Application of Variable Frequency Microwave (VFM) to Adhesives

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ABSTRACT

Microwave processing of adhesives is a relatively new technology alternative that provides new approaches for enhancing material properties as well as economic advantages through energy savings and accelerated product development. Alternative in the sense that most adhesives are normally cured in ambient conditions or in ovens. However, the most commonly used facilities for microwave processing of materials operate on fixed frequency microwaves (FFM), e.g., 2.45 GHz. This paper presents a review of microwave technologies, processing methods and industrial applications, using variable frequency microwave (VFM) facilities. The technique offers rapid, uniform and selective heating over a large volume at a high energy coupling efficiency. This is accomplished using a preselected bandwidth sweeping around a central frequency by employing tunable frequency sources. Successful applications of these modern facilities include finding out the optimum cavity conditions of glass or carbon fibre reinforced thermoplastic matrix composites, and of adhesives, e.g., two-part five-minute Araldite, and the joining of the above-mentioned composite materials with, or without, primers. Finding out the optimum cavity conditions of a material has helped identify the best frequency range to process the material using microwave energy and by means of the VFM facility. Microwave energy has been used to rapidly cure several types of two-part epoxy based adhesives, e.g., Araldite. Bond strengths obtained using variable frequency microwave (VFM) techniques are compared with adhesive joints cured in fixed frequency microwave (FFM) conditions.

Keywords: microwaves, variable frequency microwaves, curing adhesives, Araldite and lap shear bond strength.

1. INTRODUCTION

Over the last twenty years, or so, there has been a rapid growth in the use of adhesives, particularly in structural and engineering applications. Rapid and major developments in the technology of adhesives have been reported. This growth has also led to focus on somewhat more basic studies of the science of adhesion and technology of adhesives. Such studies have been aided greatly by the availability of tools at the disposal of the researchers, for example, X-ray photoelectron spectroscopy (XPS), secondary-ion mass spectroscopy (SIMS), and scanning electron microscopy (SEM) [1]. This paper presents techniques for curing adhesives used in structural and engineering applications utilising variable frequency microwave (VFM) facilities.

The factors that hinder the use of microwaves in materials processing are declining, so that prospects for the development of this technology seem to be very promising [2]. The two mechanisms of orientation polarisation and interfacial space charge polarisation, together with D.C. conductivity, form the basis of high frequency heating [3, 4]. Clearly, advantages in utilising microwave technologies for curing adhesives include better penetrating radiation, controlled electric field distribution, and selective and volumetric heating. However, the most commonly used facilities for microwave processing of materials and curing of adhesives use a fixed frequency, e.g., 2.45 GHz. Variable frequency microwave (VFM) facilities present a new alternative for microwave processing. The technique is most applicable to advanced materials processing and chemical synthesis. It offers rapid, uniform and selective heating over a large volume and at a high energy coupling efficiency. This is accomplished using a preselected bandwidth sweeping around a central frequency by employing tunable frequency sources such as travelling wave tubes as the microwave power amplifier. Selective heating of complex samples and industrial scale-up are now possible. Typically, during VFM processing, a particular frequency of microwaves would be generated only for less than one millisecond.

Two such facilities were available during this project. One, Microcure 2100 Model 250 (Figure 1) with a maximum power output of 250W generates microwave energy in the frequency range of 2 to 7 GHz and the other, VW1500 (Figure 2), operates in the range of 6 to 18 GHz with a maximum power level of 125W. The cavity dimension of VW1500 is 250 mm x 250 mm x 300 mm (width x height x length); while, Microcure 2100 Model 250 has a cavity size of 300 mm x 275 mm x 375 mm (width x height x length).

Surret et al. [5] showed that sweep frequency heating produced more uniform curing in thermosetting composite systems (carbon fibre/epoxy resin laminates). Fathi et al., [6] while investigating the heating of thermoset polymer matrix composite materials, showed that thermal runaway and hot spots problems associated with a fixed frequency heating were overcome. Qui et al. [7] showed that frequency switching enabled modes with high heating rates and desirable heating patterns to be selected to allow uniform heating of graphite epoxy load. Ku et al. [8-11] successfully joined thermoplastic matrix composite materials without causing thermal runaway and hot spots. Advanced polymeric adhesives and encapsulants were cured and a rapid processing of flip-chip (FC) was also carried out, using a VFM facility [12]. Structural bonding of glass to plastic housing was also carried out [13]. Wei et al. [14] have looked into variable frequency heating for bonding applications in polymer composites and electronic packaging. Ku et al. [15] performed the finding of optimum cavity conditions of adhesive successfully.

There are many factors that have to be considered before employing a variable frequency microwave (VFM) irradiation for processing materials. Not all materials are suitable for microwave processing and one has to match the special characteristics of the process with the physical and chemical properties of the materials being processed. Blind applications of microwave energy in material processing will usually lead to disappointment. On the other hand, smart applications of the technology can yield greater benefits than has been anticipated [16-17].

