# Contrast Joints of Glass-fibre with Carbon-fibre Reinforced Low Density Polyethylene Composite Bonded by Microwave Irradiation

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**Abstract:** This paper contrasts the loss tangent, durability of reinforcement and the lap shear strengths of 33 percent by weight random glass fibre reinforced low density polyethylene matrix composite [LDPE/GF (33%)] with 33 percent by weight random carbon fibre reinforced low density polyethylene matrix composite [LDPE/CF (33%)] bonded using microwave irradiation. Fixed (2.45 GHz) and variable (2 – 18 GHz) frequency microwave (VFM) facilities are used to bond the two composites. With a given power level, the composites were exposed to various exposure times to microwave irradiation. The primer or coupling agent used for joining the glass-fibre-reinforced composite was 5-minute two-part adhesive, Araldite. No filler was used in joining the carbon-fibre-reinforced composite.

*Keywords:* variable frequency microwaves (vfm), loss tangent, 33 percent by weight glass-fibre-reinforced low density polyethylene [LDPE/GF (33%)], 33 percent by weight carbon-fibre-reinforced low density polyethylene [LDPE/CF (33%)], lap shear strength and Araldite.

## 1. Introduction

Thermosetting resins have dominated the market in the last thirty years as the matrices for fibre reinforced composites in structural applications, aerospace industry, sporting goods and chemical engineering.[1,2] Thermoplastics are almost exclusively used when no reinforcement is included and dominate also when short fibres are incorporated. However, in the last 12 - 15 years, thermoplastics have received increased attention in random as well as continuos fibre reinforced composite applications due to a number of attractive potential advantages. The advantages include ease of impregnation, faster and easier moulding cycle, no hazardous substances and better work environment. The most commonly used thermoplastics for matrix in continuos fibre reinforced composites are polypropylene (PP), nylon (PA), polyetherimide (PEI), polyphenylene sulphite (PPS), polyethersulphone (PES) and polyetheretherketone (PEEK). Traditionally, these fibre reinforced thermoplastic composites are joined by applying adhesives onto the surfaces to be joined and cured in ambient conditions. The curing process may take up to several days.[1, 2] At the same time, industrial microwave technology for processing polymers and polymer-based composites is currently in a state of considerable flux. This paper extends the applications horizon of microwaves in the area of reinforced thermoplastic composites joining, aiming at maximising bond quality.

Factors that hinder the use of microwaves in materials processing are declining, so the prospects for the development of this technology seem to be very promising.[3] The mechanisms that govern the energy distribution process during microwave joining of materials include dipole friction, current loss and ion jump relaxation. This results in a relatively uniform heat distribution throughout the entire exposure to microwave irradiation, immediately in front of rectangular or circular waveguides.[4-8] The material properties of greatest importance to microwave processing of a dielectric are the complex relative permittivity $\varepsilon = \varepsilon' - j\varepsilon''$ and the loss tangent, $\tan \delta = \frac{\varepsilon''}{\varepsilon'}$.[6, 9] The real part of the permittivity, $\varepsilon'$, sometimes called the dielectric constant, mostly determines how much of the incident energy is reflected at the air-sample interface, and how much is absorbed. The most important property in microwave processing is the loss tangent, $\tan \delta$, which predicts the ability of the material to convert the absorbed 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 δ, to convert microwave energy into thermal energy. In a material with a very high loss tangent, the microwave energy density will reduce with distance of penetration into the material. This phenomenon is known as the skin effect. The fast heating rate encountered using microwave energy can thus lead to reduced processing time and consequent energy efficiency. These advantages have encouraged the development of facilities for joining a range of thermoplastic composites autogenously and heterogeneously. In the heterogeneous mode, at room temperature, transparent materials, including a range of thermoplastic and thermosetting resins can be bonded using two part adhesives cured at fast rates when exposed to focused microwave irradiation.[5, 7, 10]

