Drop Weight Impact Test Fracture of Vinyl Ester Composites: Micrographs of Pilot Study

H. KU,* Y. M. CHENG,
C. SNOOK AND D. BADDELEY
Faculty of Engineering and Surveying
University of Southern Queensland, Australia

(Received June 10, 2004)
(Accepted November 8, 2004)

ABSTRACT: The shrinkage of vinyl ester particulate composites has been reduced by curing the resins under microwave conditions. The reduction in the shrinkage of the resins by microwaves will enable the manufacture of large vinyl ester composite items possible [12–15]. This project is to investigate the difference in impact strength between microwave cured vinyl ester particulate composites and those cured under ambient conditions. Drop weight impact test will be used to achieve the aim of the project [7]. The results show that the difference in the impact strength is minimal [5]. The original contribution of this paper is to view the fractured surface of composites cured under different conditions to find out whether they are the same. If they are the same, it can be deduced that the initial expansion of the composite due to microwave irradiation will not affect the final structure of the composite.

KEY WORDS: vinyl ester composite, microwaves, micrographs and Latin square

INTRODUCTION

COMPOSITE COMPONENTS MADE from vinyl ester resins by the Excellence Centre of Engineered Fiber Composites (ECEFC), University of Southern Queensland (USQ) suffer considerable shrinkage during hardening. This shrinkage is particularly serious if the fiber composite components are large. It can be more than 10%, which is much higher than that claimed by some researchers and resin manufacturers [6,16]. The main drawback of this shrinkage in a composite component is to have stresses set up internally. These stresses are usually tensile in the core of the component and compressive on the surface [18]. When these stresses act together with the applied loads during service, they may cause premature failure of the composite components.

*Author to whom correspondence should be addressed. E-mail: ku@usq.edu.au

0021-9983/05/18 1607–14 $10.00/0 DOI: 10.1177/0021998305051111

Currently, ECEFC solves the shrinkage problem by breaking a large composite component into smaller composite parts because smaller parts tend to have less shrinkage. These smaller parts are then joined together to form the overall structure. By doing this, the manufacturing lead time and costs of a composite component is significantly increased. By curing the composite under microwave conditions, the shrinkage of the material can be kept to a minimum [14,15]. This solves only half of the problems because one is not sure whether the microwave-cured composite has the same strength as that cured under ambient conditions. Cheng et al. [5] showed that the composites cured under microwaves had the same strength as that cured under ambient conditions.

The vinyl ester composite used is 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 studies [12–15].

The impact energy of a material is the amount of energy required to fracture a given volume of the material [4]. Therefore, the impact strength of a material is the energy required to initiate and propagate a crack through the material. The crack propagation energy is related to the toughness of the material and the length of that crack tip that must travel in order to fracture a component. This means the lower the value of the impact energy, the more brittle the material behaves [1].

DROP WEIGHT IMPACT TEST

The standard tests for impact strength of a material include Charpy test, Izod test, drop weight impact test, chip impacter test, and compression-after-impact (CAI) and tension-after-impact (TAI) tests. The preference for drop weight impact test over the more conventional methods, for example, Charpy and Izod tests, is due to the limitations that are experienced while trying to perform impact testing on composite materials. Another main advantage of using drop weight impact test over other standard tests is its ability to reproduce conditions under which real life component would be subject to impact loading. This means that if a material specimen or an actual item was to be tested, replication of the testing arrangement should be possible, provided enough testing samples should be produced. Furthermore, the advantage of using the drop weight impact test over pendulum impact test methods is that the specimen does not usually have to be clamped, depending on the testing arrangement [7].

The method of using the drop weight impact includes the use of a falling weight that impacts the specimen. This impact striker is known as a tup (shown in Figure 1), which falls through a vertical guide tube that directs it to the center of a specimen (see Figure 2). The guide tube must be perpendicular to the impact surface as stated in the American testing standards [3]. The energy released from the drop weight test is,

$$E = mgh - l \tag{1}$$

where E is the energy (J), m is the mass of tup (kg), g is the gravity (m/s^2), h is the height (m), and l are the losses incurred by friction and other sources (J). The loss is negligible in the test.