Successful applications of these modern facilities in this work include the finding of optimum cavity conditions of glass or carbon fibre reinforced thermoplastic matrix composites, e.g., 33% by weight glass fibre reinforced low-density polyethylene [LDPE/GF (33%)]. Characterisation of primers, e.g., two-part five-minute Araldite, and joining of the above-mentioned thermoplastic composite materials with, or without, adhesives was also carried out [11, 18-19].

2. VARIABLE FREQUENCY MICROWAVES

The idea of variable frequency microwave (VFM) facility was first conceptualised in 1979 and it was not until 1992 that the first VFM processing system was designed and built using a high power travelling wave tube (TWT) amplifier capable of supplying up to 2.5 kW power over the frequency range of 4 to 8 GHz [20-21]. The frequency range can be extended by the addition of other TWTs.

Microwave-based processing of materials can be broadly divided into either single-mode or multimode cavities. The single mode cavity approach makes use of a tunable microwave capacity especifically designed to support a single resonant mode at the frequency of the microwave source. This ensures maximum coupling of the microwave energy to the load. However, the single mode nature of the cavity limits the area of high electric field intensity and, thus, the size, shape and positioning of the material to be processed. The multimode cavity approach makes use of a cavity that is overmoded, which means it is large enough to support a number of high-order modes, often at the same frequency. However, the power distribution at a single frequency is uneven and can result in multiple hot spots [22].

The VFM uses a TWT high power, broadband amplifier to sweep a range of frequencies of approximately an octave in bandwidth. The concept behind this approach is that continuous sweeping through several cavity modes within a period of a few milliseconds results in time-averaged uniformity of heating throughout the load. The resulting relative power distribution in a plane with a fixed frequency (2.45 GHz) heating is not uniform. There is no mode control and hence the coupling efficiency is uncontrolled. Moreover, there is limited scaleup and high potential for hot spots and thermal runaway. On the other hand, the resulting relative power distribution in a similar plane with VFM heating is uniform. There is a selective frequency control, high-energy coupling efficiency, scalability to large processing volumes and uniform heating throughout [23].

3. FINDING THE OPTIMUM CAVITY CONDITIONS OF ADHESIVE USING VARIABLE FREQUENCY MICROWAVES (VFM)

The adhesive selected for the above process and later for joining the LDPE/GF (33%) composite was five-minute two-part adhesive containing 100% liquid epoxy and 8% amine, which is microwave reactive and has a trade name of Araldite supplied by Selleys Chemical Company Pty. Limited of New Zealand [8-9, 16]. The characterisation option of the VFM facilities was used to measure the characteristics of the cavity when a sample was loaded. The procedure followed was a sequence of operations whereby the user graphically sees how the cavity, with material loaded, would operate over the selected frequency range. The input power is selected on the basis of the measured loss tangent of the material [11, 24-5]. The higher the estimated loss, the lower the power level to be selected.

During characterisation of the cavity loaded with Araldite, temperature variations were obtained as well as incident power and reflected power levels from the cavity containing the sample via a monitor. The incident and reflected power levels versus frequency together with the percentage of reflectance against frequencies were monitored and recorded. From these data, the bandwidth values most suitable for microwave processing were chosen. The operation bandwidth for Microcure 2100 Model 250 is from 2 GHz to 8 GHz. To find the optimum cavity conditions for the two-part five-minute rapid Araldite a power of 50 W was selected and the maximum temperature reached was 100^{o}C. The percent reflectance against frequency for rapid Araldite is shown in Figure 3 [11]. The reflectance is the ratio of the reflected power to the incident power. The lowest percent reflectance for the Araldite was from 6.5

GH to 8 GHz and so the best frequency range to process this adhesive was found to be from 6.5 to 8 GHz.

4. JOINING OF LDPE/GF (33%) COMPOSITE WITH ARALDITE USING VARIABLE FREQUENCY MICROWAVES (VFM)

The two mirror image test pieces of LDPE/GF (33%) composite were cut from a standard tensile test piece (Figure 4) for composite materials [8,9]. Lap joint was selected for joining the two tensile test pieces. The lapped area was 20 mm x 10 mm. The lapped areas were first roughened by rubbing them with coarse, grade 80, emery paper. They were then cleaned in methanol solution and allowed to dry in air before applying the adhesive onto them. After applying the Araldite (1 to 1.5 ml), the two pieces were pressure-bound using a dielectric (rubber) band (Figure 4). This fixed the relative position between the two test pieces and pressure was applied onto the lap joint. The pressure on the lap joint was estimated to be 4 N. The LDPE/GF (33%) composite test pieces were then joined using VFM by placing them in the cavity of the facility. 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 selected for the test [26].

The best frequency to process Araldite using Microcure 2100 with a frequency range between 2 GHz to 8 GHz was from 6.5 GHz to 8 GHz [11,18]. Since the material was processed with a variable frequency sweep, it was necessary to find out the centre frequency for the sweep. Since the best processing range for the adhesive was from 6.5 GHz to 8GHz, the centre frequency sweep was 7.25 GHz and the bandwidth of sweep was greater than 1.0 GHz [27-8]. The sweep time ranged from 0.1 second to

100 seconds and the chosen sweep time was 0.1 second [27]. Since the material loss tangent was low, a power level of 200 W was selected [24-5, 29]. For the sake of tensile shear tests, several sets of test pieces were joined at different durations and details are given later.

