyzed as illustrated in Figure 4. The results of fracture toughness and other important parameters for vinyl ester composite cured under different conditions were shown in Table 2. Specimens with the highest and lowest fracture toughness value were chosen for investigating their fracture because quick results were required as the rent of using the SEM was expensive. The specimen chosen were from the 180 watt microwave power with 60 seconds (highest fracture toughness value) and 360 watt microwave power with 80 seconds (lowest fracture toughness value). It is expected that the fracture surface with highest value of fracture toughness, K_{IC} would have lesser flaws than the specimen with lowest value (Chew, 2004). Of course, there is much more information that be surprisingly found from the micrograph such the percentage of fly ash contained in the composites, form of matrix for plastic deformation and so on.

The first specimen chosen was the 180-watt microwave power with an exposure of 60 seconds. The five critical points (Figure 4) were analyzed by magnification of up to 500 times as illustrated in Figures 5 through 9. Figure 5 is the micrograph of area 1; it shows the cleavage fracture at the tip of the chevron cut and there is some electrical discharge in the image (transparent part). Figure 6 illustrates the micrograph of area 2, the stretch-zone. Figure 7 is the micrograph of area 3; it depicts that some matrix is elongated. Figure 8 is the micrograph of area 4; it illustrates some micro voids and some scratches on the fractured surface. Figure 9 is the micrograph of area 5; it shows the matrix failed in brittle manner but there is some dirt in the image.

The second specimen chosen was the 360-watt power level with an 80 exposure of seconds. Similarly, five critical points were analyzed by magnification of up to 500 times as depicted in Figures 10 through 14. Figure 10 is the micrograph of area 1; it shows that matrixes are averagely elongated to initiate crack. Figure 11 is the micrograph of area 2; it depicts that some micro voids exist in the matrix. Figure 12 is the micrograph of area 3; it illustrates that most of the fracture surface has been damaged. Figure 13 is the micrograph of area 4; it shows that the material has failed in brittle manner and some micro voids exist. Figure 14 is the micrograph of area 5; it illustrates that the matrix has failed in brittle manner and electrical discharge exists in the image.

4. Discussion and Conclusion

Though the results obtained from the SEM analysis is not very vigorous, they show some important features such brittle or ductile behaviour and elongation of the

fractured surface of the composite. The sample with lower fracture toughness value is found to have more bubbles in the composite than those with higher toughness value. Therefore, the microwave power level and exposure time appear to correlate with the K_{IC} values obtained.

Figure 6 (area 2, 180 W power and 60 seconds) shows that the sample was stretched; the elongation at peak, the elongation at break, and the fracture toughness of the sample are1.214 mm, 1.557 mm and 52.72 MPa$\sqrt{m}$ respectively. Figure 11 (area 2, 360 W power and 80 seconds) illustrates that micro voids existed in the matrixes; the elongation at peak, the elongation at break, and the fracture toughness of the sample are1.121 mm 1.445 mm and 48.49 MPa$\sqrt{m}$ respectively. From the elongations and fracture toughness values of the two samples, it can found that the micrographs give reasonable explanations to the mechanical behavior of the samples. The stretched sample (Figure 6) had higher elongations and fracture toughness values than its counterparts (Figure 11). Figure 7 (area 3, 180 W power and 60 seconds) also illustrates that the matrixes were elongated and stretched. Figure 12 (area 3, 360 W power and 80 seconds) shows that the fractured surface was damaged. This phenomenon can also be explained by the differences in elongation and fracture toughness values as explained for area 2 (Figures 6 and 11). From the two areas investigated, they show that specimen microwaved at 180 W for 60 seconds is tougher than its counterparts.

The surface of chevron edge cut of specimen is non-conductive and has a corroded surface. Therefore, it must be made conductive by coating with gold because the microscope works by focusing the electron beam on the surface of sample. However, the original dimension of specimen is too big to fit into the gold-coating test rig; the specimen could not therefore be coated evenly. Hence, part of the fractured specimen must be cut away as shown in Figure 15. Magnification should be increased to 10, 000 times or more to obtain more information from the image. A few of the micrographs acquired shows that the surface of the chevron cut has deteriorated by being scratched or compressed by other materials. This may damage the important features of the crack. Therefore, the specimens have to be kept in a solid container should be taken for viewing under SEM as soon as possible after tensile tests.