## 2. Thermoplastics as matrices in composites

Issues that are of importance when selecting a polymer for use as a composite matrix are reinforcement-matrix compatibility in terms of bonding, mechanical properties, thermal properties, cost, etc., though perhaps the most important aspect may be its processability, ie, how easy it is to deal with it in manufacturing situations. Among the many issues that may be considered part of the processability are viscosity, processing temperature, processing time and health concerns. Low viscosity is vital in achieving reinforcement impregnation, where each reinforcing fibre should be surrounded by the matrix without voids. While thermoplastics only need to be melted, shaped, and then cooled to achieve dimensional stability in a matter of seconds at one extreme, thermosets may take several days to fully crosslink the polymer. In contrast, the molecular structure of thermoplastics makes them chemically inert if processed correctly, meaning that no hazardous substance need to be considered. On the other hand, the molten thermoplastic and the heated machinery may cause severe burns.[1]

While thermosets heavily dominate as matrices in structural composite applications for reasons of good mechanical and thermal properties, low cost, and low viscosity to mention a few, the interest in thermoplastics is driven by several potential advantages. Among the prime reasons behind the increased interest in the usage of thermoplastic matrices are advantages in areas as toughness, processing time, recyclability, and work environment. In general, a high-performance thermoplastic will outdo a standard-performance thermoset in most respects except cost and vice versa.

A thermoplastic is usually fully polymerised when delivered from the supplier, meaning that all chemical reactions are complete and the user can concentrate entirely on physical phenomena, such as heat transfer and flow. However, there are some rare exceptions to this rule. The user may choose to take care of part of the polymerisation starting off with low molecular weight prepolymer, thus avoiding the high viscosity disadvantage during reinforcement impregnation. Courtesy of the low molecular weight, the polymer fluid may have a viscosity comparable to that of a thermoset resin. After the reinforcement is impregnated, the final polymerisation process takes place and the molecular weight thus drastically increases.

One of the main features of amorphous thermoplastics is that they are dissolvable in common industrial solvents. This means that the reinforcement can be impregnated with a low viscosity solution, thus avoiding the problem of high melt viscosity, but it also means that the solidified polymer is not solvent resistant. For solvent-impregnated reinforcement, the residue solvent that was not completely driven off after impregnation is a serious concern since it impairs the quality of the composite. Amorphous thermoplastics have very good surface finish since they do not shrink much when they solidify and there is no differential shrinkage from the presence of crystalline regions .[1] This is in fact a very important property of thermoplastics used as matrices of composites.

In this research, low-density polyethylene was chosen as the thermoplastic matrix for the composites because of its wide applications and acceptance. Polyethylene is probably the most commonly polymer found in our daily life. It is the most popular plastic in the world. It is a large global business segment.[11, 12] Low-density polyethylene was chosen in place of the high density one because of its better interaction with microwave energy due to its amorphous structure.[9]

## 3. Materials / microwaves interactions

In conventional microwave processing, microwave energy is launched at a fixed frequency of either 915 MHz or 2.45 GHz or 5.8 GHz or 24.125 GHz into a waveguide or cavity and it brought with it the inherent heating uniformity problems like hot spots and thermal runaway.[9-10, 13] Thermal runaway is the uncontrolled rise in temperature in some hotter parts of a material subject to microwave heating. This is because the hotter parts will absorb more microwave energy than any other part of the material and convert it into heat.[4] A US based company developed a new technique for microwave processing, known as variable frequency microwave (VFM) technique, to solve the problems brought about by fixed frequency microwave processing. The technique was geared towards advanced materials processing and chemical synthesis. It offered rapid, uniform and selective heating over a large volume at a high energy coupling efficiency. This was accomplished using preselected bandwidth sweeping around a central frequency employing tunable sources such as travelling wave tubes as the microwave power amplifier. Selective heating of complex samples and industrial scale-up were now viable.[13, 14] Successful applications have been reported in the areas of curing advanced polymeric encapsulants, rapid processing of flip-chip underfills, materials characterisation, curing profiles for various adhesives, structural bonding of glass to plastic housing.[14, 15]

