Productivity improvement of composites processing through the use of industrial microwave technologies

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ABSTRACT:

This paper starts with the characteristics and advantages of microwaves processing. The shortcomings of fixed frequency, typically at 2.45 GHz were also mentioned. On account of this, the newly developed variable frequency microwave (VFM) fabrication was mentioned and adopted in place of the fixed frequency process. Two cases of fixed frequency microwave processing of materials were described; the characteristics, pros and cons of each case was mentioned and commented. Two cases of processing materials using variable frequency microwave facility (VFMF) were mentioned; the advantages and limitations of each case were discussed. The microwave processing of materials provides improved mechanical, physical and electrical properties with much reduced processing time. Furthermore, variable frequency microwave processing is more superior to its fixed frequency counterpart except that the cost of the facilities of the former is much higher than the latter at this point in time but it appears that the price will drop in the coming ten years.

Keywords: fixed frequency microwaves, variable frequency microwaves, polystyrene, low density polyethylene, glass fibre, carbon fibre and Araldite.

Introduction

The word *microwave* is not new to every walk of life as there are more than 100 million microwave ovens in households all over the world [1]. Faster cooking times and energy savings over conventional cooking methods are the primary benefits [2]. On account of its great success in processing food, people believe that the microwave technology can also be wisely employed to process materials, e.g. disinfecting food, drying agricultural produce, sintering ceramics or cross-linking polymeric composites. Microwave processing of materials is an alternative for processing materials that are hard to process; and an opportunity to produce new materials and microstructures that cannot be achieved by other methods. The use of microwave irradiation for materials processing has the potential to offer similar advantages in reduced processing times and energy savings [3].

It can be argued that the most likely candidates for future production-scale applications which will take full advantage of the unique characteristics of microwaves include polymers, ceramics, adhesives, composite joining and catalytic processes. The savings envisaged include timesaving, higher yield, and environmental friendliness [4]. In crosslinking composites, autoclave curing is suitable for individually cured parts made of thin, uniform laminates. Autoclave curing is less suitable when the parts are large, thick or have uneven dimensions, or when several different parts are cured simultaneously. In these cases, the unavoidable temperature gradients inside the autoclave and the thermal inertia of the autoclave make it difficult to ensure that the parts are cured uniformly and completely. Microwave curing offers the possibility of uniform, complete and fast economical cure regardless of the geometry of the part [5]. This paper reviews and comments microwave processing of composite materials using fixed as well variable frequency microwave energy.

Materials and Microwaves Interactions.

In general, microwave processing systems consist of a microwave source, a circulator, an applicator to deliver the power to the load, and systems to control the heating. These are shown in Figure 1. Most applicators are multimode, where a lot of field patterns are excited simultaneously [1].

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, $\tan\delta = \frac{\varepsilon''}{\varepsilon'}$. 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 tangent, $\tan\delta$ or dielectric loss, which predicts the ability of the material to convert the incoming energy into heat [6].

The heating effect in most polymeric matrix composites is the result of dipolar rotation and ionic conduction because these composites contain none or very small amount of magnetic materials. Molecules that are non-polar but are asymmetrically charged may behave as dipoles in an electric field; however, their responses to microwaves are usually about an order of magnitude less than that of water. The other heating mechanism is ionic conduction. The electrical field causes dissolved ions of positive and negative charges to migrate towards oppositely charged regions. This results in multiple billiard-ball-like collisions and disruption of hydrogen bonds with water, both of which result in the generation of heat [2].

Variable frequency microwaves (vfm)

The frequency of microwave oven in our kitchens is fixed at 2.45 GHz and magnetrons are used for the generation of microwaves. The fixed frequency microwave facility mentioned in this paper is also 2.45 GHz. Variable frequency microwave (VFM) technology is a new technique for microwave processing introduced to solve the problems brought about by fixed frequency microwave processing. The technique has been applied 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 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 are now viable [7, 8]. At the heart of the VFM technology is a high power, broadband, helix travelling wave tube (TWT), which has been used in the VFM furnace (VFMF) constructed to date [9]. During VFM processing, a variable frequency of microwaves would be launched into a multimode cavity sequentially for the duration specified, e.g. one millisecond.