More information can be obtained if computer software is used to analyze and simulate the experimental results, such as the area of maximum fracture toughness etc. The software package called ANSYS is suggested because it is user-friendly and can be incorporated with other software such as PRO-ENGINEER and so on.

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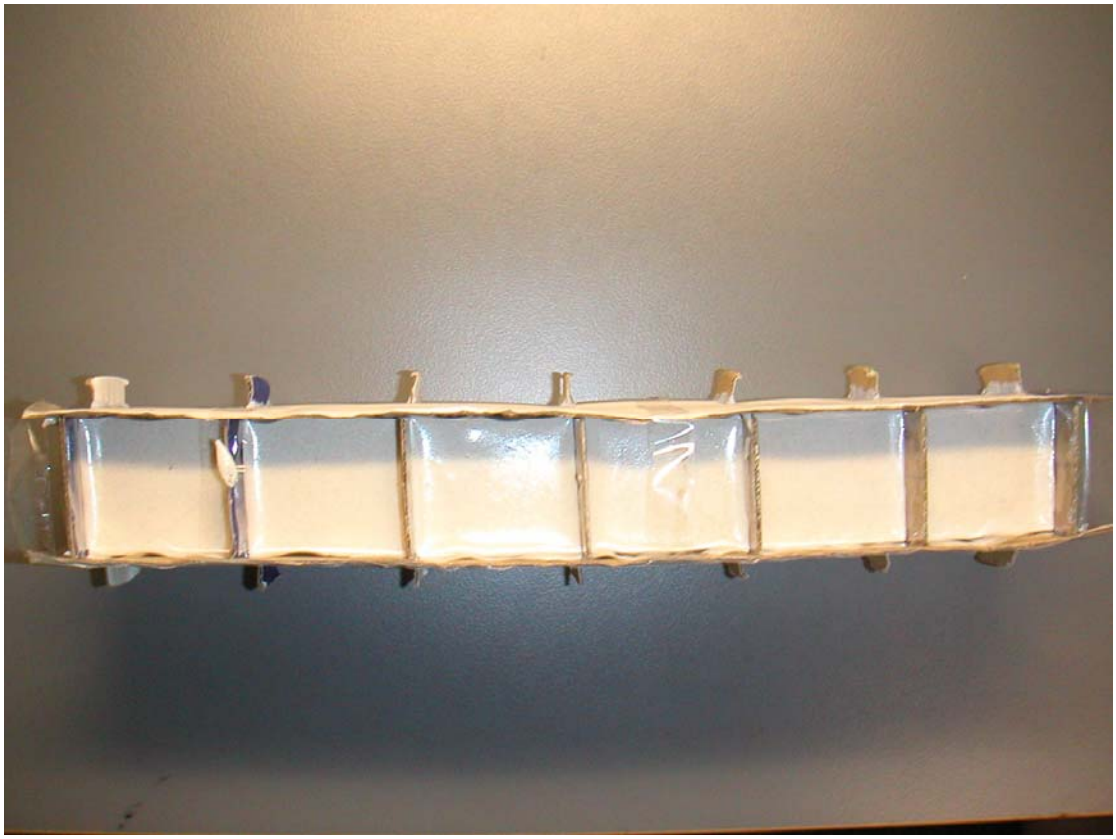

Figure 1: The mould for short bar specimens

Figure 2: The short bar specimens

Figure 3: Six samples of fracture specimens.

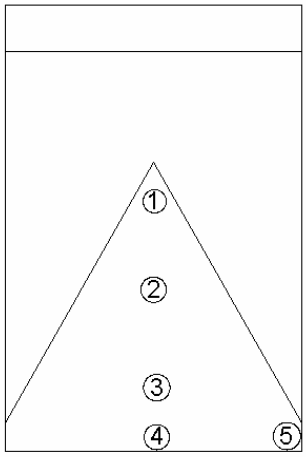

Figure 4: Five critical points for the fractured surface to be analysed.