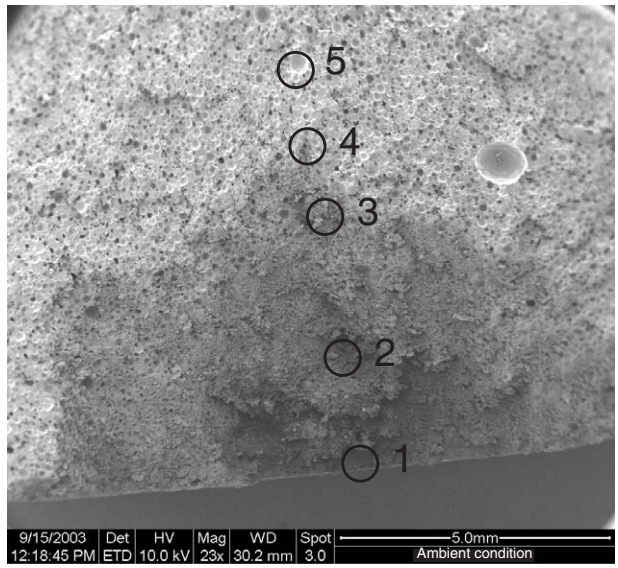

Figure 1. *Points chosen to be investigated with specimens cured under ambient conditions.*

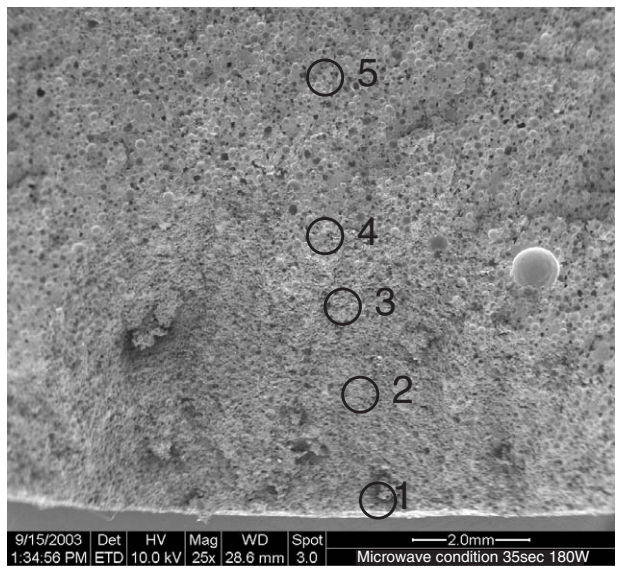

Figure 2. *Points chosen to be investigated with specimens cured with microwaves of 180 W for 35 s.*

In testing composite materials, the constant weight and varying height method has to be used because the composite material is strain rate sensitive [3,7]. Ubachs [21] found that the mean height to impact the samples of epoxy resin with 33% by weight of particle reinforcement was 900–1000 mm. Since the mechanical properties, including impact strength of vinyl ester resins are inferior to those of epoxy resins, it is expected that the

Table 1. Average energy required to fracture specimens cured with a power level of 180 W.

Curing condition	Microwave with a power level of 180 W			
Specimens type	Fractured specimens			
Symbols representation	σ = Standard deviation; m = meter			
Specimens materials	VE/FLYASH (33%)			
	Ambient condition	Microwave cured		
Exposure time	(0 s)	30 s	35 s	40 s
Energy used to initiate the crack	8.84 J ($\sigma = 0.893$)	8.22 J ($\sigma = 0.803$)	8.81 J ($\sigma = 1.160$)	8.66 J ($\sigma = 1.064$)
Energy used to propagate the crack	2.16 J ($\sigma = 0.590$)	2.62 J ($\sigma = 0.818$)	2.16 J ($\sigma = 0.878$)	2.16 J ($\sigma = 0.574$)
Total energy dissipated	11.00 J ($\sigma = 0.355$)	10.84 J ($\sigma = 0.297$)	10.97 J ($\sigma = 0.386$)	10.82 J ($\sigma = 0.528$)
Displacement at peak force	0.0018 m ($\sigma = 0.0001$)	0.0017 m ($\sigma = 0.0001$)	0.0018 m ($\sigma = 0.0001$)	0.0017 m ($\sigma = 0.0001$)

samples will fail when the mean height of dropping the tup is <900 mm [2]. The formal classification of test configuration is given in Table 1 [7].

Instrumentation is incorporated to reduce the number of samples required and to increase accuracy. The required items are an accelerometer, a charge amplifier, an A/D converter, and a data acquisition equipment. The setup of the equipment is shown in Figure 3 [7,18]. Instrumented test data can provide more detailed information about the impact event including force distribution, peak force, and duration. The results from previous experimentation gave an indication of the requirements needed for such instrumentation. They are as follows [18]:

- acceleration response range of 0–4000 m/s^2;
- frequency response of 0.1 Hz–5 kHz;
- unidirectional response;
- minimum sample rate of 100 kHz.