Figure 2 shows the phenomena of fixed frequency microwave heating; areas with higher electric field strength are heated more, resulting in hot spots, which will generally stay in the same positions over the heating period and will finally lead to thermal runaway. There will be non uniform heating because of non uniform electric field. Figure 3 shows the electric field patterns in heating with variable frequency microwave power. On account of the sequentially launched microwaves at variable frequencies, the electric field patterns in a particular sample plane changes in less than one millisecond. A time-averaged heating results and the temperature rise in the sample will be uniform. One of the advantages of variable frequency microwave processing over conventional fixed frequency one is its ability to provide uniform heating over a large volume at a high energy coupling efficiency [8]. Another prominent advantage of it will be selective heating on different parts of a material which has different dielectric properties over the bulk of the material. In materials, with frequency sensitive dielectric behaviours, the central frequency can be adjusted to increase the dielectric loss of the materials. Thus heating rate can be changed without changing the power. The microwave incident power can be pulsed or continuously varied to provide some control over the heating profile of the workload [10].

Fixed frequency curing or processing of composite materials

Case 1

Ku et al. used a fixed frequency facility shown in Figure 4 to join glass fibre reinforced thermoplastic composites. With reference to Figure 4, the incident waves are generated by the magnetron located on the top of the equipment. The microwaves travel downwards through three sections of WR340 waveguide and interact with the test pieces located in the second section before being reflected back by the top face of the adjustable plunger. The test pieces are shown in Figure 5 shows the two halves of standard test pieces for composite materials. The lapped area was made 10 mm x 20 mm. After applying the filler, the two pieces were tightened by a dielectric band, which encircled the lapped areas four times as depicted in Figure 5. After tightening with a dielectric band, the two halves of the test pieces were positioned in the slot across the waveguide as illustrated in Figure 6. It was estimated that the pressure applied by the dielectric band to the test pieces was 4 N [11].

Two composite materials were joined and their lap shear strength tested; they are thirty three percent by weight glass fibre reinforced nylon 66 [NYLON/GF (33%)] and thirty three percent by weight glass fibre reinforced low density polyethylene [LDPE/GF (33%)]. After joining, the joints were lap shear tested. 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 selected for the test [12].

Figure 7 shows the lap shear strength of Nylon 66/GF (33%) joined by a fixed frequency microwave facility in a slotted rectangular waveguide. With glass fibre reinforced Nylon 66, the peak lap shear strengths were obtained at exposure times of 35 and 55 seconds for the power levels of 400 W and 240 W respectively; they are depicted in Figure 7. They were 32% and 28% respectively higher than those obtained by curing the adhesive at room temperature conditions but the times required were only 1.0 % and 1.53 % of their counterparts. Any excess Araldite that spilled over the sides and opposite faces of the interfaces of the test pieces had to be totally removed as the primer facing the microwave energy directly could bring about thermal runaway and the parent material could burn, depending on the degree of spill-over of the adhesive [11]

Figure 8 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 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) [13, 14]. 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. In both cases, the results obtained were similar to the work of another researcher using high-density polyethylene [15].

Case 2

Paulauskas et al. joined glass and urethane-based composites with glass fibre reinforcement using epoxy-based adhesive, 100% Goodrich 582E. In some cases, 0.1% or 1.0% by weight of carbon black powder was added to the adhesive to enhance microwave absorption. All samples were exposed to varying power levels and duration of exposure to microwave irradiation. The researchers understood that microwave processing of the composites will reduce the curing time and the addition of black carbon powder would further reduce the processing time. The microwave system used could supply a maximum power of 5.5 kW and its frequency was 2.45 GHz. The dimensions of the multimode cavity were 61 cm x 61 cm x 61 cm. An analysis of all data showed that curing time was decreased as the electric field intensity of the microwaves increased. The study also found that microwave processing would be within a third or a quarter of conventional curing time [16].

