

# Shrinkage Reduction of Thermoset Matrix Particle Reinforced Composites During Curing Using Microwaves Irradiation

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*Composites made from vinyl ester resins suffer considerable shrinkage during curing and after solidification. This shrinkage is particularly serious if the fibre composite components are large and can be up to 12% (Schwartz, 1992; Clarke, 1996; Matthews and Rawlings, 1994). Currently, the shrinkage problem is solved 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. However, the manufacturing lead-time and costs of a composite component are significantly increased. This project attempts to reduce the shrinkage of the components cast from uncured 33% by weight or 44% by volume of fly ash particulate-reinforced vinyl esters [VE/FLYASH (33%)]. This is accomplished by shortening the curing time of the resins using penetrating microwave energy. The formation of pinholes in the samples resulted in the expansion of the volume of the materials in the initial stage of the curing process. Microwave irradiation did reduce curing shrinkage in the composite.*

**Keywords:** Vinyl Ester, Shrinkage, Pinholes and Microwave Irradiation

## Introduction

The most common thermosets used as composite matrices are unsaturated polyesters, epoxies and vinyl esters. Unsaturated polyesters dominate the market, whereas epoxies are preferred in high-performance applications. Unsaturated polyester offers an attractive combination of low price, reasonably good properties, and simple processing. However, basic unsaturated polyester formulations have drawbacks in terms of poor temperature and ultra-violet tolerance. Additives may significantly reduce these disadvantages to suit most applications. Where mechanical properties and temperature tolerance of unsaturated polyesters do not meet the required performance criteria, epoxies are often used due to their significant superiority in this respect. Of course, these improved properties come at a higher price and epoxies are commonly used in areas where cost tolerance is highest (Astrom, 1997). When epoxy resins are used to make composite structures, there are three main drawbacks (Pritchard, 1999): i) because they have a two-step hardening process, they are slow to cure, and they require a minimum postcure of 2 to 4 hours at 120°C to achieve 70 - 80% of optimal properties; ii) they usually have high viscosities which limit their tendency to wet glass fibres efficiently; iii) the use of amine hardeners increases the susceptibility of the cured resins to acid attack.

With these issues in mind, the so-called epoxy vinyl ester range of resins (vinyl ester resins) was developed in the 1960s (Pritchard, 1999). Vinyl esters, as they are usually called, are closely related chemically to both unsaturated polyesters and epoxies and in most respects represent a compromise between the two. They were developed in an attempt to combine the fast and simple crosslinking of unsaturated polyesters with the mechanical and thermal properties of epoxies (Astrom, 1997).

To achieve these objectives, ie to reduce the shrinkage of vinyl esters, it is necessary to apply microwave energy in a multimode oven cavity to samples of vinyl ester resins under controlled conditions. A commercial 1.8 kW microwave oven was used. The power rating of 1.8 kW was actually achieved by launching microwaves from two 0.9 kW magnetrons. The power inputs were designed to vary from 10% (180 W) to 100% (1800 W) in steps of 180 W. Health concerns with vinyl esters are considered synonymous with the most common crosslinking agent, the styrene, and not with the polymers themselves. Styrene is volatile and evaporates easily and becomes an inhalation hazard.

## Microwaves/Material Interactions

The material properties of greatest importance in microwave processing of a dielectric are the complex relative permittivity  $\epsilon = \epsilon' - j\epsilon''$  and the loss tangent,  $\tan \delta = \frac{\epsilon''}{\epsilon'}$  (Metaxas and Meredith, 1983). The real part of the permittivity,  $\epsilon'$ , 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 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  $\epsilon'$  is needed to enable adequate penetration and should be combined with high values of  $\epsilon''$  and  $\tan \delta$  in order to convert a significant amount of 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  $\frac{1}{e}$  or 36.79% of the energy has been absorbed. It was also approximately given by (Bows, 1999):

$$D_p = \left( \frac{4.8}{f} \right) \frac{\sqrt{\epsilon'}}{\epsilon''} = \frac{4.8}{f} \frac{1}{\sqrt{\epsilon''} \tan \delta} \quad (1)$$

where  $D_p$  is in cm,  $f$  is in GHz,  $\epsilon'$  is the dielectric constant, mostly determines how much of the incident energy is reflected at the air-sample interface, and how much enters the sample and  $\tan \delta$  is the loss tangent or dielectric loss, which predicts the ability of the material to convert the incoming energy into heat.