When microwave energy of a fixed frequency, eg 2.45 GHz was launched into a waveguide eg WR340, as depicted in figure 1(a), containing a piece of material, some areas of the material would experience higher electric field strength than the others; the situation would even be more serious if the microwave energy was launched into a multimode cavity because many resonant modes will be established. Figure 1(b) shows the fixed electric field pattern across any cross section of the joint of the test pieces during fixed frequency heating. Those areas with higher electric field strength would be heated more, creating hot spots, which could even lead to thermal runaway. With variable frequency microwave heating, as shown in figure 2(a), more than one thousand frequencies were launched into the cavity sequentially.[14] Each incident frequency set up its own electric field pattern across any cross section of the joint of the test pieces, and therefore resulted in hot spots at different locations at different time, as shown in figure 2 (b). Different areas were heated under different frequencies at different times. When a sufficient bandwidth was used, every element of the test piece would experience hot spots at one or more frequencies during sweeping. Therefore, time-averaged uniform heating could be achieved with proper adjustment of the frequency sweep rate and sweep range. Another advantage of the VFM heating is the capability of providing precise frequency tuning to optimise the coupling efficiency.

## 4. Fixed frequency microwave processing of materials

In the fixed frequency microwave facility configuration, a focus, high-energy rate, fixed frequency (2.45 GHz) equipment, as shown in Figure 3 is selected. Two halves of lap shear test piece of the sample as shown in Figure 4 were joined together using microwave energy with Araldite as primer for glass-fibre-reinforced composite. No primer is used in joining the carbon-fibre-reinforced composite. The primer used, which was microwave reactive, was two part adhesive containing 100% liquid epoxy and 8% amine.[9] The lapped area for the joint was 10 mm x 20 mm. The bond surfaces were first roughened with coarse, grade 80 emery paper. The roughened surfaces were then cleaned and degreased by immersing them in methanol. After drying, five-minute two-part Araldite of around 1.0 to 1.5 cubic centimetres was applied to the two roughened surfaces to increase the mechanical keying or interlocking.[16] The two test pieces were then brought together and the total pressure applied was about 4 $N/cm^2$.

The fixed frequency equipment involves the use of a $TE_{10}$ mode rectangular waveguide operating in a standing wave configuration. Slots were machined in the waveguide allowing the adhesive layer on the specimens to pass through the microwave region. LDPE/GF (33%) or LDPE/CF (33%) composite specimens with the same lap area and surface treatment were placed in a standard rectangular waveguide as depicted in Figure 5.[13, 17] To avoid microwave radiation leakage, the slotted waveguide was enclosed in a modified commercial microwave oven case (Figure 3). One to one and a half millilitre of Araldite were smeared on both surfaces of the lapped area. A short circuit was adjusted to ensure that the maximum of the standing wave coincided with the lapped area of the specimen.[9, 13] The input power to the system was in a step function and could only be 240W, 400W, 640W and 800W. The power was changed by altering the power of the source. The duration of exposure could be increased in steps of 1 second. The apparatus used in the fixed frequency processing has been described in other papers and will not be discussed here.[6,7] For LDPE/GF (33%), the samples were exposed to 400 W and 800 W of power at different exposure times. For LDPE/CF (33%), the samples were exposed to 100W, 400 W and 800 W of power at different exposure times. The 100W of power was achieved by setting the variable frequency microwave (VFM) facility to a fixed frequency of 2.5 (the machine can be set only to the nearest 0.1 GHz) GHz. The results will be discussed in the later section.

## 5. VFM processing of materials

The VFMF used consist of a Microcure 2100 Model 250 with a frequency sweep range of 2 - 8 GHz operating at a nominal power of 250W, and of a Wari-Wave VW1500 with a frequency range of 6.5 – 18 GHz at a nominal power of 125 W. The VFM facilities consist of a curing cavity and an oven control system, which is linked to a PC for program input. The dimensions of the cavity for Microcure 2100 Model 2500 are 300 mm x 275 mm x 375 mm. Program with the required parameters was then written and input to control the VFMF via a PC. In one of the VFMF, Microcure 2100 Model 250, the input power level could be varied in steps of 10 W, starting from 50 W to 250W. During cavity characterisation, the actual amount of power that passed through the test pieces with respect to time was measured using fibre optic; in addition, the power reflected back from the material could also be detected. By this way, the best frequency range to process a material by microwaves can be found.