5. JOINING OF LDPE/GF (33%) COMPOSITE WITH ARALDITE USING FIXED FREQUENCY MICROWAVES (FFM)

To discuss the processing of adhesives using variable frequency microwaves without mentioning their curing using its fixed frequency counterpart will be incomplete. Two cases of curing Araldite using fixed frequency microwaves are discussed here. The first one involved 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%) composite specimens with the same lap area and surface treatment were placed in a standard rectangular waveguide as depicted in Figure 5 [8, 30]. To avoid microwave radiation leakage, the slotted waveguide was enclosed in a modified commercial microwave oven case (Figure 6). 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 [8, 9]. The samples were exposed to 400 W and 800 W of power at different exposure times. The magnetron was operating at 2.45 GHz. The bonds formed were lap shear tested and the results are outlined later.

The other microwave technique utilised domestic microwave oven (Menumaster Model 3100i). The power selected for the experiment was 1 kW and the operating frequency was again fixed at 2.45 GHz. The lapped area in this case was 10 mm x 10 mm. All the specimens received the same surface treatment as in the former two cases. Again the same amount of Araldite (1 to 1.5 ml) was smeared on the lapped area before the specimens were placed into the cavity of the commercial microwave oven.

6. EXPERIMENTAL RESULTS

6.1 Curing of Adhesives by VFM

With the VFM, no bond was formed, if the exposure time was less than 150 seconds. Bonds started to form at a joining time of 180 seconds. At an exposure time of 450 seconds or more, the parent material was weakened because when it was subjected to tensile shear stress test, the failure occurred in the parent material [31]. Figure 7 shows that the lap shear bond strengths had the same characteristics as in the case of fixed frequency facilities, i.e., the bond strength did not improve much with increasing duration of microwave radiation. The average lap shear bond strength obtained was 189 N/cm^2. This meant that its strength was only 21.5% higher than those cured in ambient conditions [11]. Most of the failures were in the bondline. In addition, the VFM needed longer time to join the materials. This was due to the low maximum power output of 250 W used.

6.2 Curing of Adhesives by FFM (2.45 GHz)

Figure 8 shows the bond strength of LDPE/GF (33%) composite 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 8); 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 8 [8-11]. In both cases, the results obtained were similar to the work of another researcher using high density polyethylene [30]. 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 bond 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 [8,10]. When longer exposure times were used, the average lap shear bond strength was found to be 41% higher. 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 8. It was 18% higher than that obtained by curing in ambient conditions and the processing time was only 5.0% of its counterpart.

Figure 9 shows that the lap shear bond strength of HDPE joined by Araldite and cured in ambient conditions. The tensile shear strength of HDPE is 130 N/cm^2 [30, 32]. The bond strength was up to 95% of that of the parent material after 60 minutes and only marginally improved after this time [33]. Figure 10 shows the lap shear bond strengths of HDPE joined by 2.45 GHz commercial microwave oven using Araldite. It was found that the bond strength was above 90% of that of the parent material and remarkably consistent with, and very close to, the maximum lap shear bond strength of the HDPE specimens that were cured conventionally. The difference was in processing time; with microwave radiation the time taken to achieve 95% of the parent material strength was 325 seconds; while the time taken by curing the adhesive in ambient conditions was 60 minutes, which is 11 times longer than its counterpart. In terms of manufacturing time, it is shown that microwave processing of Araldite is superior to that cured in ambient conditions.

7. CONCLUSION

Finding the optimum cavity conditions of a material using VFM facility is the first step in microwave curing or processing of materials. Whether, fixed or variable microwave frequency is used, finding the optimum cavity conditions of a material is a 'must' because it identifies the exact frequency range within which the maximum coupling of the microwave energy into the load can be obtained. It is a preferred way of using microwave energy because without it time, effort and money would be spent unwisely.

Using a fixed frequency for joining of materials using a commercial microwave oven and Araldite as adhesive, it was found that the lap shear bond strength achieved was almost the same as that obtained when cured in ambient conditions. In both cases, increasing the time for curing did not significantly increase the lap shear bond strength. The only advantage of the application of microwave radiation in this case was processing time, which was eleven times less than its counterpart. On the other hand, when focused microwave energy in a slotted rectangular waveguide for joining materials was used, the advantage was more than faster production time. With a power level of 400W (line 400PE1 in Figure 8), the lap shear bond strength is 18% higher than it parent material counterpart. With a power level of 800W and a longer exposure time, the lap shear bond strength (line 800PE2 in Figure 8) is 41% higher than that of the parent material. The time taken to achieve the maximum lap shear bond strength, 219 N/cm^2, using the focused microwave radiation process and a power level of 800 W was 45- 70 seconds, In comparison, the time taken to obtain the peak lap shear bond strength, 62 N/cm^2, utilising a commercial microwave oven by a power level of 1000 W, was 325 seconds. The time taken by the latter was 7 times longer than the former. It can, therefore, be argued that commercial microwave ovens are generally not suitable for materials processing applications [34].