Note that  $\epsilon'$  and  $\epsilon''$  can be dependent on both temperature and frequency, the extent of which depends on the materials. Some details of microwaves/materials interactions are shown in Fig 1 (Sheppard, 1988).

## Vinyl Ester Resins and Their Crosslinking

Unsaturated resins such as polyesters and vinyl esters have ester groups in their structures. Esters are susceptible to hydrolysis and this process is accelerated and catalysed by the presence of acids or bases. Vinyl esters contain substantially less ester groups than polyesters as they contain

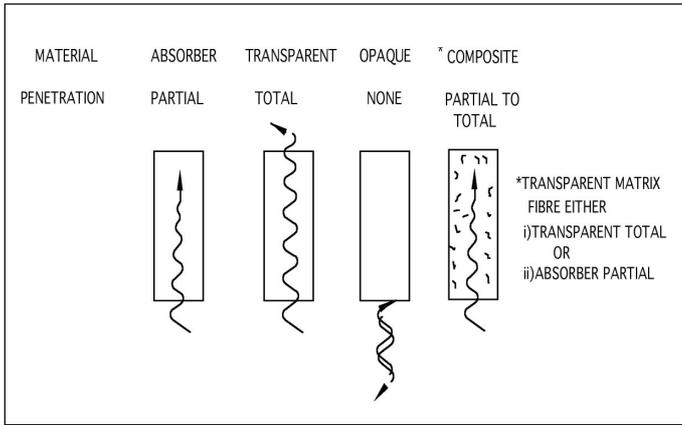


Figure 1 – Interaction of Microwaves with Materials

only one at each end of the resin molecule. This is illustrated by the structure of bisphenol A vinyl ester in Fig 2. This means that vinyl esters, just like epoxies, have few possible crosslink sites per molecule. Vinyl esters of high molecular weight will therefore have relatively low crosslink density and thus lower modulus than if the starting point is a lower molecular-weight resin. Vinyl esters crosslink in time frames and under conditions similar to those of unsaturated polyesters, ie fairly quickly and often at room temperature (Astrom, 1997). Methacrylic acid is used to manufacture the vinyl esters. This means that next to each ester linkage is a large methyl group. This group occupies a lot of space and sterically hinders any molecule approaching the ester group by impeding their access. These two aspects of the design of the vinyl ester molecule combine to make them more chemically resistant than polyesters (Pritchard, 1999).

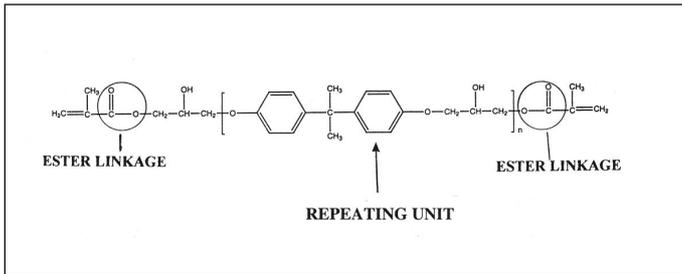


Figure 2 – The Structure of Bisphenol A Vinyl Ester (Astrom, 1997)

### Crosslinking of Vinyl Esters

The polymerisation product between methacrylic acid and bisphenol A is vinyl ester, which can be a highly viscous liquid at room temperature or a low melting point solid, depending on the bisphenol A used. For further processing, the polymer is dissolved in a low molecular monomer, or reactive diluent, usually styrene, the result is a low viscosity liquid referred to as resin. With the addition of a small amount of initiator to the resin the crosslinking reaction, or curing, is initiated. The initiator used is usually an organic peroxide, eg methyl ethyl ketone peroxide. The added amount is about 1 to 2% by volume. The peroxide decomposes after it is added to the resin and the reaction is exothermic. The initiator is a molecule that produces free radicals. The free radicals attack one of the double bonds on the ends of the resin and bonds to one of the carbon atoms, thus producing a new free radical at the other carbon atom. The initiation and crosslinking processes are illustrated in Fig 3. This newly created free radical is then free to react with another double bond. Since the small monomer molecules and the styrene molecules move much more freely within the resin than the high molecular weight polymer molecules, this double bond very likely belongs to a styrene molecule, as illustrated in the bridging step of Fig 3. The bridging step creates a new free radical on the styrene, which is then free to react with another double bond and so on. Obviously the styrene is not only used as solvent, but actively takes part in the chemical reaction.