The Composite Samples

The vinyl ester resin used is Hetron 922 PAS in summer and Hetron 922 PAW in winter. The vinyl ester is dissolved in 50% by weight of styrene. In this study, Hetron 922 PAW was used. It is based on the reaction between methacrylic acid and diglycidylether of bisphenol-A. The resin–hardener ratio used in the experiment was 98% resin by volume and 2% hardener by volume [2]. The reinforcer was flyash (ceramic hollow spheres) particulate and it made 44% by volume or 33% by weight in the cured vinyl ester composite [VE/FLYASH (33%)]. Forty four percent by volume or 33% by weight of flyash in the composite is considered optimum by the ECEFC because the composite will have a reasonable fluidity for casting combined with a good tensile strength in service.

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 colorless liquid and is first mixed with the colorless accelerator. After the flyash is added to the mixture, they are mixed to give the uncured composite. Table 2 shows the mass, in grams, of resin, accelerator,

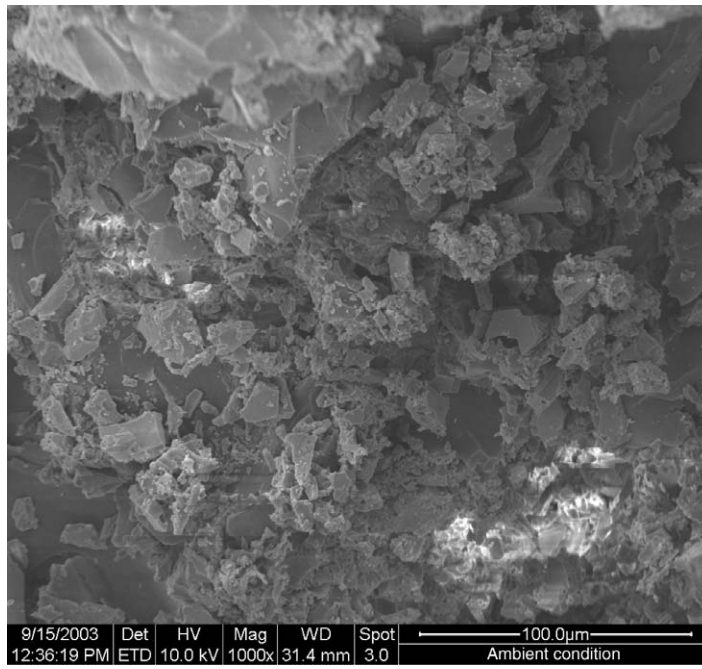

Figure 3. *Area 1, ambient cured, magnified 1000 times.*

Table 2. Weight of materials required to make 250 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		301.8 (g)	5.6 (g)	154 (g)	–

and flyash required respectively to make a volume of 250 mL of uncured composite (of 44% by volume or 33% by weight of flyash). The uncured composite was then poured into the molds of PVC tubes for curing in ambient or microwave conditions [14]. The molds are depicted in Figure 4. The slots were made by inserting plastic sheets of suitable thickness. Figure 5 shows some of the VE/FLYASH (33%) short bar specimens ready for the tests.

Microwaves/Material Interactions

The material properties of greatest importance in microwave processing of a dielectric are the complex relative permittivity $\varepsilon = \varepsilon' - j\varepsilon''$ and the loss tangent, $\tan\delta = \varepsilon''/\varepsilon'$ [17]. The real part of the permittivity, ε', sometimes called the dielectric constant, mostly determines how much of the incident energy is reflected at the air–sample interface, and how much enters the sample. The most important property in microwave processing is the loss