A standard Instron Tensile testing machine was employed to lap shear test the bonded samples. With acceptable joints, failures occurred as a fibre tear “peel” of the urethane substrate. The processing times varied from 10 to 40 minutes depending on the power level. It is a pity that the input power level was not numerically specified but it was written as high, medium and low. High power was for short duration, medium power was for medium duration and low power was for long duration. The ultimate tensile strengths, σ_B varied from 1800 N/cm^2 to 2100 N/cm^2, which were very high when compared with the work done by Ku et al. [20]. Paulauskas et al. did not specify the lapped areas of the lap joint of the test pieces but in accordance with the information provided, it is estimated that the lapped area was 325 mm^2 (26 mm x 12.5 mm) which was 1.5 times that of the work done by Ku et al. [16,17].

A satisfactory microwave sample when submitted to the single lap shear test demonstrated a fibre tear "peel" type failure on the urethane substrate. The average ultimate tensile strengths of samples with high input power (not specified) and short duration of exposure (10 – 13 minutes) was 1400 N/cm^2. The study also found that a nonuniform formation of bubbles occurred in the adhesive in the joint area. However, good mechanical strengths were obtained with these bubbled samples when they were subjected to tensile test.

Samples with 0.1 % or 1.0% by weight of carbon black powder added to the adhesive showed good mechanical strength. The high input power and short curing time of theses samples showed some bubbles in the interface. This may account for the increased in lap shear strength. For samples with no voids, fractures occurred by near surface fibre tear of the composite. The maximum lap shear strengths were found in samples with medium cure times (14 – 25 minutes) and medium power levels (not specified) used [16].

Comments on cases

Cases 1 and 2 showed that the microwave processed samples exhibited higher lap shear strengths and more plasticity than the conventionally processed (ambient cured in Case 1) specimens. In both cases, epoxy-based adhesive were used as primers and they could couple well with microwaves. Araldite used in Case 1 had a loss tangent of 0.117 at 2.45 GHz and at 20^{o}C [17]; the epoxy-based adhesive, 100% Goodrich 582E adhesive used in Case 2 seemed to couple better as the lap shear strengths obtained in this case were higher. In Case 2, the adhesive used in some samples had added carbon black powder; this would improve the coupling efficiency between the adhesive and the microwaves. It appears that if carbon black powder was also added to the Araldite, the lap shear strengths of the joints of the samples in Case 1 could be improved. The curing times used in Case 2 were also much longer than in Case 1. If longer curing times (3-6 minutes, depending on the thermoplastic matrix material) were used in Case 1, the Araldite would be overcured with bubbles along the interface of the joints and the lap shear strengths of the samples were poor. This was also true with high power levels in Case 2. It is a pity that the power levels of the microwave energy used in Case 2 were not given, otherwise more comparison can be made and more useful results can be achieved. Microwave irradiation had brought about diffusion bonding in the test pieces in both cases; this was proved by viewing the interfaces of the joined samples under scanning electron microscope (SEM).

In Case 1, the microwaves had been launched to the composite test pieces directly through a WR340 rectangular waveguide, which was used as an applicator. This resulted in high energy rate of joining the composites; the adhesive, the parent materials and the glass fibre melted into each other and hence the mechanical property, lap shear stress of samples cured by microwaves were higher than those cured under ambient conditions [11, 18, 19]. In case 2, the microwaves were launched into a multimode cavity of 61 cm x 61 cm x 61 cm, which was relatively large and could be used in industry without much modification. The large cavity was compensated by the large power generator of 5.5 kW and samples cured by microwaves also had higher tensile strengths than those cured conventionally. However, the cost of the facility could be very high.