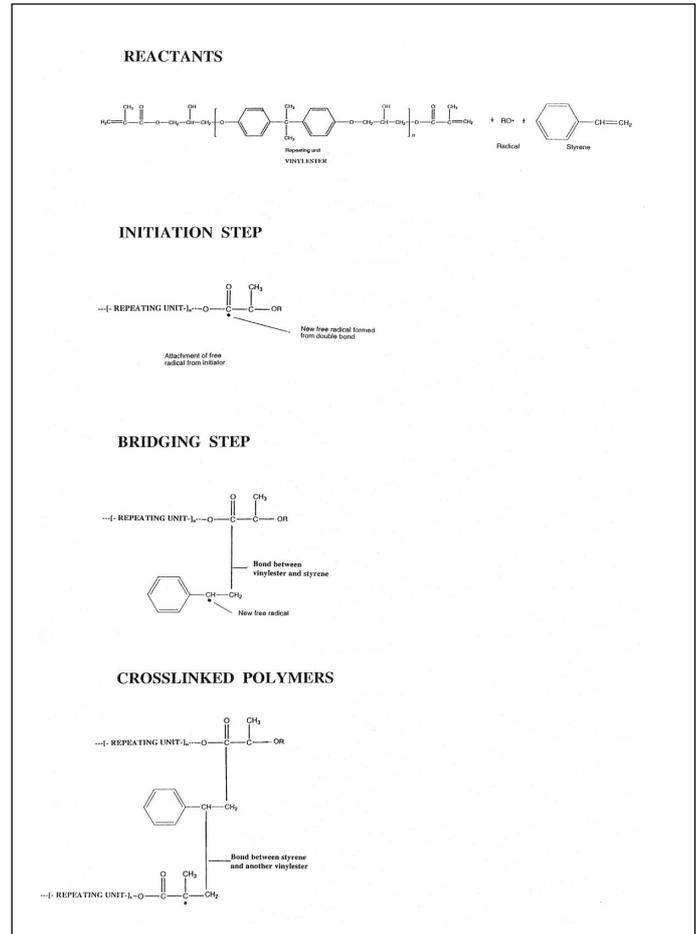


Figure 3 – Schematic of Addition or Free Radical Crosslinking of Vinyl Ester (Astrom, 1997)

As the molecular weight of the crosslinking polymer increases, it gradually starts to impair the diffusional mobility of the growing molecules and the reaction rate slows down. When the movement of the free radicals is also impaired they are prevented from finding new double bonds to continue the reaction which then stops. The result of the crosslinking reaction is a gigantic 3D molecule that from a macroscopic point of view leads to the transformation of the liquid resin into a rigid solid. Fig 4 shows typical temperature time relations for crosslinking of a vinyl ester following addition of initiator (Astrom, 1997). The three solid curves on the right hand side of the Fig represent room temperature crosslinking of vinyl esters. The different curves illustrate different amounts of initiator, inhibitor, accelerator, ambient temperature and humidity or volume of

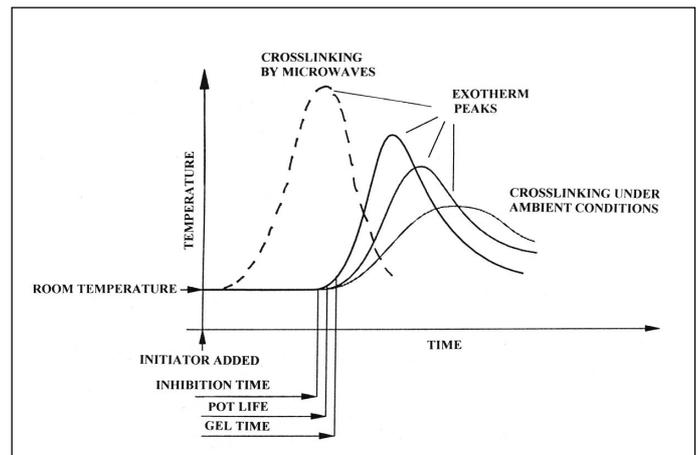


Figure 4 – Temperature Time Relationships for Crosslinking of Vinyl Ester (Astrom, 1997)