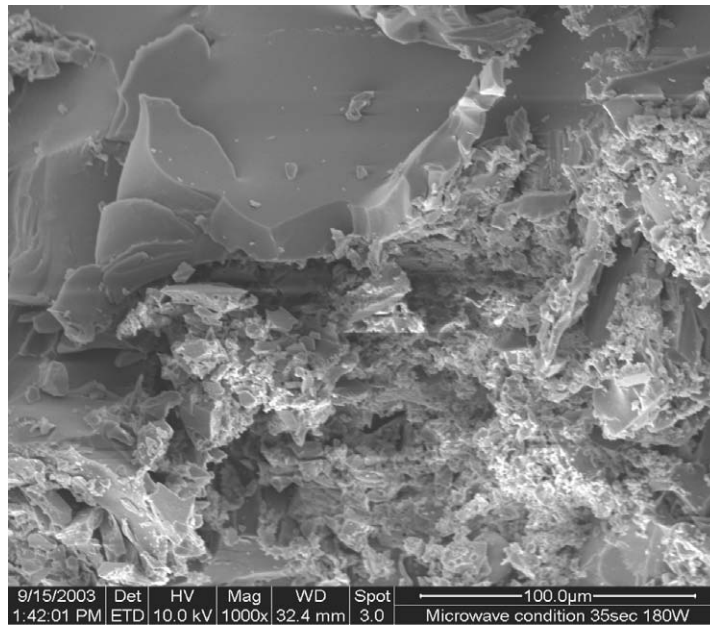

Figure 4. *Area 1, microwave cured (180 W for 35 s), magnified 1000 times.*

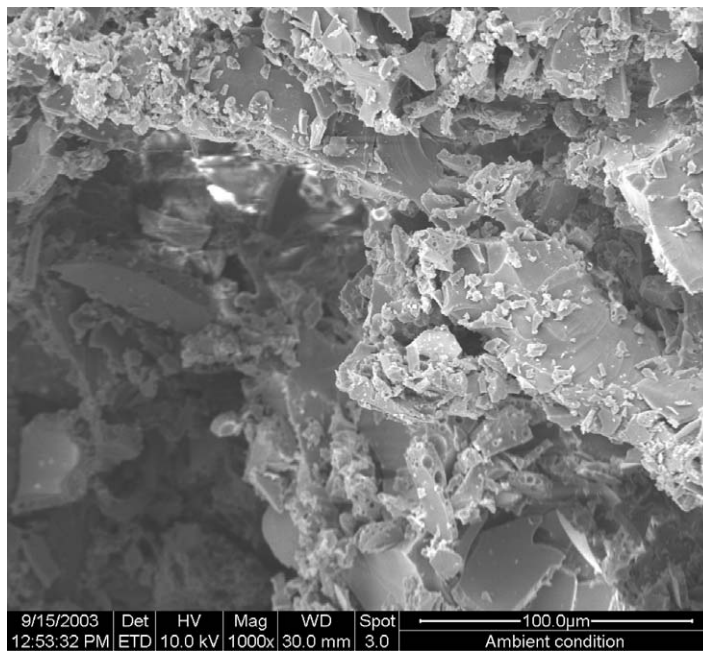

Figure 5. *Area 2, ambient cured, magnified 1000 times.*

tangent, $\tan\delta$ or dielectric loss, which predicts the ability of the material to convert the incoming energy into heat. For optimum microwave energy coupling, a moderate value of ε', to enable adequate penetration, should be combined with high values of ε'' and $\tan\delta$, to convert microwave energy into thermal energy.

Microwaves heat materials internally and the depth of penetration of the energy varies in different materials. The depth is controlled by the dielectric properties. Penetration depth is defined as the depth at which approximately $1/e$ (36.79%) of the energy has been absorbed. It is also approximately given by [3]:

$$D_{\mathrm{p}} = \left(\frac{4.8}{f}\right)\frac{\sqrt{\varepsilon'}}{\varepsilon''} = \frac{4.8}{f}\frac{1}{\sqrt{\varepsilon'}}\frac{1}{\tan\delta} \tag{2}$$

where D_{p} is in cm, f is in GHz, and ε' is the dielectric constant.

Note that ε' and ε'' can be dependent on both temperature and frequency, the extent of which depends on the materials.

Interaction of Microwaves with VE/FLYASH (33%)

Whether a material will absorb microwave energy and convert it into heat depends on its relative complex permittivity and loss tangent. Ku et al. [11] showed that liquid rapid Araldite (epoxy resin) has a dielectric constant of 2.81 and a loss tangent of 0.244 at 2.45 GHz at room temperature. The loss tangent is quite high and it is expected that Araldite will absorb microwaves readily and convert it into heat. Vinyl ester resin is produced from modified epoxy resin and methacrylic acid and epoxy resin absorbs microwave irradiation readily. It is therefore expected that it will also absorb microwaves readily [9,10,20]. A possible risk in applying microwave energy to the vinyl ester composite is the interaction of the styrene in the resin with the high voltage (HV) transformer in the oven. The oven cavity is spot welded together and is not necessarily water/air/steam proof. Styrene is a highly flammable vapor and is given off during the curing process of the composite. High vapor concentrations of styrene may cause explosions. The gas may explode if it is ignited by an electric arc or the heat of the HV components. The oven does not have an exhaust fan. A blower motor inside sucks air through the air filter at the front and cools the HV transformer as the air passes. The air from the fan is blown into a duct and it cools the magnetrons. Some air is forced into the cavity at the back and then out of the steam exhaust outlet at the back. This is where the styrene-containing air will interact with HV transformer and ignition or explosion may result. Due to this, the oven was modified to ensure that ignition or explosion would not happen. Details of the modifications have been mentioned in another paper [13]. The microwave facility used in this study is shown in Figure 6.