Variable frequency curing or processing of composite materials

Case 3

Ku et al. joined glass fibre and carbon fibre reinforced thermoplastic composites using variable frequency microwave facilities (VFMF) shown in Figures 9 and 10 respectively. Figure 9 shows Wari-Wave VW 1500 microwave oven; its frequency range is from 6.5-18 GHz and its maximum power is 125 W. Its cavity dimensions are 250 mm x 250 mm x 300 mm. Figure 10 illustrates Microcure 2100 model 125 microwave facility; its frequency range is from 2 – 8 GHz and its maximum power is 250 W. Its cavity size dimensions are 300 mm x 275 mm x 375 mm. Before joining, characterization of the joined materials was required to find out the best frequency band(s) to process the material with variable frequency microwave (VFM) energy. Table 1 shows the optimum frequency bands to process the 3 materials studied in the frequency range of 2 GHz to 18 GHz [18, 20]. The 3 materials are:

1. Nylon66/GF (33%);

2. LDPE/GF (33%) and

3. LDPE/CF (33%)

The best frequency to process Nylon66 GF/ (33%) are from 8.3 - 9.0 GHz and 10.8 - 12.0 GHz as shown in Table 1 [21]. Because of frequency restriction, Wari-Wave VW 1500 VFMF has to be used.

The programme for joining Nylon66/GF (33%) was as follows: central frequency = 7.25 GHz; bandwidth = 1.5 GHz; sweep time = 0.1 secs; power output = 200 Watts; set temperature = 100 ^{o}C; deadband = 1 ^{o}C; duration = 30 - 100 seconds; maximum temperature = 105 ^{o}C. Figure 11 illustrates the bond strengths of nylon66/GF (33%) against different exposure time intervals. The centre sweep frequency, 7.25 GHz and its sweep bandwidth, 1.5 GHz, were found to be most suitable for processing the primer, rapid Araldite as well. During most of the exposure period, the shear strengths of the test pieces were found to be above 500 N/cm^2 and test pieces failed at bondline. At an exposure time of 30 seconds, the bond strength of the test piece joined by VFM was around 503 N/cm^2, which was 1.5% higher than that obtained from the fixed frequency facilities using a power level of 400 W. Similarly, at an exposure time of 70 seconds, the bond strength using VFM was 523 N/cm^2, which was 9 % higher than that procured from its rival operating at 240 W. Figure 11 shows that, within limits, the longer the time of exposure to microwave energy, the higher will be the bondline strength of the material. At an exposure time of 100 seconds, the bond strength was 653 N/cm^2.

Figure 12 shows that lap shear strengths of LDPE/GF (33%) obtained ranged 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 was 156 N/cm^2, which was very low but was reasonable because Selleys pointed out that Araldite is not suitable for joining low-density polyethylene (LDPE) [22]. 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 [19].

With VFM, no Araldite was used and no bond was formed if the processing time for LDPE/CF (33%) 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 13 shows that lap shear strengths obtained ranged 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 failed 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. 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.

Both materials, LDPE/GF (33%) and LDPE/CF (33%) were bonded at a frequency range most suitable to process them (see Table 1) [21]. 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 12 and 13, 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} [6]. The thermoplastic matrix in the composites is 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 [23, 24].

Case 4

Fathi, et al. used variable frequency microwave furnace to reduce the post-cure time of isocyanate/epoxy systems from 8 hours at 240°C to 60 minutes at 200°C. Several experiments were conducted using different samples. All the dimensions of the plate-shaped samples were 10 cm x 10 cm x 2.5 mm. The temperature across the samples was measured using a four-channel, Luxtron fibre-optic temperature monitoring system. The heating of a series of plate configuration was performed with 1, 2, 3, 4 and 8 plates staking. The glass transition temperatures (T_g) of a three plates stacking configuration processed at 175 °C for 60 minutes using 4-5 GHz at 150 Watts was measured in different locations; the results ranged from 165 °C to 172 °C with an average of 168 °C. Eight plates were stacked and uniformly processed at 200 °C for 60 minutes, the glass transition temperature of each of the plates were measured. The T_g of the middle sample from the 8 stack configuration was measured at 5 different locations. They ranged from 190 °C to 206 °C with an average of 196 °C. The glass transition temperatures in the two experiments were close to the cure-soak temperature. The heating rate was in the range of 6.0 to 7.5 ^{o}C per minute. All samples were cured uniformly without warpage. The measured T_g values showed that all samples were fully cured. Some samples were heated at fixed frequencies (the VFMF can be set to a fixed frequency for processing materials) at 200 Watts using similar sample configurations as those in VFM curing. Only parts of the samples were cured while others retained the grey colour of the starting materials. This was due to hot spots formation in the materials [10].