Using VFM for joining LDPE/GF (33%) composite and Araldite as primer, it was found that the lap shear bond strength was only 21.5% more than that obtained by curing in ambient conditions. The processing time taken to cure the adhesive was relatively long (180 – 420 seconds) as compared to that required for curing by the focused microwave facility. The above drawbacks are entirely due to the low power output of the VFM facility. This can be overcome by employing a larger power facility, e.g., the 2kW power of Microcure 2100 Model 2000. The quality of bonds produced by the VFM processing has been much superior than its fixed frequency counterpart. Figure 11 shows that the bond quality obtained via VFM processing is ideal and no hot spot can be identified. On the other hand, hot spots were found in curing by FFM as shown in Figure 12 (see white arrow). Hot spots are spots in a piece of material (load) which absorb microwave energy of a particular frequency, eg. 2.45 GHz more than other parts of it. Those particular spots become heated faster than their neighbours and this is due to the inhomogeneous property of the material.

Many of the above adhesive curing experiments are experimental and time-consuming. Computer simulations of variable frequency heating or curing are, therefore, recommended [28]. The cost of high power commercial VFM facilities, e.g., Microcure 2100 Model 2000, is currently relatively high and in most industrial scale applications only the high power facilities will meet the demand of the loads. It is likely that multiple single-frequency sources, e.g., magnetrons or narrow-band sources (tens of MHz) would be preferred, as they are currently inexpensive and more powerful than amplified TWT devices [28].

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Figure 1: Cavity of Microcure 2100 Model 250 with a Frequency Range of 2 - 7 GHz and a Maximum Power of 250 W

Figure 2: Cavity of VW 1500 Model with a Frequency range of 6-18 GHz and a Maximum Power of 125 W

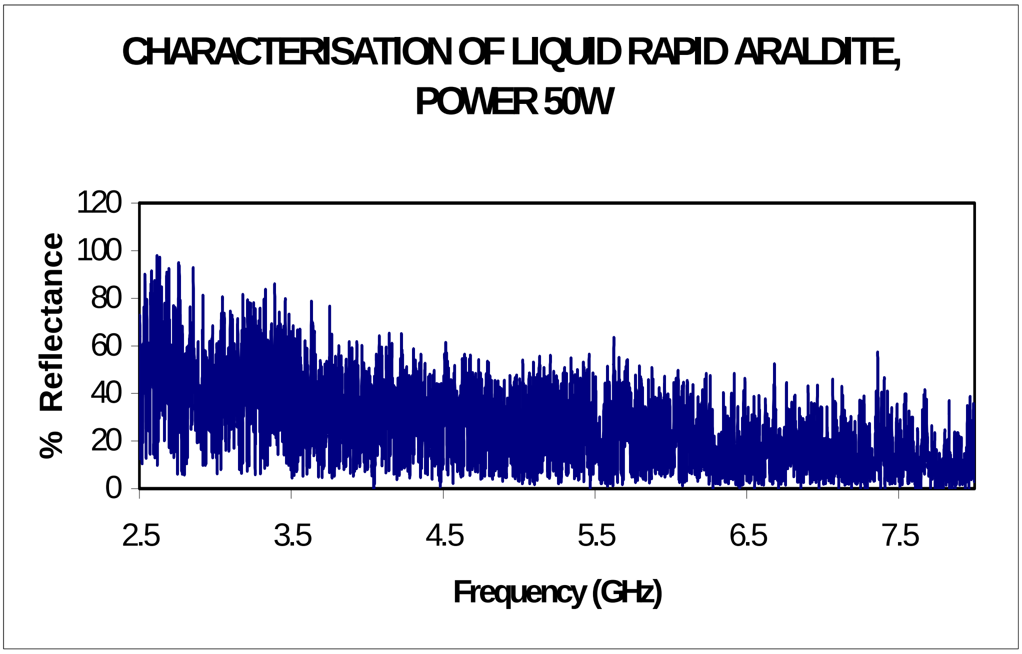

Figure 3: Percentage Reflectance against Frequency in Finding the Optimum Cavity Conditions for Araldite