Sample Size

In this study, VE/FLYASH (33%) was exposed to microwave irradiations of 180 and 360 W. The duration of exposure for both power levels was 30, 35, and 40 s. With the above varying parameters of power levels and duration of exposure in mind, sample size for each set of parameters can be determined. Latin Square is used to establish the

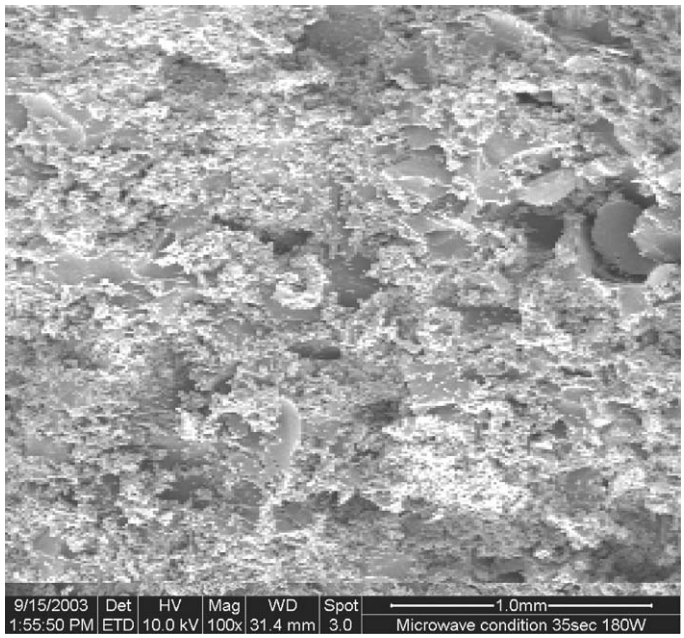

Figure 6. *Area 2, microwave cured (180 W for 35 s), magnified 1000 times.*

Table 3. Latin square for different treatments of vinyl ester composites by microwaves.

360* (30)#	360 (35)	360 (40)
180 (30)	180 (35)	180 (40)
0 (30)	0 (35)	0 (40)

*Power level
#Duration of exposure

required sample size for each type of composite [8]. If all variables are taken into account when establishing the Latin Square, the matrix will be a 3×3 matrix (Table 3). Zero power level means no microwave irradiation and the samples are cured under ambient conditions. On account of the zero power level, the number of samples required will be $2 \times 3 = 6$ because the combination of elements of the first column with the first elements of the three rows will be null, i.e., cured under ambient conditions. Three uncured short bar specimens were exposed to microwaves each time. At the same time, three similar short bar composites were cured under ambient conditions and their fracture toughness values were used as a benchmark for comparison.

Energy Consumed in Breaking the Samples

Comparison of average energy used to initiate the crack can provide good indication of the initial failure of the specimens among these groups. Table 1 shows the results of the average energy used to initiate the crack between the specimens cured under ambient and microwave conditions with a power level of 180 W. Samples cured with

microwaves for 30 s tended to require less energy to initiate the crack. It requires 0.62 J of energy less than those cured under ambient conditions. In addition, the spread of this group was smallest as compared with others. From specimens cured with microwaves for 40 s, the average energy required to initiate the crack was found almost identical to those cured for 35 s. The difference in average energy required to break the specimens was only 0.15 J between them and the difference in the group cured under ambient conditions was 0.18 J. The spread was smaller than those microwaved for 35 s. The amount of energy required to initiate crack in specimens cured with microwaves for 35 s was very close to that required to do the same for samples cured under ambient conditions. The difference between them was found to be 0.03 J. After impact testing, the two specimens were further investigated for the fracture behavior with the aid of a scanning electron microscope (SEM).

RESULTS AND DISCUSSION

Figures 1 and 2 show the five locations studied under SEM for ambient-cured and microwave-cured (180 W and 35 s) samples, respectively. Figures 3 and 4 illustrate area 1 of ambient-cured and microwave-cured samples, respectively. The magnification for both locations is 1000 times. It is observed that there is more powder in the crushed zone of the sample cured under microwave conditions. Otherwise, the difference between the two figures was not much. Figures 5 and 6 illustrate area 2 of ambient-cured and microwave-cured samples, respectively. More powder was also found in the crushed zone of the microwave-cured sample. Similar phenomena were observed with three other areas, 3, 4, and 5 as shown in Figures 7–12 for ambient-cured and