Fathi, et al. also developed computer model to better understand the quantitative differences between fixed and variable frequency processing, and identify the cause and predict the locations of hot spots. The computer model also worked accurately [10].

In addition to this, it can be argued that the finite-difference time-domain (FDTD) software developed by Kung and Chuah can be slightly modified for simulating the results in cases 3 and 4 [25].

Comments on cases

In case 4, it was confirmed that microwave heating could reduce the processing time of PMCs when compared to conventional curing, and that VFMF could eliminate hot spots and thermal runaway associated with fixed frequency heating. The electronic tuning system makes VFMF a versatile processing tool to an array of materials. In case 3, it was found that material processed will be able to absorb more of the launched microwave energy when the frequency launched by the oven is so selected that it is the best frequency to process the material. This can only be achieved by using VFMF.

By comparing Figures 7 and 11, it was found that if the processing time was the same, the lap shear strengths obtained by VFMF were higher than those cured by fixed frequency facility despite the power used was only half of that of it counterparts.

Both cases 3 and 4 indicate that VFMF processing of composites gave stronger lap shear strength than the fixed frequency facilities, which in turn cure composites better than the conventional methods.

The arguments made by Chew can be applied in all cases in this study [26]. It will also be interesting to include microwave filters proposed by Khalaj-Amirhosseini in the waveguides of cases 1 and 2 [27].

Conclusion

In general, variable frequency microwave processing of materials is superior to its fixed frequency rival. On the other hand, the fixed frequency facilities are much cheaper than its counterpart at this point in time. However, this is likely to change in the coming ten years. Whether it is fixed or variable frequency of microwave processing, the curing time for composites has been largely reduced with the enhanced or comparable materials properties. In addition, the glass transition temperature (T_g) for all composites shifted to a higher temperature when the materials were processed using microwave irradiation. This was due to higher curing extent. With VFM Processing, composites containing carbon fibre and metal particles can be processed without arcing.

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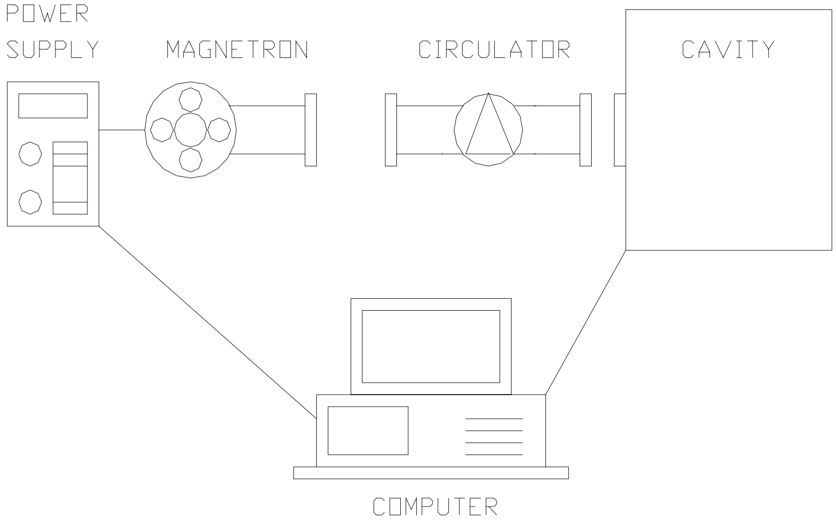
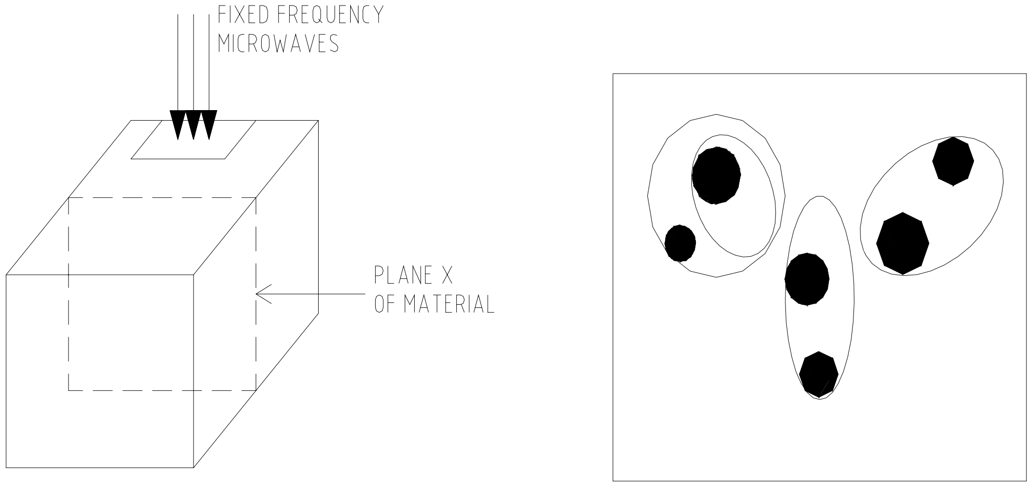