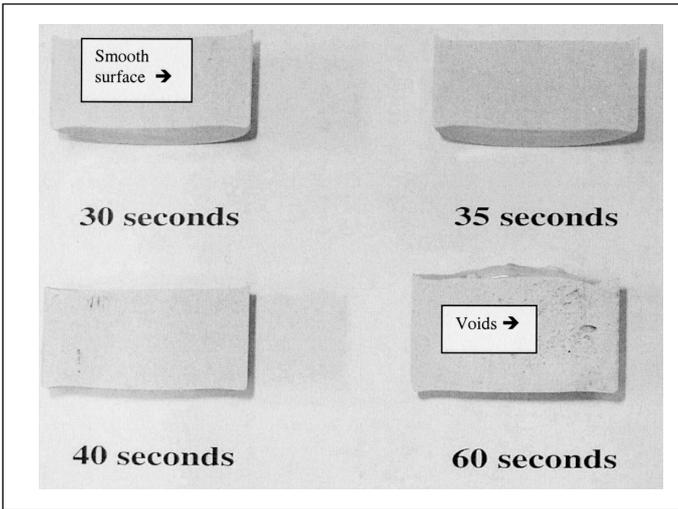


Figure 5 – The Cross Sections of 4 Composite Samples Exposed to Different Duration of Microwave Irradiation

resin. A reduced amount of initiator and accelerator, as well as an increased amount of inhibitor, leads to later crosslinking at lower temperature, and the opposite is true. The temperature does not immediately increase after addition of an initiator despite free radicals being produced. The crosslinking reaction does not start and the temperature does not increase until all inhibitor molecules have reacted with free radicals, which corresponds to inhibition time. As crosslinking commences, the pot life is over. The resin becomes a rubbery solid quickly and the gel time is reached. The crosslinking activity now accelerates very rapidly until the increasing molecular weight of the crosslinking polymer starts restricting molecular movement, which occurs around the maximum temperature and the crosslinking gradually tapers off. On the other hand, the dashed line curve on the left hand side of the Fig illustrates the hypothetical crosslinking as a result of the application of microwave to the resin. In this case, the inhibition time is short and maximum temperature is reached quickly. It is anticipated that the result of such a curing reduces the shrinkage of vinyl ester because of its initial thermal expansion, which is observed by the initial rise of the level of the composite.

### Shrinkage in VE/FLYASH (33%)

In this study, Hetrion 922 PAW, a vinyl ester was used. The vinyl ester was dissolved in 50% by weight of styrene. Suppliers of the raw vinyl ester resins claim that shrinkage in cured vinyl esters without filler was around 5 to 6%. The engineers in the Excellence Centre for Engineered Fibre Composites (ECEFC), University of Southern Queensland (USQ) found that the shrinkage varied from 10 to 12% for their large components made from resins with 33% by weight of filler. Lubin also claimed the same amount of shrinkage for the resin with no filler but with 50% by weight of styrene (Lubin, 1982). In order to estimate the real shrinkage percentage, experiments were carried out. Two HYSIL glass beakers of 50 ml and 200 ml were employed for the experiment. The beaker thickness was 2.6 mm but its composition was unknown. The resin to accelerator ratio used in the experiment was 98% resin by volume and

| Parameters                     | Materials | Resin    | Accelerator | Flyash | 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) | —         |

Table 1 – Weight of Materials Required to Make 500 ml of VE/FLYASH (33%)

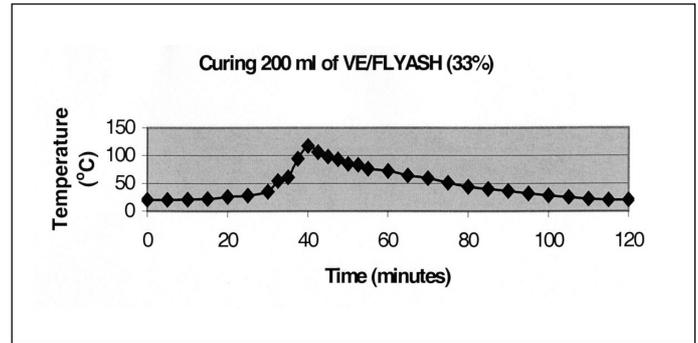


Figure 6 – Relationship between Temperature and Time in Curing 200 ml of Vinyl Ester Composite, VE/FLYASH (33%) under Ambient Conditions

2% accelerator by volume. The reinforcer was fly ash (ceramic hollow spheres) particulate made up of 44% by volume or 33% by weight in the cured vinyl ester composite. Usually a 44% by volume or 33% by weight of fly ash in a composite was considered optimum by the ECEFC group because it was considered necessary for the composite to have a reasonable fluidity for casting combined with a good tensile strength in service. The resin was a colourless liquid and it was first mixed with the red accelerator. After that the fly ash was added to the mixture and they were 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 250 ml of uncured composite (of 44% by volume of fly ash or of 33% by weight).