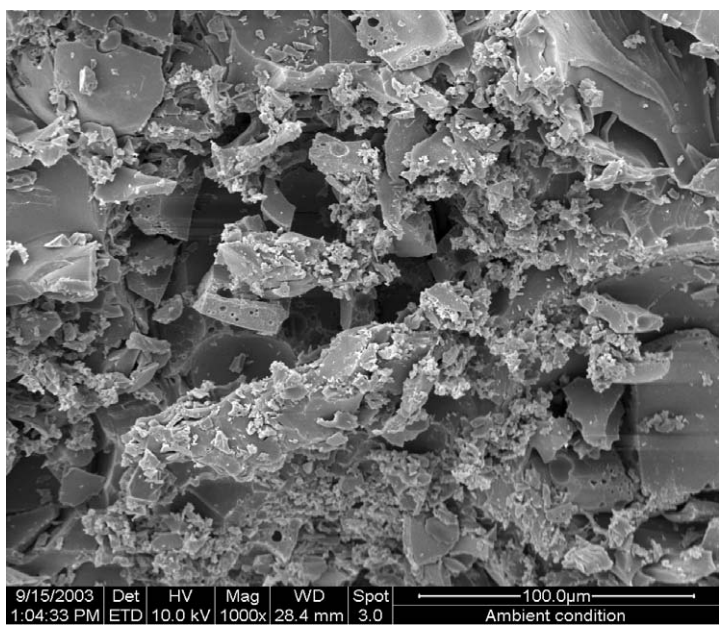

Figure 7. *Area 3, ambient cured, magnified 1000 times.*

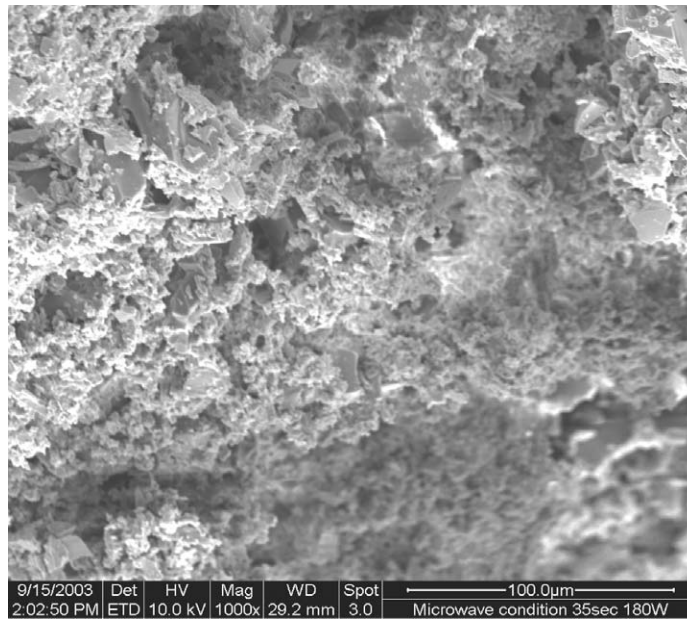

Figure 8. *Area 3, microwave cured (180 W for 35 s), magnified 1000 times.*

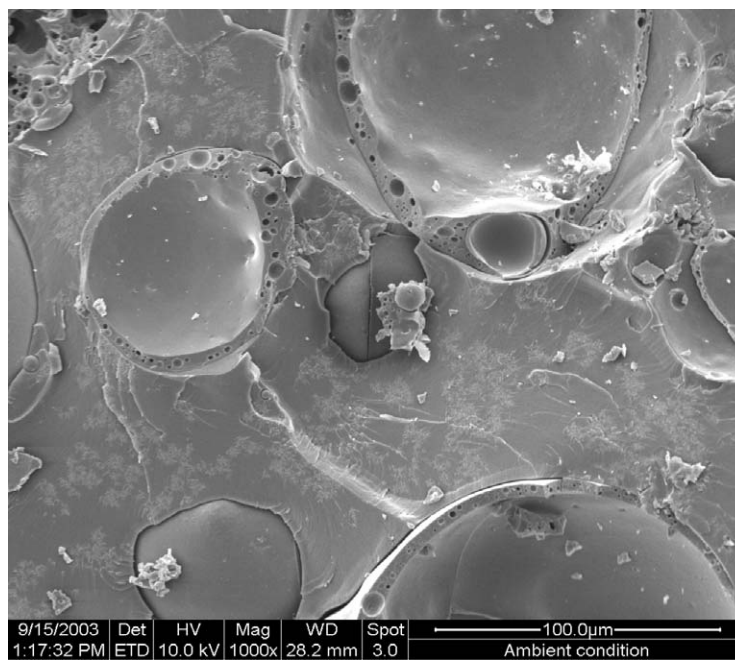

Figure 9. *Area 4, ambient cured, magnified 1000 times.*

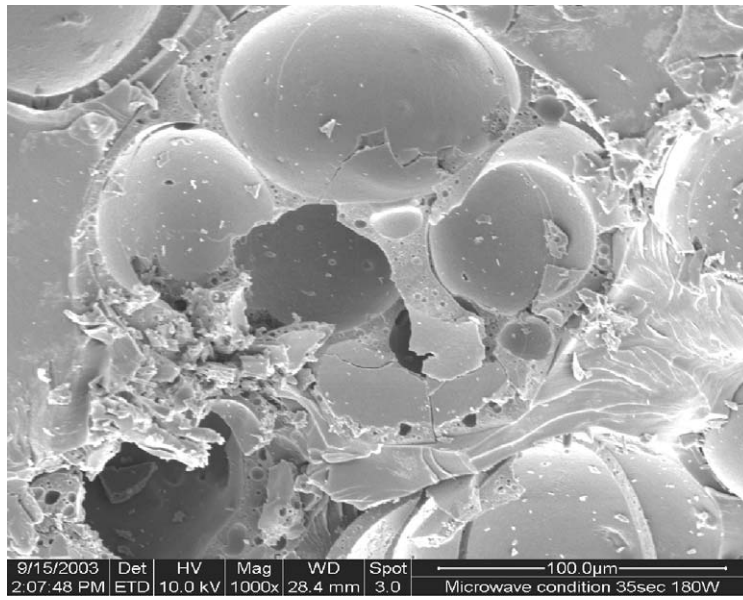

Figure 10. *Area 4, microwave cured (180 W for 35 s), magnified 1000 times.*

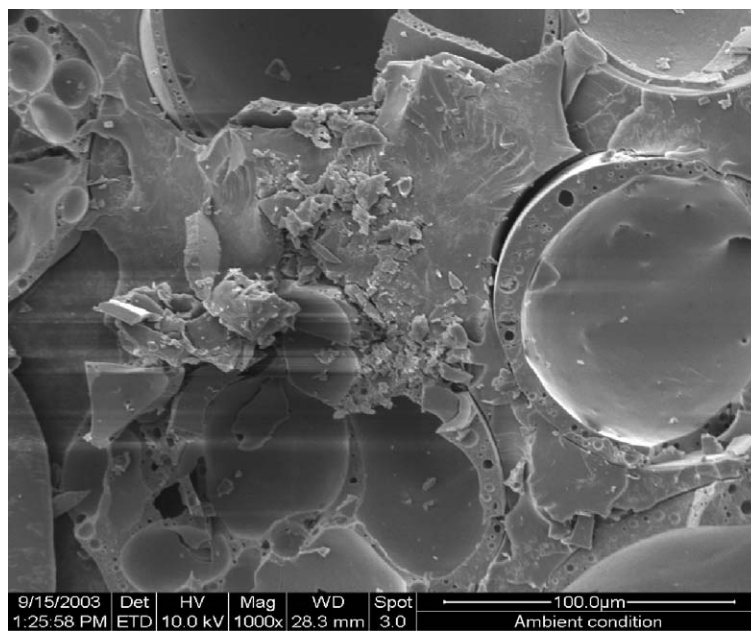

Figure 11. *Area 5, ambient cured, magnified 1000 times.*

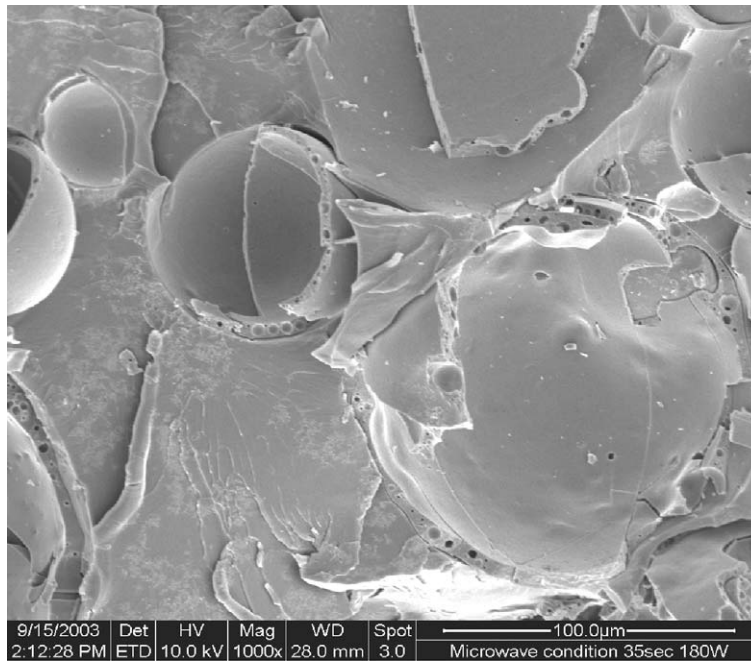

Figure 12. *Area 5, microwave cured (180 W for 35 s), magnified 1000 times.*