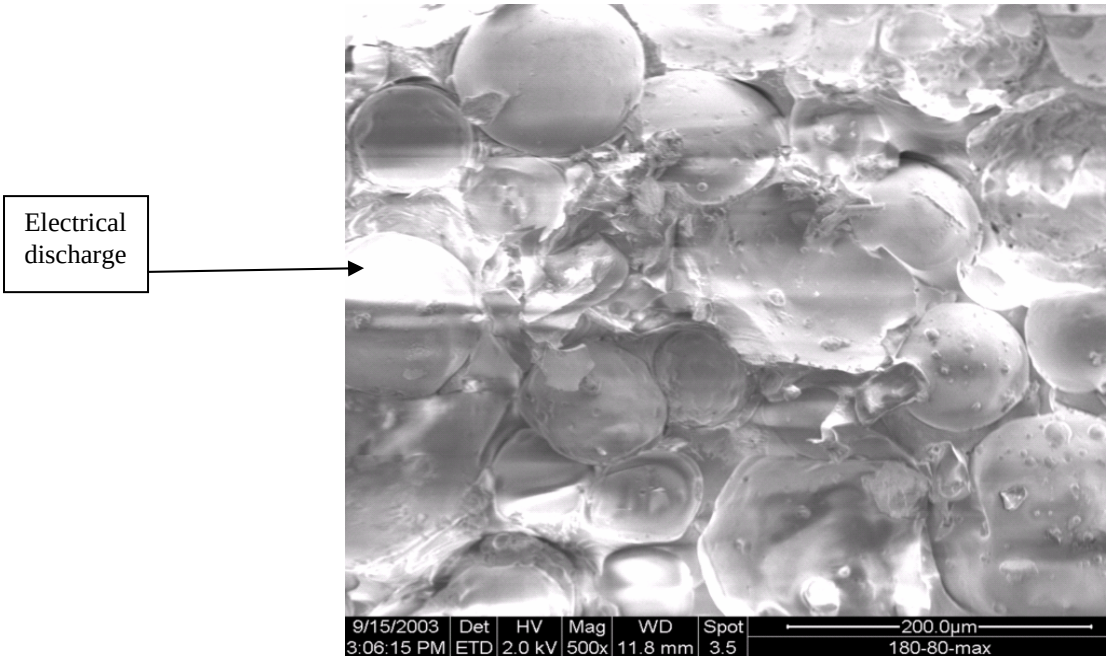

Figure 5: Micrograph of area 1 showing the cleavage fracture at the tip of the chevron cut and there is some electrical discharge in the image.

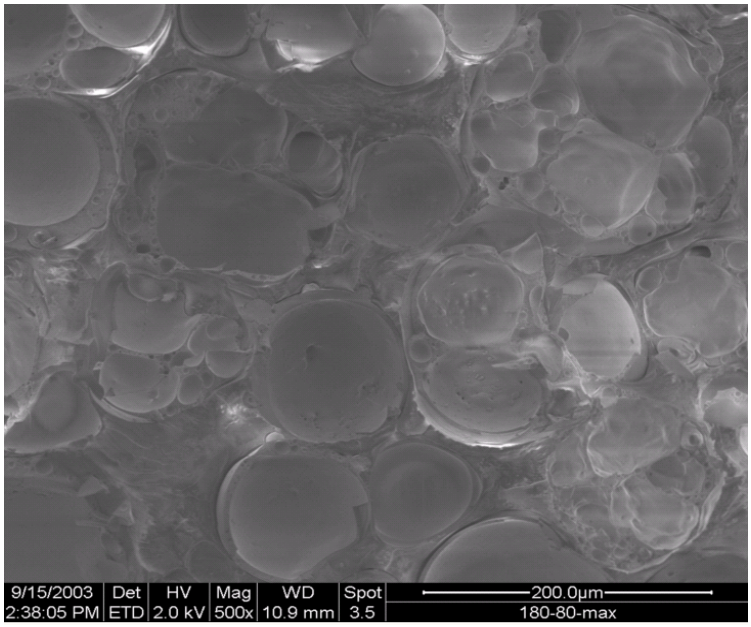

Figure 6: Micrograph of area 2 is the stretch-zone.