After mixing, 200 ml of the composite was poured into a beaker with a volume of 200 ml and the rest was poured into a 50-ml beaker. Data of temperature against time for the beakers were collected. As soon as the composite was poured in the beaker, temperature measurements were carried out in minute interval from the top of the beakers at three points around the centre. The average value of the measurements was used. Fig 6 shows the relationship between temperature against time obtained during curing of 200 ml of vinyl ester in ambient conditions. The curve is similar to that found in one of the three solid graphs in Fig 4. The inhibition time was 5 minutes. After 30 minutes, the temperature was 54°C and a crust formed on the surface. After 40 minutes, the temperature peaked and was 106°C. The temperature then began to drop. Room temperature was regained after 115 minutes. To determine the initial and final volumes of the composite in the beaker, the height of the level of VE/FLYASH (33%) was measured by a digital height gauge. The initial height was 48.24 mm, which represents a volume of 200 ml. 24 hours later, the height was re-measured and was found to be 47.19 mm. The linear shrinkage of the composite after

$$\text{curing was } \frac{48.24 \text{ mm} - 47.19 \text{ mm}}{48.24 \text{ mm}} = 0.0218.$$

The volumetric shrinkage of the composite can be expressed as (Kalpakjian, 1991):

$$V_{\text{cured}} = V_{\text{uncured}} \times \left(1 - \frac{\Delta L}{L_0}\right)^3 \quad (2)$$

$$\text{Therefore, } V_{\text{cured}} = 200 \text{ ml } (1 - 0.0218)^3 = 187.20 \text{ ml}$$

$$\text{The shrinkage is } \frac{200 \text{ ml} - 187.20 \text{ ml}}{200 \text{ ml}} \times 100\% = 6.4\%$$

Linear shrinkage was used in determining the volumetric shrinkage because the sample shrank proportionally in all directions. The shrinkage values for other volumes of composites cured are shown in Table 2. From the results of these experiments, it was found that the larger the volume of the composite, the larger the shrinkage and the higher the peak temperature would be. This is probably because of the higher curing rate encountered which is in line with the historical data kept by the ECEFC.

|                         |       |        |       |       |
|-------------------------|-------|--------|-------|-------|
| Original volume (ml)    | 600   | 400    | 200   | 50    |
| Final volume (ml)       | 535.8 | 363.94 | 187.2 | 47.44 |
| Ambient temperature     | 16    | 16     | 20    | 20    |
| Relative humidity       | 52    | 52     | 19    | 19    |
| Peak temperature (°C)   | 143   | 139    | 106   | 85    |
| Gel time (minutes)      | 60    | 65     | 32.5  | 35    |
| Percentage of shrinkage | 10.7  | 9.02   | 6.40  | 5.13  |

Table 2 – Comparison of Original and Final Volumes of VE/FLYASH (33%)

### Interaction of microwaves and VE/FLYASH (33%)

The tendency of a material to absorb microwave energy and convert it into heat depends on its relative complex permittivity and loss tangent. Ku et al 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 (Ku et al, 2001). 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 vinyl ester resins will also absorb microwaves readily (Peters, 1998; Ku et al, 1999a; 1999b). 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 used was spot welded together and was not necessarily water/air/steam proof. Styrene is a highly flammable vapour and is given off during the curing process of the composite. High vapour 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 did not have an exhaust fan and instead a blower motor inside was used to suck the air through the air filter at the front and to cool the HV transformer as the air passed through. The air from the fan was blown into a duct and cooled the magnetrons. Some air was forced into the cavity at the back and then out of the steam exhaust outlet at the back.

In another series of tests, two beakers each containing 50 ml of VE/FLYASH (33%) were exposed to microwave irradiation of 180 W and at exposure times of 35 seconds and 40 seconds respectively. The ambient temperature was 22°C but the oven cavity temperature was 25°C. The oven cavity temperature was measured by opening the oven door immediately after the power was off and an infrared thermometer was used to measure the temperatures at three points around the base of the cavity. The temperature measurements obtained after exposure to

|   |       |       |       |       |
|---|-------|-------|-------|-------|
| Microwave exposure time (seconds)           | 0     | 30    | 35    | 40    |
| Oven cavity Temperature (°C)                | 20    | 28    | 25    | 25    |
| Temperature after microwave exposure        | NA    | 44    | 40    | 45    |
| Original volume (ml)                        | 50    | 50    | 50    | 50    |
| Final volume (ml)                           | 47.36 | 48.85 | 49.64 | 51.43 |
| Volume shrinkage (%)                        | 5.13  | 2.3   | 0.72  | -0.06 |
| Volume at maximum temperature (ml)          | NA    | 51.16 | 50.00 | 51.00 |
| Time to reach gel time (minutes)            | 48    | 8     | 5     | 5     |
| Maximum temperature                         | 85    | 137   | 137   | 145   |
| Time to reach maximum temperature (minutes) | 45    | 9     | 6     | 6     |

Table 3 – Volume Shrinkage and Other Parameters for 50 ml of VE/FLYASH (33%) Exposed to 180-W Microwaves at Different Duration

microwaves for 35 seconds and 40 seconds respectively were 40°C and 45°C respectively. The shape of the graphs of the temperature against time of curing the composite using microwave energy is similar to that observed in Fig 4.