Figure 1: Microwave System for Materials Processing



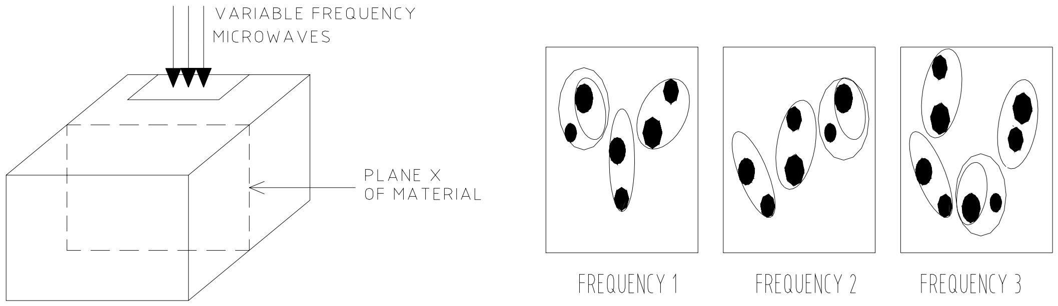
(a) Multiple reflections of incident microwaves resulting in a field pattern within the cavity

(b) field pattern at plane X due to fixed frequency microwaves (black represents field strength)

(c) Sample at plane X of the material

(d) Non uniform heating in sample at plane X due to non uniform electric field

Figure 2: Non uniform heating by fixed frequency microwaves



(a) Multiple reflections of incident microwaves resulting in a field pattern within the cavity

(b) Field patterns at plane X with three different frequencies variable frequencies (black represents field strength)