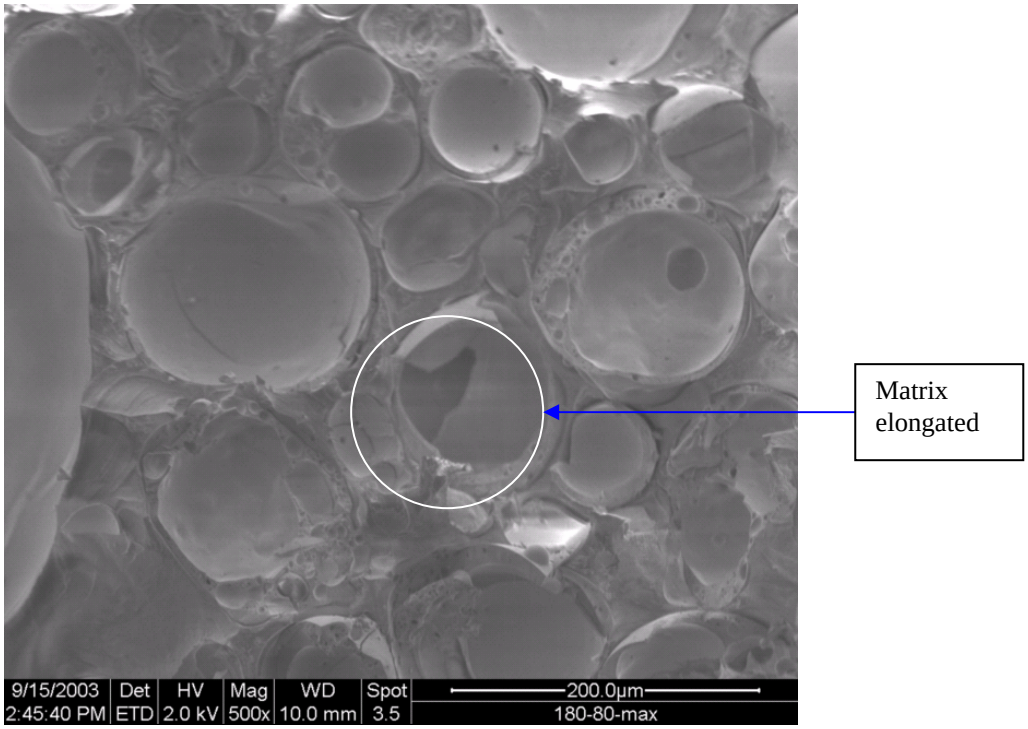

Figure 7: Micrograph of area 3 shows some matrices are elongated.