### 5.1 Program for LDPE/GF (33%)

Microcure 2100 model 250 VFM oven has to be used for bonding LDPE/GF (33%). The frequency range for this equipment is from 2 to 8 GHz but the best frequency range to process LDPE/GF (33%) is from 9.0 – 12.5 GHz as shown in Table 1.[18] It is therefore necessary to make a compromise and a central frequency of 7.45 GHz has to be selected. Since the bandwidth of the sweep should be greater or equal to 1.1 GHz, the selected bandwidth was 1.1 GHz. [19] The actual start and stop frequencies would be centre frequency $\pm \frac{bandwidth}{2}$, ie the sweep would be from 6.9 GHz to 8.0 GHz. Because the sweep time could range from 0.1 second to 100 seconds, the chosen sweep time was 0.1 second. Since the material loss tangent was relatively low, a power level of 200 W was selected.[20, 21] The processing temperature was set at 95$^{O}$C with a deadband (precision) of 1$^{O}$C and the longest processing time was set at 7 minutes. The maximum permitted temperature was set at 100$^{O}$C, above that the machine was switched off automatically. A maximum temperature of 100 $^{o}$C was selected because it was very near to the melting point of one of the main constituents of the composite, the low-density polyethylene (LDPE). The reason for setting this maximum temperature was to avoid excessive temperature rise, which forms hot spots and thermal runaway. The primer used was Araldite. Programs for other duration of exposure were also written. Results of the process will be given in the result section later on.

### 5.2 Program for LDPE/CF (33%)

In this study, Wari-Wave VW 1500 has to be used because of the frequency range chosen. The frequency range for this equipment is from 6.5 – 18 GHz and the best frequency range to process LDPE/CF (33%) is 8.5 – 9.0 GHz and 10.7 – 12.0 GHz as shown in Table 1.[18] The parameters for joining this material are central frequency = 9 GHz, bandwidth = 1.5 GHz, power level = 100 W, set temperature = 95 $^{o}$C and maximum temperature = 100 $^{o}$C. Since the bandwidth of the sweep should be greater or equal to 1.1 GHz, the selected bandwidth was 1.5 GHz.[19] The actual start and stop frequencies would be centre frequency $\pm \frac{bandwidth}{2}$, ie the sweep would be from 8.25 GHz to 9.75 GHz. The chosen sweep time was 0.1 second. Since the material loss tangent was relatively high, a power level of 100 W was selected.[20, 21]

## 6. Results

### 6.1 Results by using fixed frequency facility

#### 6.1.1 Fixed frequency results for LDPE/GF (33%)

A Shimadzu tensile testing machine was used for the lap shear test. A load range of 2000 N and a load rate of 600 N per minute were used for the test.[21] Figure 6 shows the lap shear of LDPE/GF (33%) joined by a fixed frequency microwave facility in a slotted rectangular waveguide. At the fixed frequency of 2.45 GHz and a power level of 800 W, and at microwave exposure times ranging from 25 to 40 seconds, the cluster of bond strengths was best represented by their average value of 151 N/cm$^2$ (line 800PE1 in Figure 6); while those resulting from microwave energy exposure in the range of 45 to 65 seconds were represented by their average value of 219 N/cm$^2$ (line 800PE2 in Figure 6).[6, 7, 10, 16] In both cases, the results obtained were similar to the work of another researcher using high-density polyethylene.[17] A step change in behaviour was also noted but the reasons for it have to be explored through a more thorough study of the material characteristics and properties.

At shorter exposure times, the recorded average lap shear strength was only 97% of that obtained by curing in ambient conditions and it could be argued that no surface melting of the adherend and hence diffusion of parent material to the adhesive had not taken place.[6, 18] When longer exposure times were used, the average lap shear strength was found to be 41% higher than those obtained by curing in ambient conditions. The processing times were also merely 1% and 1.5%, respectively, of the ambient cured ones. At a power level of 400 W, the cluster of lap shear bond strengths, obtained by exposing to fixed frequency microwaves from 135 to 240 seconds, were best represented by their average value of 185 N/cm$^2$ (line 400PE1) as depicted in Figure 6. It was 18% higher than that obtained by curing in ambient conditions and the processing time was only 5.0% of its counterpart.