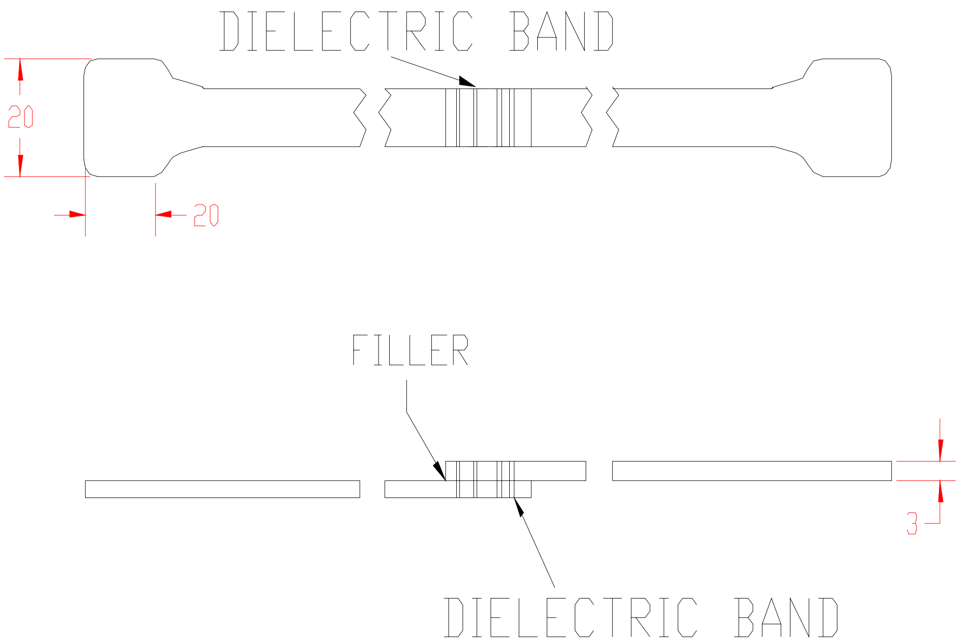

Figure 4: Two Mirror Image Test Pieces of LDPE/GF (33%) Composite

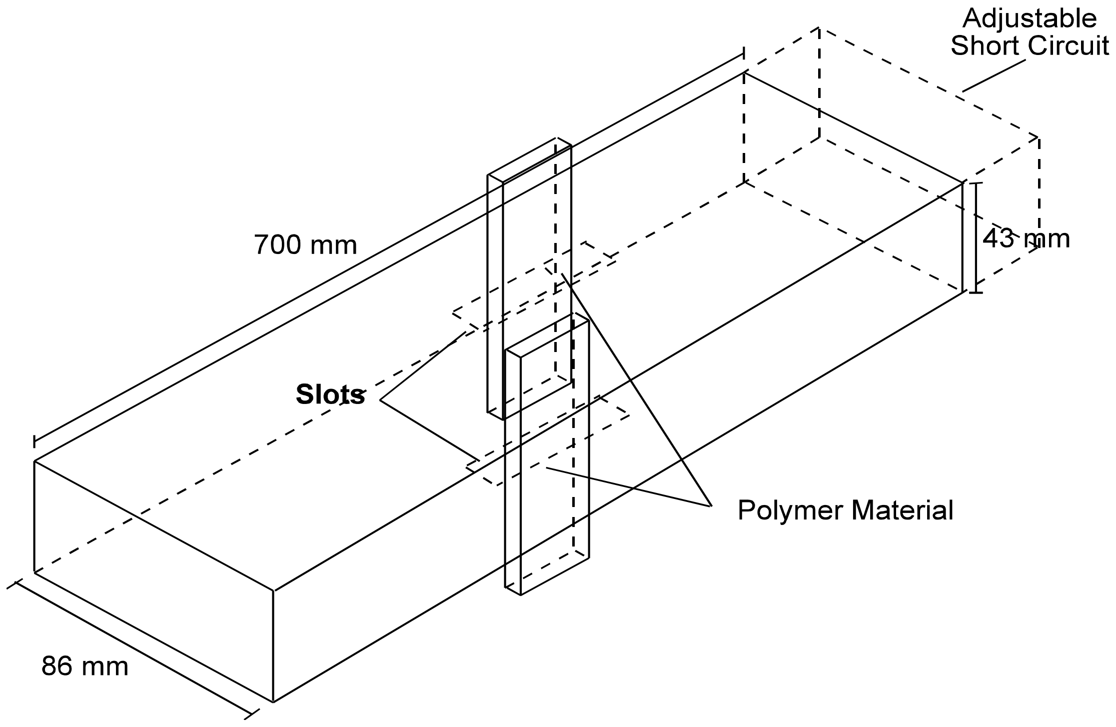

Figure 5: Slotted Waveguide Used for Joining HDPE Using Araldite

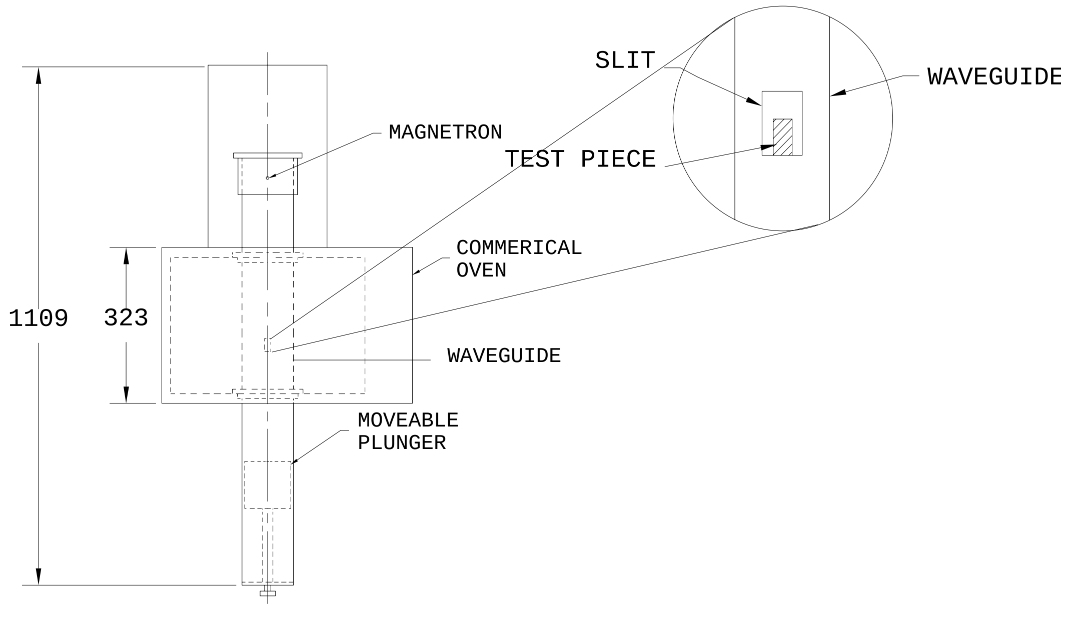

Figure 6: Slotted Rectangular Waveguide Microwave Configuration with All dimensions in mm