Referring to Table 3 and row 7, it can be found that the volumes at maximum temperature were equal to or higher than the original ones except for the case when the composite was not microwaved. This means that the volumes of the composite expanded after being microwaved and attained a maximum volume at the highest temperature, which was actually observed and measured. The filler neither interacted nor gave off gas in the curing process. Some styrene vapour was given off and there was very little loss in mass. From then on, the volumes of the composite shrunk and attained their final volume after 24 hours. It can be argued that the final shrinkage in volume of the composite had been compensated for by the expansion in the initial stage of the curing process.

Due to the initial expansion of the volumes of the composite during curing by microwaves, it is possible that there were pinholes left in the cured composite. To investigate this possible outcome, four composite samples cured by microwave irradiation of 180 W with different duration of exposure were sawn across and they are shown in Fig 5. The cross sections of the samples were carefully examined. The top left-hand corner sample was exposed to microwaves of 180 W for 30 seconds. Its final shrinkage in volume was 2.3%. From the appearance of the cross section, pinholes could not be found and it can be argued that the strength for the sample would be expected to be good. The top right-hand corner sample was exposed to microwaves for 35 seconds. Its final shrinkage in volume was 0.72%. Pinholes were not found and its strength was considered to be good. The bottom left-hand corner sample was exposed to microwaves for 40 seconds. Its volume expanded by 0.06%, ie virtually no change in volume. Pinholes could not be found and the strength of the sample would be expected would be expected to be good. The bottom right-hand corner sample was exposed to microwaves of 180 W for 60 seconds. Its volume expanded by 15%. Voids were everywhere in the sample. It was expected that the strength of the sample was substantially reduced. Based on visual inspection, the impact strength of the samples after microwave exposure would be expected to be good. However, drop weight impact tests were carried out to confirm this. The results were favourable and details of the test are discussed in the later section-drop weight impact test.

### Curing 200 ml of VE/FLYASH (33%) by Microwave Energy

Fig 7 shows the temperature against time of curing 200 ml of the composite using microwave energy of 180 W and an exposure time of 35 seconds. After it was microwaved for 35 seconds, the temperature of the composite was 45°C. Comparing Fig 6 and 7, it can be found that the inhibition time for Fig 6 is 5 minutes while that of Fig 7 is virtually zero, because the reaction between the inhibitor molecules and the free radicals is greatly sped up by microwave energy absorbed. The sharp increase in temperature indicated that curing took place quickly from

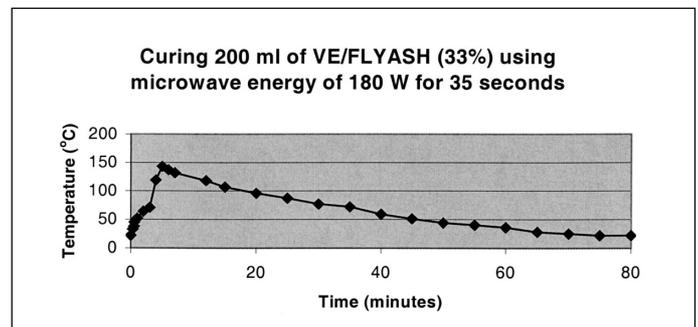


Figure 7 – The Relationship between Temperature and Time in Curing 200 ml of Vinyl Ester Composite, VE/FLYASH (33%) using Microwave Energy of 180 W and an Exposure Time of 35 Seconds

this point onwards. The gel time had been reached. For Fig 6, this occurred at 32.5 minutes while that of Fig 7 occurred at 3 minutes. The latter reached such a condition faster than the former by 11 times. For Fig 6, the peak temperature was 117°C at 37.5 minutes and that of Fig 7 was 143°C at 5 minutes. The time for the latter to reach peak temperature, after gel time, was only 2 minutes while that of the former was 5 minutes. The time required to reach peak temperature for the former was 2.5 times faster than that of the latter.