(c) Sample at plane X of the material

(d) Uiform heating in sample at plane X due to non uniform electric field

Figure 3: Uniform heating by fixed frequency microwaves

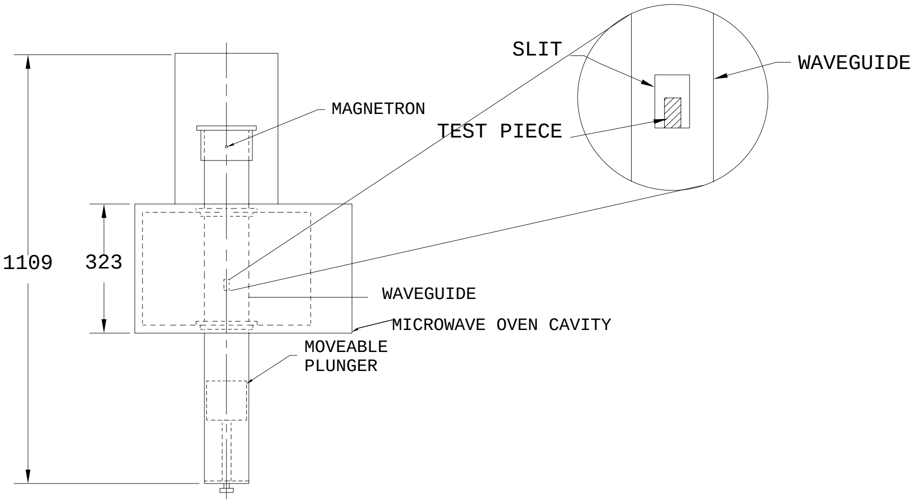


Figure 4: Microwave facilities configuration

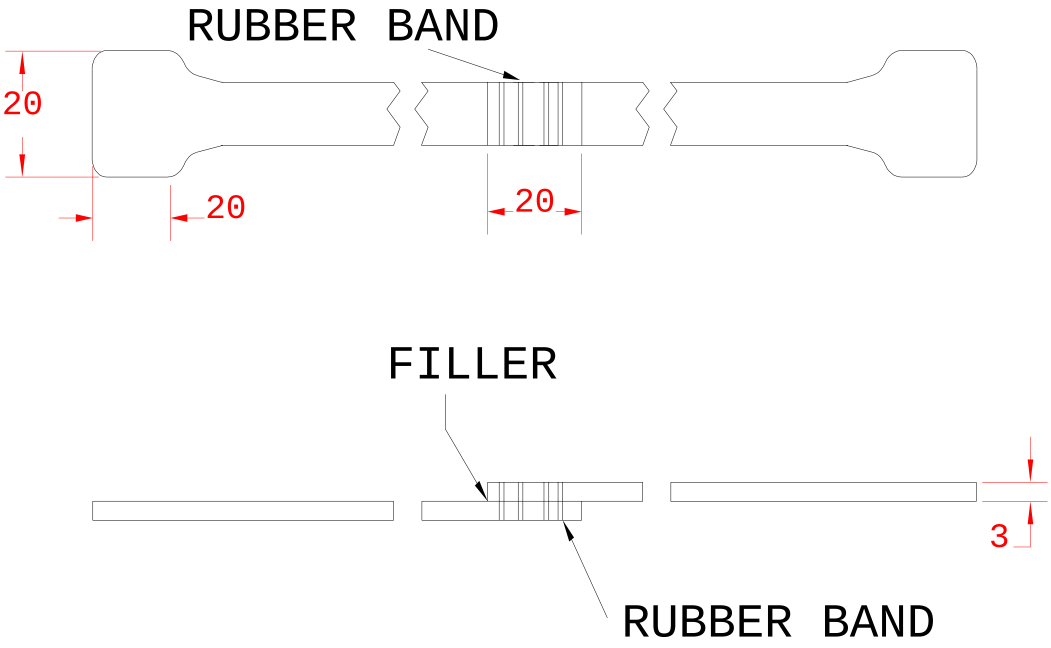