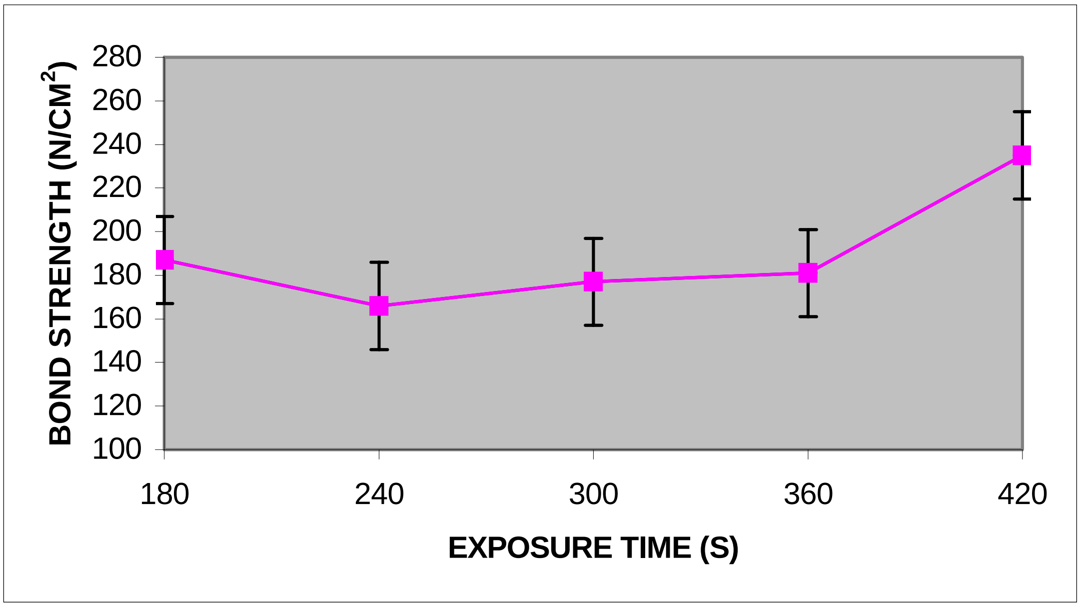

Figure 7: Lap Shear Bond Strengths of LDPE/GF (33%) Joined by VFM using Araldite with a Power Level of 200W.

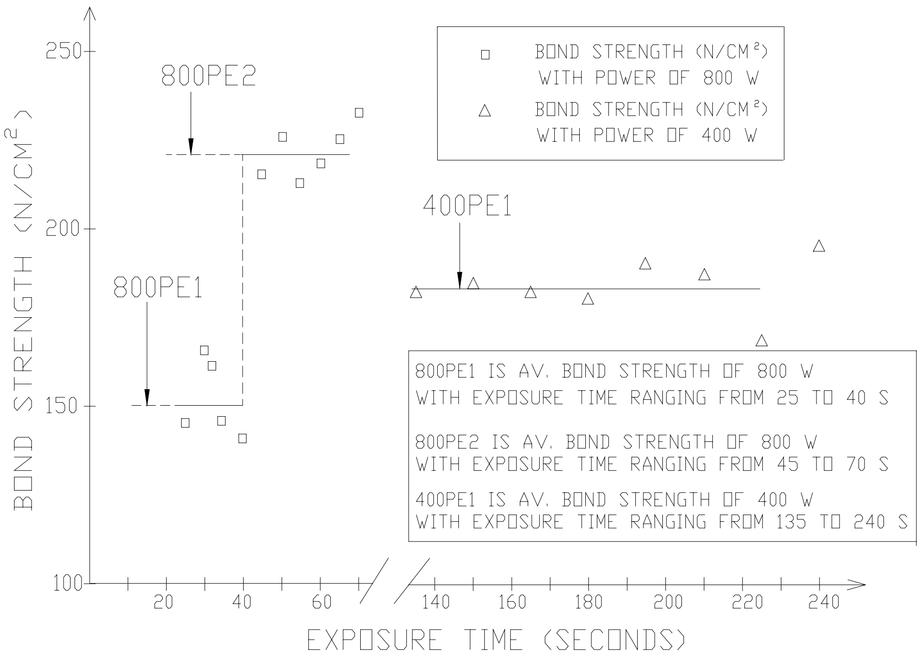

Figure 8: Lap Shear Bond Strength of LDPE/GF (33%) Composite Joined by FFM (2.45 GHz) in a Slotted Rectangular Waveguide using Rapid Araldite