### 6.1.2 Fixed frequency results for LDPE/CF (33%)

With reference to Figure 7 and the power level of 240 W, it was found that the peak bond strength of LDPE/CF (33%) was 299 N (tensile strength = $\frac{force}{area} = \frac{299N}{10mmx3mm} = 9.97N/mm^2$) and was obtained at 10 seconds of exposure.[7] Failure occurred at the parent material. The strength was very low as compared with the strength of parent material, 878 N (tensile strength = 29.3 N/mm$^2$). The parent material might have been damaged by excessive absorption of microwave energy. Consider the graph with the power level of 400 W, it was again found that the peak bond strength was 257 N (tensile strength = 8.57 N/mm$^2$) and the time of exposure was 7 seconds. The failure was at the parent material. Again, the strength of the parent material was weakened by excessive exposure to microwave energy.

Now consider joining of LDPE/CF (33%) using VFM at a fixed frequency of 2.5 GHz. Looking at Figure 8, it can be found that the peak bond strength is 432 N (tensile strength = $\frac{432N}{10mm x 3mm} = 14.4$ N/mm$^2$) at an exposure time of 18 seconds, which was only 49.1% of the tensile strength of the material (tensile strength = 29.3 N/mm$^2$). Failures for the first two points were at the bondline and failures for the last two points were at the parent material. In the first two cases, incomplete bonding gave rise to weaker bonds.[7, 16] In the last two cases, the parent material was weakened by excessive exposure of microwave energy.[22] When the exposure times were made above 20 seconds, arcing of graphite took place and the test pieces were deformed.

## 6.2 Results of processing using VFMF

### 6.2.1 LDPE/GF (33%) processing results using VFMF

With VFM facility and Araldite, no bond was formed if the processing time was less than 150 seconds. Bonds started to form at an exposure time of 180 seconds. At an exposure time of 450 seconds or over, the parent material was weakened because when it was subjected to lap shear test, failure occurred at the parent material. Figure 9 shows that lap shear strengths obtained range from 187 N/cm$^2$ at an exposure time of 180 seconds to 265 N/cm$^2$ at an exposure time of 420 seconds. The average lap shear strength of this material with the Araldite cured under ambient conditions is 156 N/cm$^2$, which is very low but is reasonable because Selleys (undated) pointed out that Araldite is not suitable for joining low-density polyethylene (LDPE).[23] In this study, Araldite was therefore intentionally used to join LDPE matrix composite to investigate whether microwave energy would improve the lap shear strength of the joint. The peak lap shear strength obtained by using VFMF is 70% higher than the average lap shear strength obtained by curing it in ambient conditions. The time required to achieve the required strength has, however, been reduced to 0.5 % only. At an exposure time of 420 seconds, the test piece fails at the parent material, which has strength of 1423 N (tensile strength = 47.43 N/mm$^2$). This implies that the lap shear strength was more than the peak lap shear strength of 265 N/cm$^2$.

### 6.2.2 LDPE/CF (33%) processing results using VFMF

With VFM, no Araldite was used and no bond was formed if the processing time was less than 40 seconds. Bonds started to form at an exposure time of 40 seconds or over. At an exposure time of 90 seconds or over, the parent material was weakened because when it was subjected to a lap shear test, failure occurred at the parent material and the bond quality was poor and was discarded. Figure 10 shows that lap shear strengths obtained range from 180 N/cm$^2$ at an exposure time of 40 seconds to 230 N/cm$^2$ at an exposure time of 80 seconds. At an exposure time of 80 seconds, the test piece fails at the parent material. This means that the lap shear strength was more than 230 N/cm$^2$. This also implies that the parent material [LDPE/CF (33%)] was weakened by the excessive exposure to microwave irradiation. This behaviour is similar to that of LDPE/GF (33%) and PS/GF (33%) in other study when the composite materials were exposed to excessive microwave irradiation.[22] In general, the lap shear strengths obtained using VFM facility was higher than its counterpart because VFM facility has a multi-mode cavity, whereas the focused rectangular waveguide configuration has a single ($TE_{10}$) mode cavity operating in a standing wave. The samples in the VFM facility were exposed to microwave irradiation more evenly. On the other hand, the samples in the focused rectangular waveguide configuration were directly irradiated by microwave energy and greater harm was done to the carbon fibres of the composite.