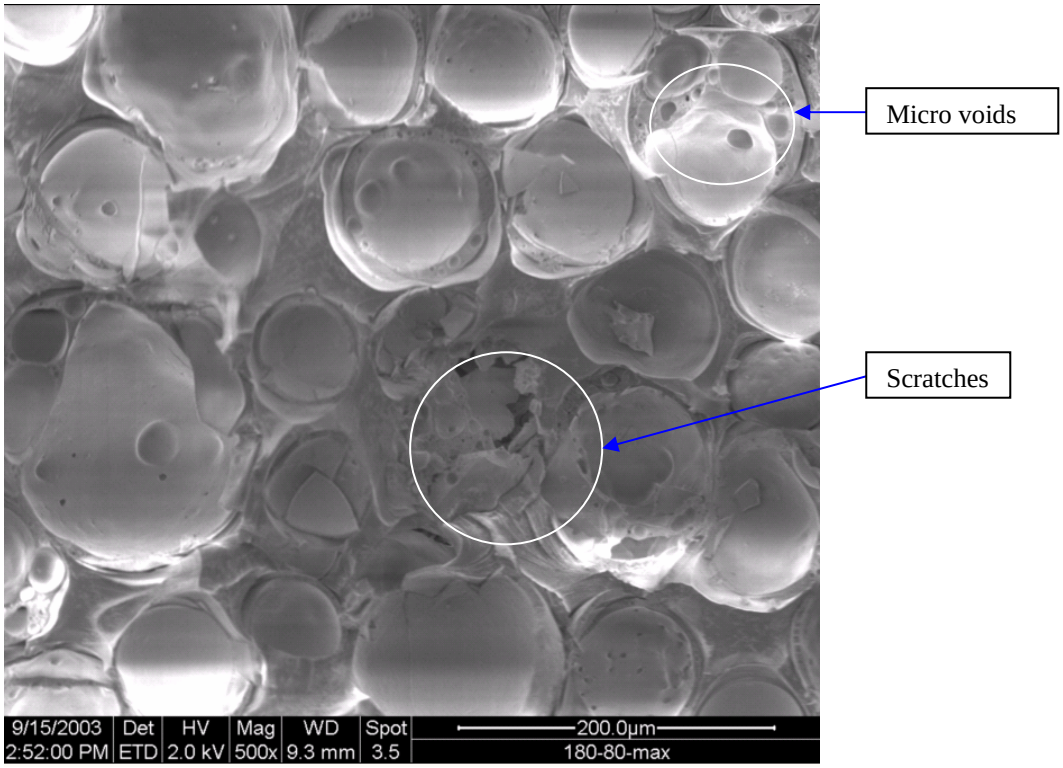

Figure 8: Micrograph of area 4 shows some micro voids and some scratches on the fractured surface

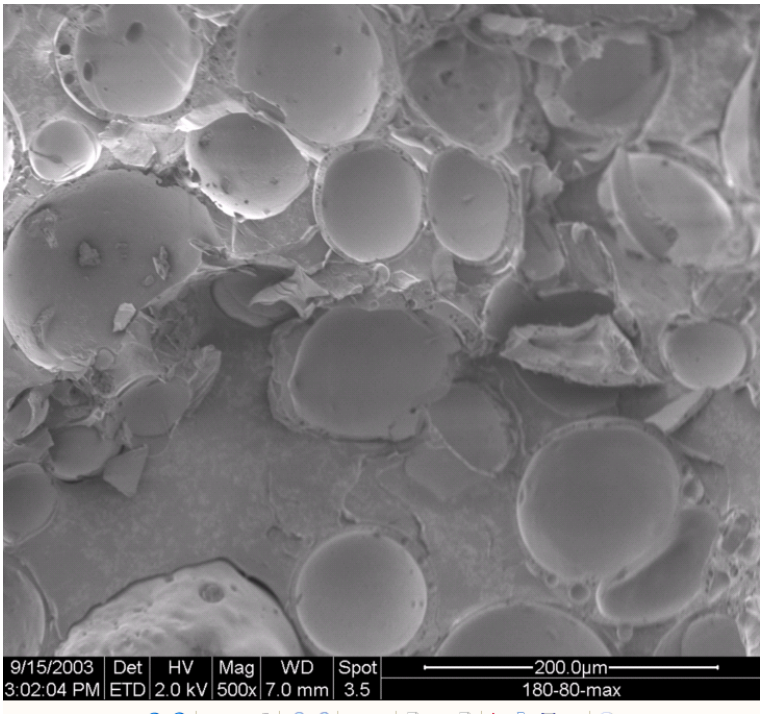

Figure 9: Micrograph of area 5 shows the brittle failure of the matrix.