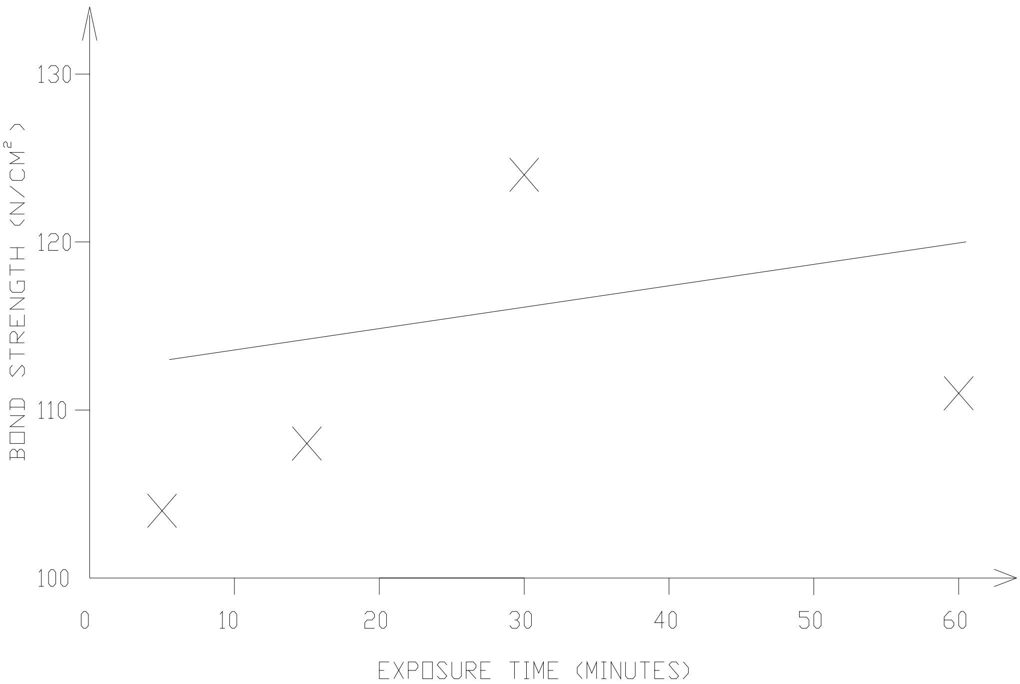

Figure 9: Lap Shear Bond Strength for Araldite used in joining HDPE and Cured in Ambient Conditions

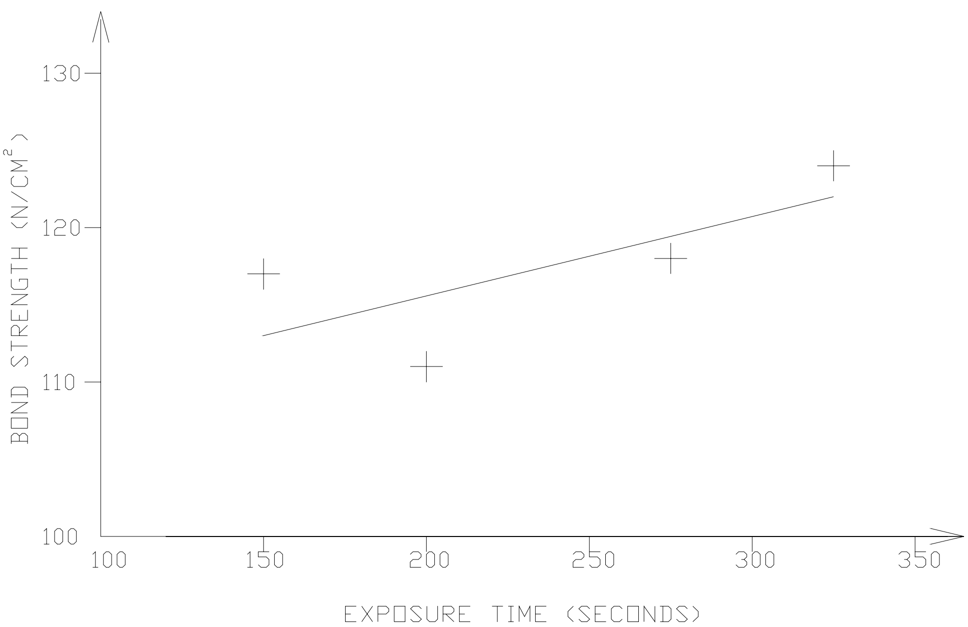

Figure 10: Lap Shear Bond Strength for Araldite used in joining HDPE and Cured in Microwave Oven at 2.45 GHz and a power level of 1kW