In another set of experiments, two beakers each containing 200 ml of VE/FLYASH (33%) were then exposed to microwave irradiation of 180 W for 30 and 40 seconds respectively. The results are summarised in Table 4. The values of the volume shrinkage at exposure times of 30 seconds, 35 seconds and 40 seconds are -1.16%, 0.32% and -0.03% respectively. It appears that the best microwave (180 W) exposure time for curing the 200 ml composite without shrinkage is also between 35 seconds to 40 seconds. It tends to be nearer to 40 seconds. The curing time for the ambient cured composites was 100 minutes, whereas those for the microwave-exposed composites are 70 minutes (with 30 seconds exposure time), 75 minutes (with 35 seconds exposure time) and 70 minutes (with 40 seconds exposure time) respectively. Their curing times are nearly 40% faster than the ambient cured one.

|   |        |        |        |        |
|---|--------|--------|--------|--------|
| Microwave exposure time (seconds)           | 0      | 30     | 35     | 40     |
| Oven cavity Temperature (°C)                | 20     | 28     | 25     | 25     |
| Temperature after microwave exposure        | NA     | 41     | 45     | 52     |
| Original volume (ml)                        | 200    | 200    | 200    | 200    |
| Final volume (ml)                           | 187.2  | 202.32 | 199.36 | 200.06 |
| Volume shrinkage (%)                        | 6.4    | -1.16  | 0.32   | -0.03  |
| Volume at maximum temperature (ml)          | 187.22 | 204.64 | 201.28 | 201.00 |
| Time to reach gel time (minutes)            | 32.5   | 3      | 1      | 1      |
| Maximum temperature                         | 117    | 143    | 144    | 145    |
| Time to reach maximum temperature (minutes) | 37.5   | 9      | 6      | 6      |

Table 4 – Volume Shrinkage and Other Parameters for 200 ml of VE/FLYASH (33%) Exposed to 180-W Microwaves at Different Duration

|   |        |        |        |        |
|---|--------|--------|--------|--------|
| Microwave exposure time (seconds)           | 0      | 55     | 60     | 65     |
| Oven cavity Temperature (°C)                | 16     | 24     | 22     | 22     |
| Relative humidity (%)                       | 52     | 48     | 46     | 47     |
| Temperature after microwave exposure        | NA     | 30     | 32     | 34     |
| Original volume (ml)                        | 400    | 400    | 400    | 400    |
| Final volume (ml)                           | 363.64 | 387.69 | 389.26 | 390.81 |
| Volume shrinkage (%)                        | 9.09   | 3.00   | 2.70   | 2.30   |
| Volume at maximum temperature (ml)          | 382.2  | 392.2  | 393.9  | 394.1  |
| Time to reach gel time (minutes)            | 62.5   | 7.0    | 7.0    | 6.0    |
| Maximum temperature                         | 139    | 138    | 148    | 150    |
| Time to reach maximum temperature (minutes) | 67.5   | 10.0   | 10.0   | 9.0    |

Table 5 – Volume Shrinkage and Other Parameters for 400 ml of VE/FLYASH (33%) Exposed to 180-W Microwaves at Different Duration

Referring to Table 4 and row 7, it can be found that the volumes at maximum temperature are higher than the original ones. The maximum volumes of the composites were calculated from the rise in height in the level of the samples in the beakers. This means that the volumes of the composite expanded after being microwaved and attained a maximum volume at the highest temperature. From then on, the volumes of the composite shrunk and attained their final volumes after 24 hours. It can be argued that the final shrinkage in volume of the composite had been compensated by the expansion in the initial stage of the curing process.

### Drop Weight Impact Test

The preference for drop weight impact test over the more conventional methods, eg, Charpy and Izod tests, is due to the limitations that are experienced while trying to perform impact testing on composite materials. The test enables the reproduction of conditions for which components are subjected to real life situations (Anonymous, 1993). The drop weight impact test includes the use of a falling weight which impacts the specimen. In this test, the impact striker is known as a tup which falls through a vertical guide tube that directs it to the centre of a specimen (see Fig 8). The guide tube must be perpendicular to the impact surface as stated in the American testing standards (ASTM, 1990). The energy released from the drop weight test can be expressed as:

$$E = mgh - I \tag{3}$$

where E: energy (Joules)  
m: mass of tup (kg)  
g: gravity (m/s<sup>2</sup>)  
h: height (m) and  
I: losses incurred by friction and other sources (Joules)

The loss is due to friction and was assumed to be negligible in the test.