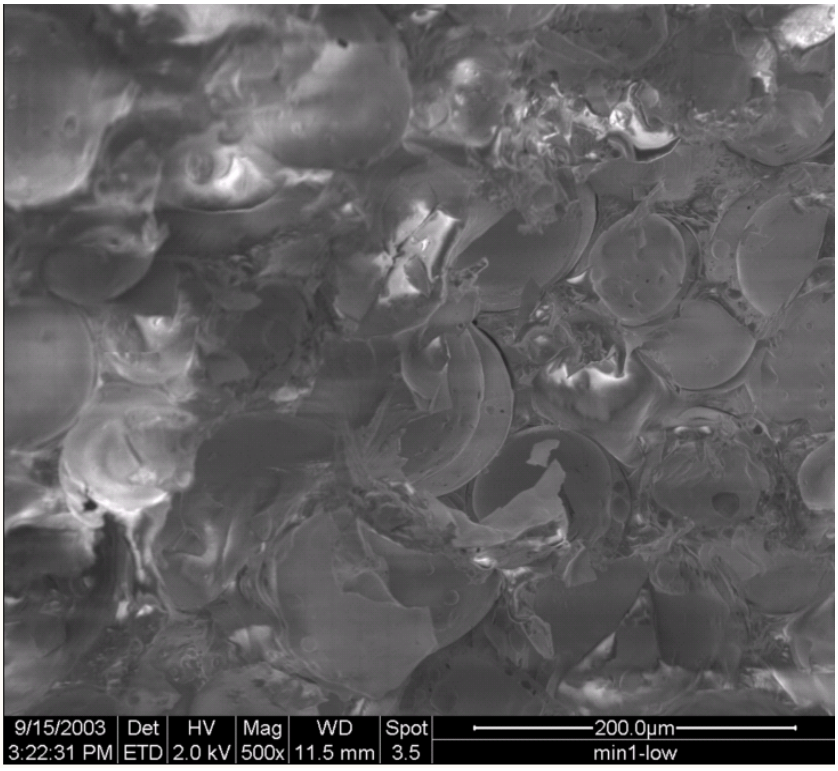

Figure 10: The micrograph of area 1 showing the matrixes are averagely elongated to initiate crack.

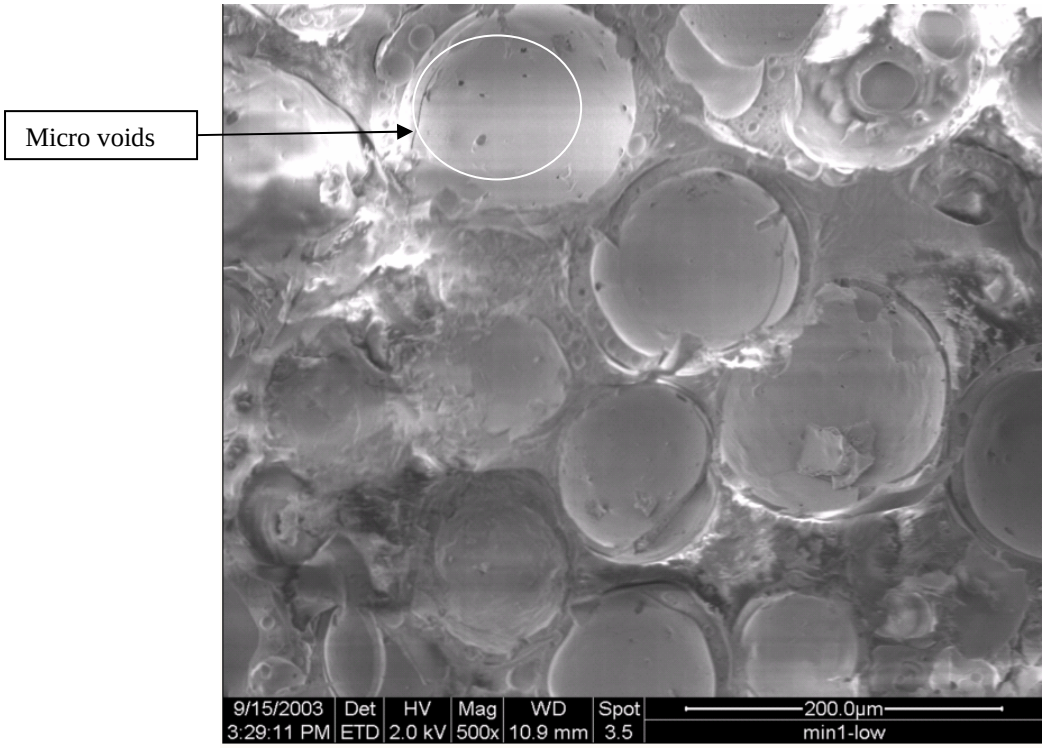

Figure 11: The micrograph of area 2 shows that some micro voids exist in the matrix.