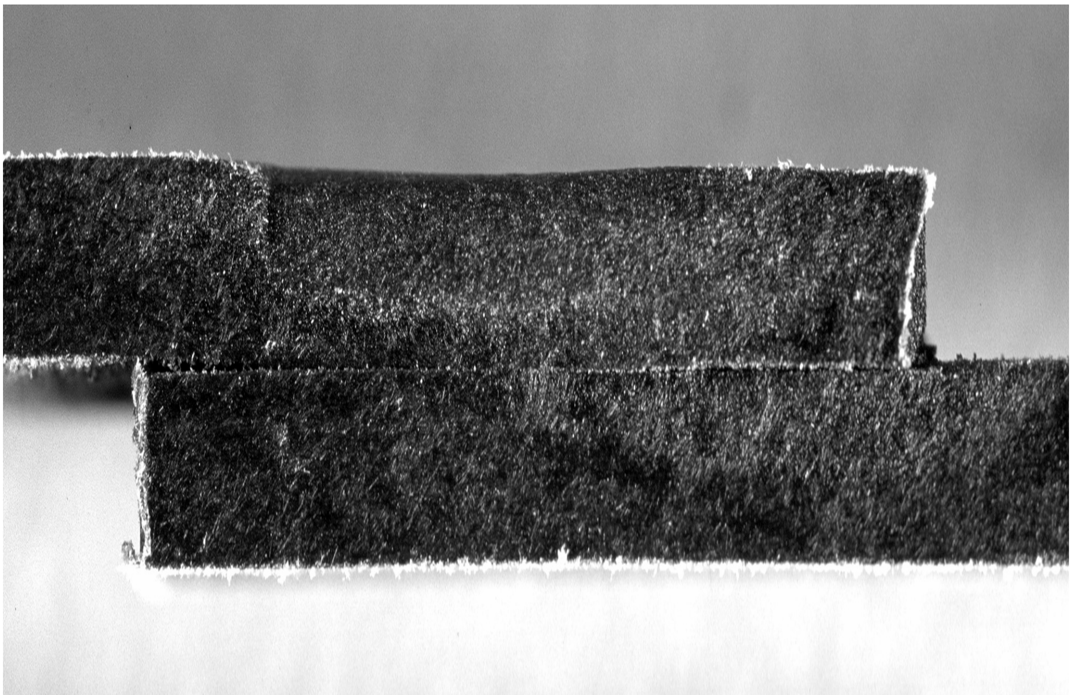

Figure 11 No Hot Spot found in the Lap Shear Bond Obtained through VFM Processing

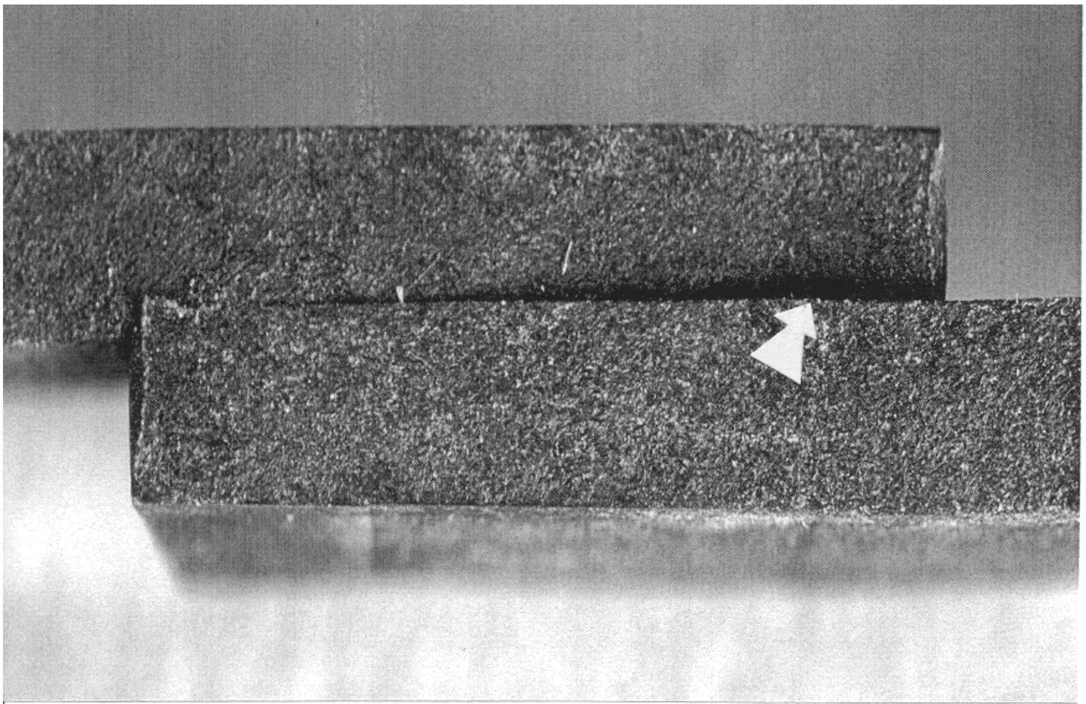

Figure 12 Hot spots are present (as indicated by white arrow) in the Lap Shear Bond Obtained through FFM (fixed frequency microwave) Processing.