## 7 Discussion

Referring to Figures 6 through 10, the average lap shear strengths of the two composites at different powel levels, using fixed and variable frequency microwave facilities are summarised in Table 2. Comparing the lap shear strengths of the two types of materials, procured by exposing the test pieces to a power level of 400 W at fixed frequency of 2.45 GHz, at different times, it was found that the average lap shear strengths of LDPE/GF (33%) and LDPE/CF (33%) were 19% and –37% respectively higher (or lower) than those cured in ambient conditions. It is found that LDPE/GF (33%) benefits if bonded at a power level of 400W. LDPE/CF (33%) has a negative impact because the composite was damaged by excessive microwave dosage.[22]

From Table 2, it is found that LDPE/GF (33%) is best joined by fixed frequency facility and up to a certain limit, the higher the power level used, the higher the lap shear strength is. Araldite must also be used as the binding agent to absorb the microwave energy, otherwise no bond will form. This is due to its relatively low loss tangent of the material. The microwave energy is then converted to heat to melt the parent material. On the other hand, LDPE/CF (33%) is best bonded by VFM facility. When, fixed frequency facility is used, up to a certain limit, the lower the power level, the higher the lap shear strength is (see Table 2). In both cases, no Araldite is required, as the loss of the material is high and can absorb the microwaves by itself.

The next thing worth discussing will be the lap shear strengths of the two materials obtained by processing them with VFMF. Both materials were bonded at a frequency range most suitable to process them (see Table 1).[18] The power used for LDPE/GF (33%) was 200 W because its loss tangent is relatively low. On the other hand, the power used for LDPE/CF (33%) was 100 W. Referring to Figures 9 and 10, the average lap shear strengths for LDPE/GF (33%) and LDPE/CF (33%) are 190 N/cm$^2$, and 196 N/cm$^2$ respectively. They are 22% and 26% higher than the average lap shear strengths cured under ambient conditions respectively. It is found that the improvement of lap shear strength for both materials joined by using VFMF was not much and it was low but it confirmed that microwave irradiation did improve the joint strength.

The peak lap shear strengths for bonding LDPE/GF (33%) and LDPE/CF using VFM facilities were 235 N/cm$^2$ and 230 N/cm$^2$ respectively. The difference was minimal. On the other hand, when the duration of exposure is concerned, it is found that the exposure times required for LDPE/GF (33%) and LDPE/CF (33%) to get into their peak lap shear strength are 420 seconds and 80 seconds respectively. The latter is much shorter and hence the energy required to bring the two materials to their peak lap shear strengths is significantly different. The saving in power was $\frac{200W}{100W}$ = 2 times; the saving in time was $\frac{420 \sec ond}{80 \sec onds}$ = 5.25 times. Therefore the energy saving is 2 x 5.25 x 100% = 1050 %. This is entirely due to the much higher loss tangent for LDPE/CF (33%), which has high loss tangent carbon reinforcement. On the other hand, the loss tangent of glass fibre is very low, 0.53 x $10^{-4}$.[4, 24] The thermoplastic matrix in the composites are the same and need not be taken into consideration while comparing the dielectric properties because each composite has the same percentage by weight of LDPE. By and large, the VFMF are more superior than their fixed frequency counterpart in joining and processing materials.

## 8. Conclusion

LDPE/CF (33%) absorbs more microwave energy than its counterpart because the loss tangent of the former is much higher than that of the latter. On the other hand, carbon fibre reinforcement in LDPE/CF (33%) may absorb excessive microwave irradiation during bonding; this will weaken the strength of the parent material. This phenomenon will not happen in LDPE/GF (33%). The lap shear strengths of LDPE/GF (33%) obtained by fixed frequency microwave bonding are better than those of LDPE/CF (33%)

procured by the same facility. In VFM joining, the lap shear strengths of both materials are more or less the same but the energy and time saving from the bonding of LDPE/CF (33%) are large. Saving in the Araldite will also be significant.

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