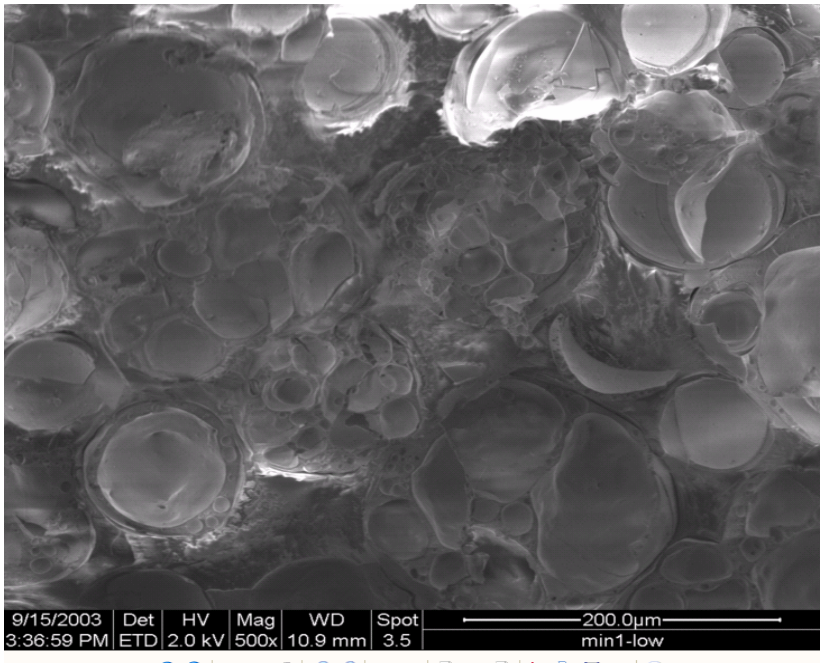

Figure 12: The micrograph of area 3 shows that most of the fractured surface has been damaged.

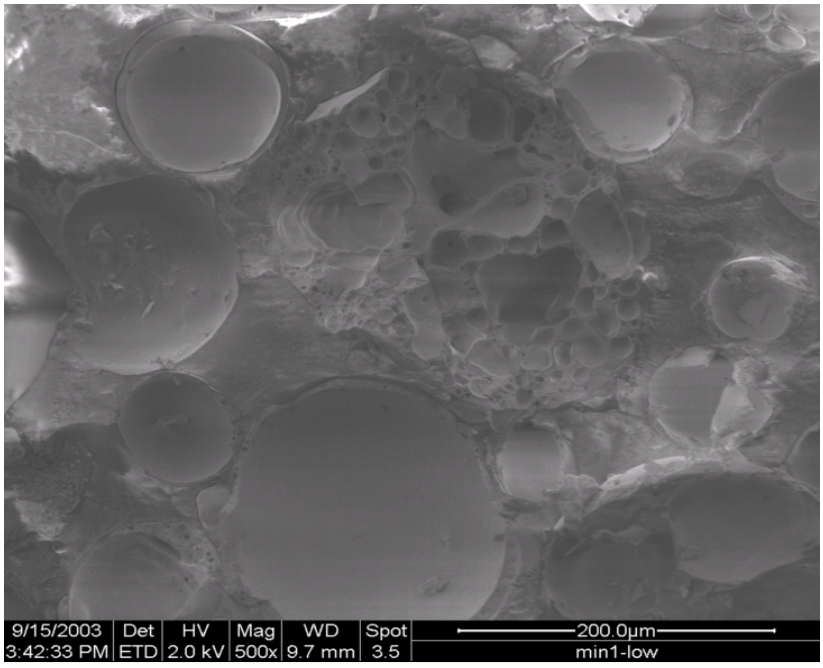

Figure 13: The micrograph of area 4 shows the brittle failure of material and some micro voids exist.