In testing composite materials, the constant weight and varying height method has to be used because these materials are strain rate sensitive (ASTM, 1990; Cooper, 2000). Ubachs found that the mean height to

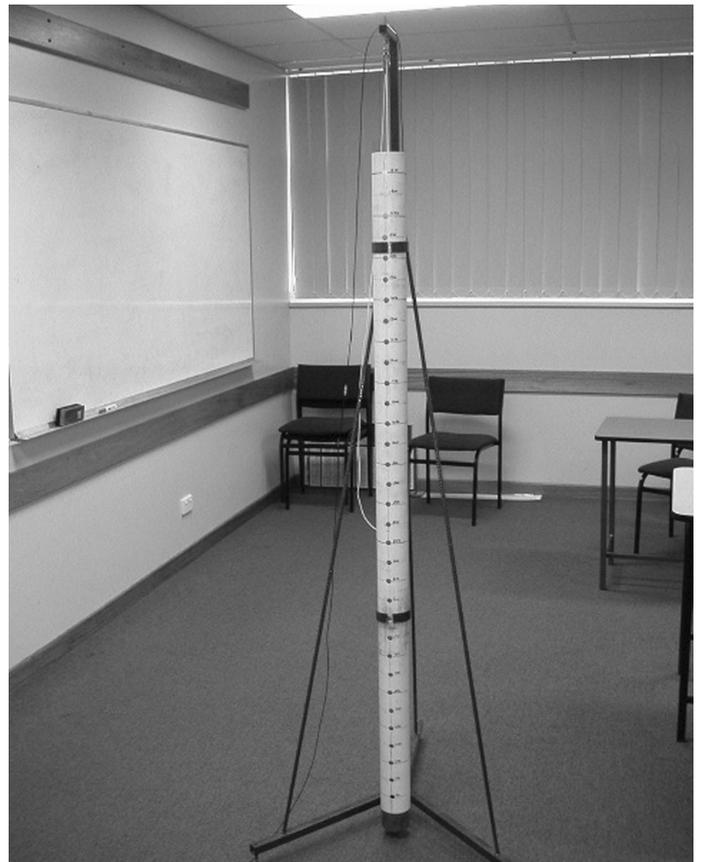


Figure 8 – Complete Drop Weight Impact Test Rig

impact the samples with 33% by weight of particle reinforcement was 900 to 1000 mm (Ubachs, 1999).

The samples tested were made by casting VE/FLYASH (33%) into PVC tubular moulds. They were then cured in ambient conditions or exposed to 180 W of microwave irradiation for different duration, eg 30 seconds. The size of the cast was set at a height of 50 mm and a diameter of 38 mm (Ubachs, 1999). The tests showed that the microwave cured samples broke when the height of the tub was 1050 mm and the ambient cured samples fractured when the height was 1060. This implied that the ambient cured ones had a higher impact strength than the microwaved counterparts. The difference in energy level required to break the microwave cured samples to ambient cured ones is provided as follows:

$$= \frac{\text{potential energy required to break microwave cured sample}}{\text{potential energy required to break ambient cured sample}}$$

$$= \frac{mgh_1}{mgh_2} = \frac{h_1}{h_2} = \frac{1050}{1060} = 99.0\%.$$

The tests showed that the potential energy required to break microwave cured samples is 99% of that required to break the ambient cured ones. This implied that the impact strength of the two groups of samples is almost the same.

## Conclusion

It was observed that the composite, VE/FLYASH (33%) could be exposed to microwave energy without arcing. By exposing the composite to microwaves for a short and critical period of time, the shrinkage can be reduced. There can even be no shrinkage at all. The ambient temperature and hence the oven cavity temperature played a vital role in the microwave processing of the composite and hence in the reduction of shrinkage. The higher the temperature, the higher the reaction rate as free radical production was stimulated by a rise in temperature and also promoted molecular mobility (Astrom, 1997). When the composite was exposed to prolonged microwave irradiation, a large amount of gases were given off, resulting in a dramatic increase in volume. The volume was more than doubled and there were pores in the cured composite. The strength of this type of composite was poor. By exposing the VE/FLYASH (33%) composites to appropriate microwave level and duration, the shrinkage in the curing of the composites can be as low as 0% while their impact strength was reduced by 1.0%. This is an acceptable value for most applications.

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19. The amount of initiator used to initiate polymerisation and the input power of microwave energy are varied to obtain an optimum combination of these parameters for minimum shrinkage of the composite components.



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