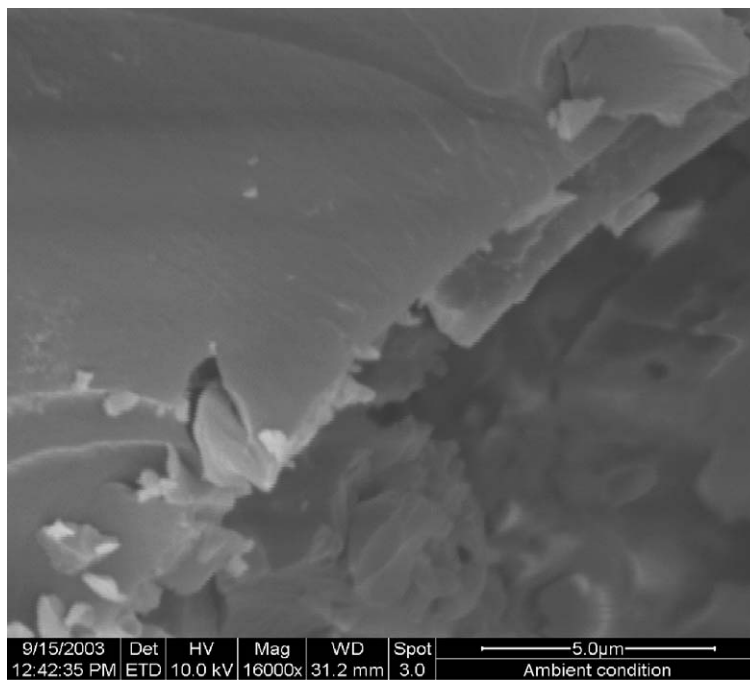

Figure 13. *Area 1, ambient cured, magnified 16,000 times.*

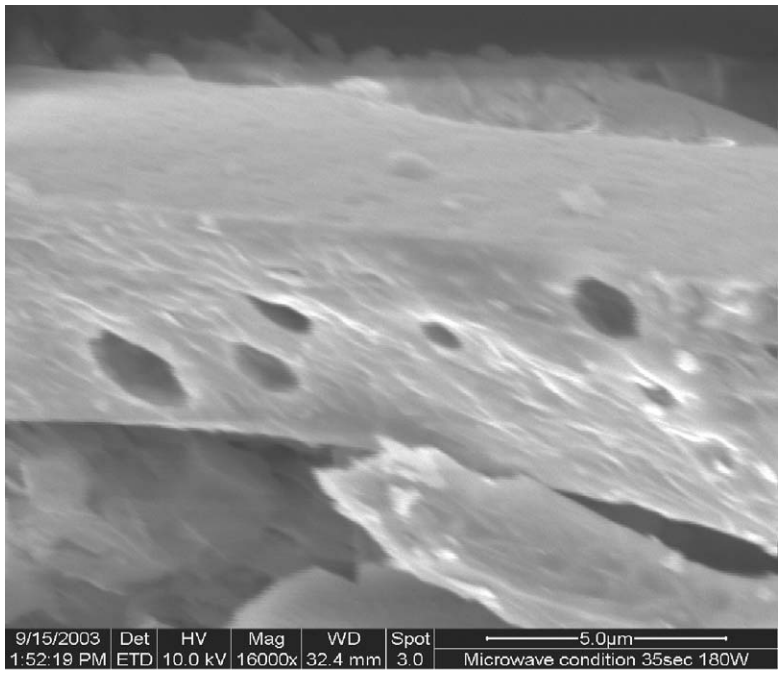

Figure 14. *Area 1, microwave cured (180 W for 35 s), magnified 16,000 times.*

microwave-cured samples, respectively. The magnification for Figures 5–12 for the locations studied is 1000 times.

Figures 13 and 14 illustrate area 1 of ambient-cured and microwave-cured samples, respectively. The magnification for both samples is 16,000 times. Flake or powder can be found in Figure 14 but not in Figure 13. This further proves that the discussion presented earlier is correct.

By and large, under 1000 times magnification, the results obtained for specimens cured under microwave conditions showed not much difference with those cured under ambient condition. The difference in average energy required to fracture or initiate the crack in these specimens was found to be very small. The more powderized appearance in the crushed zone may be due to a higher impact resistance. In addition, quite a number of specimens that were cured with microwaves tended not to fracture when they were impacted from a drop height of 400 mm; whereas most of the specimens cured under ambient conditions tended to fail at a drop height of 400 mm [5].

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