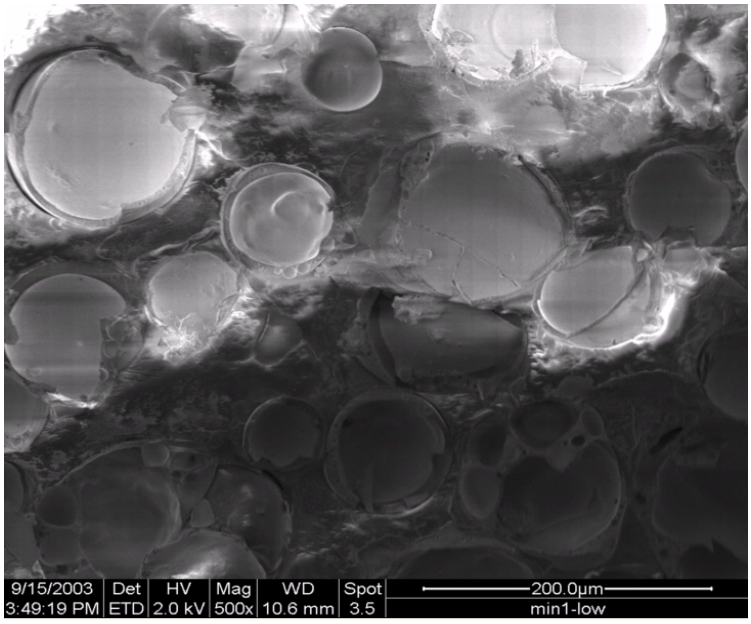

Figure 14: The micrograph of area 5 shows the brittle failure of the matrix and electrical discharge exists in the image.

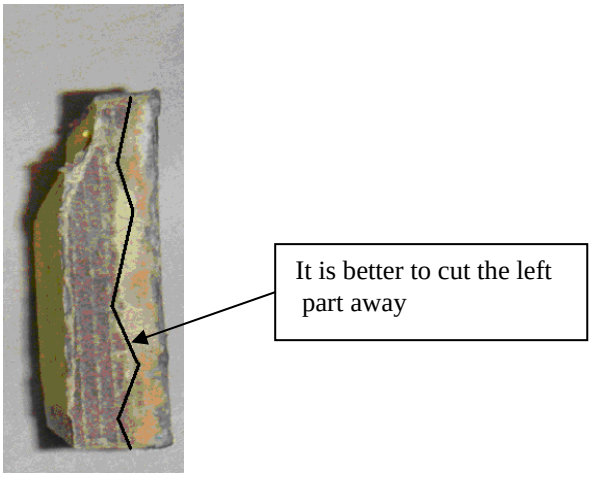

Figure 15: The gold coated part.

Table 1: Weight of materials required to make 500 ml of VE/FLYASH (33%)

Parameters / Materials	Resin	Accelerator	Fly ash	Composite
Relative density	1.1	1.0	0.7	---
Percentage by volume	56	---	44	100
Percentage by weight	67	---	33	100
Weight for 500 ml of composite	603.6 (g)	11.2 (g)	308 (g)	---

Table 2: Result of the fracture toughness and other parameters for VE cured under different conditions

Condition	Ambient	180 Watt		360 Watt	
Time	Nil	60s	80s	60s	80s
Elongation at Peak (mm)	1.214	1.254	1.162	1.234	1.121
Peak Load (N)	1365.33	1389.67	1358.67	1264.17	1281.67
Elongation at Break (mm)	1.520	1.557	1.518	1.478	1.445
Break Load (N)	1090.33	1119.17	897.33	1054.83	907.33
Fracture toughness (MPa$\sqrt{m}$)	51.65	52.72	51.41	47.85	48.49