Figure 5: Test pieces tightened by a dielectric band

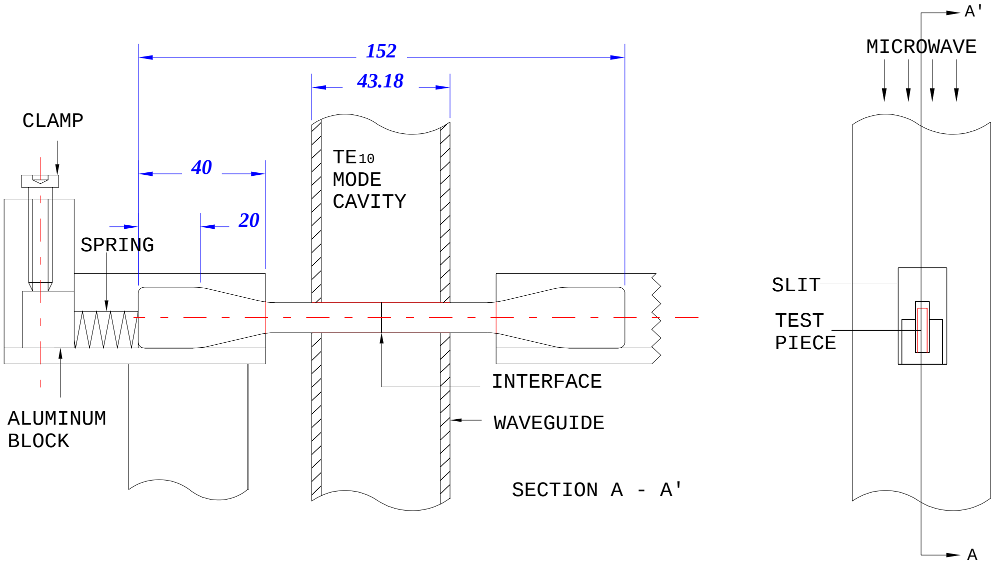


Figure 6: Test pieces in position

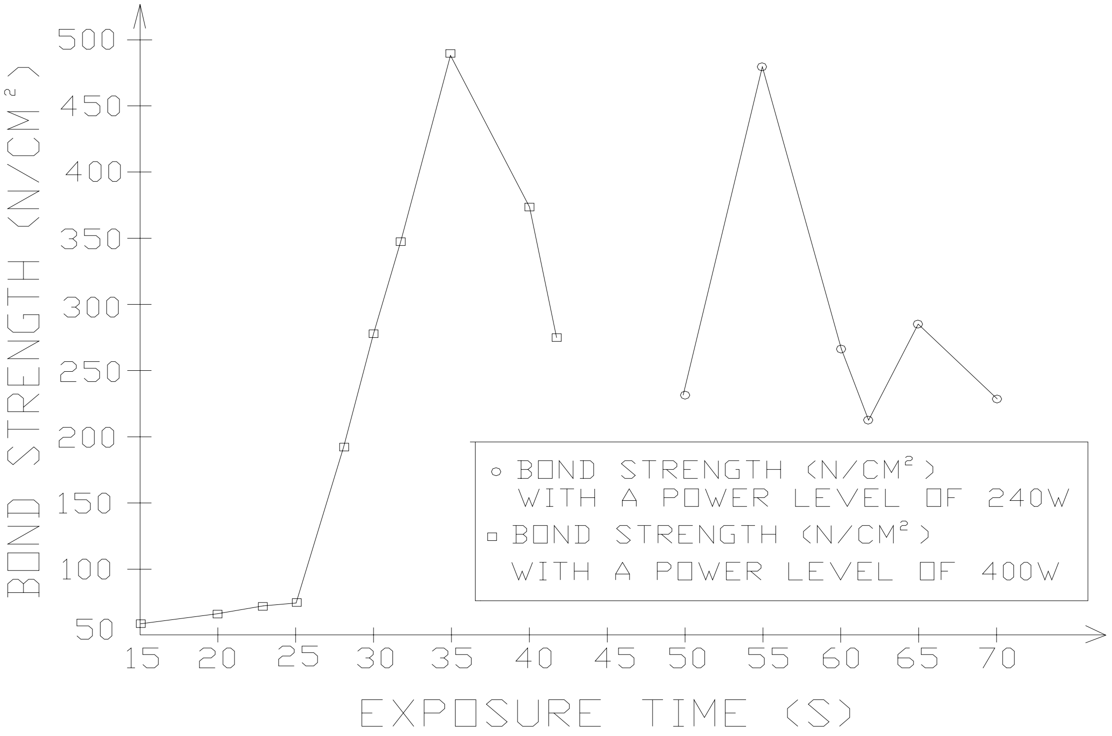


Figure 7: Lap shear strength of nylon 66/GF (33%) bonds joined by fixed frequency microwave (2.45 GHz) in a slotted rectangular waveguide using 5-minute two parts Araldite

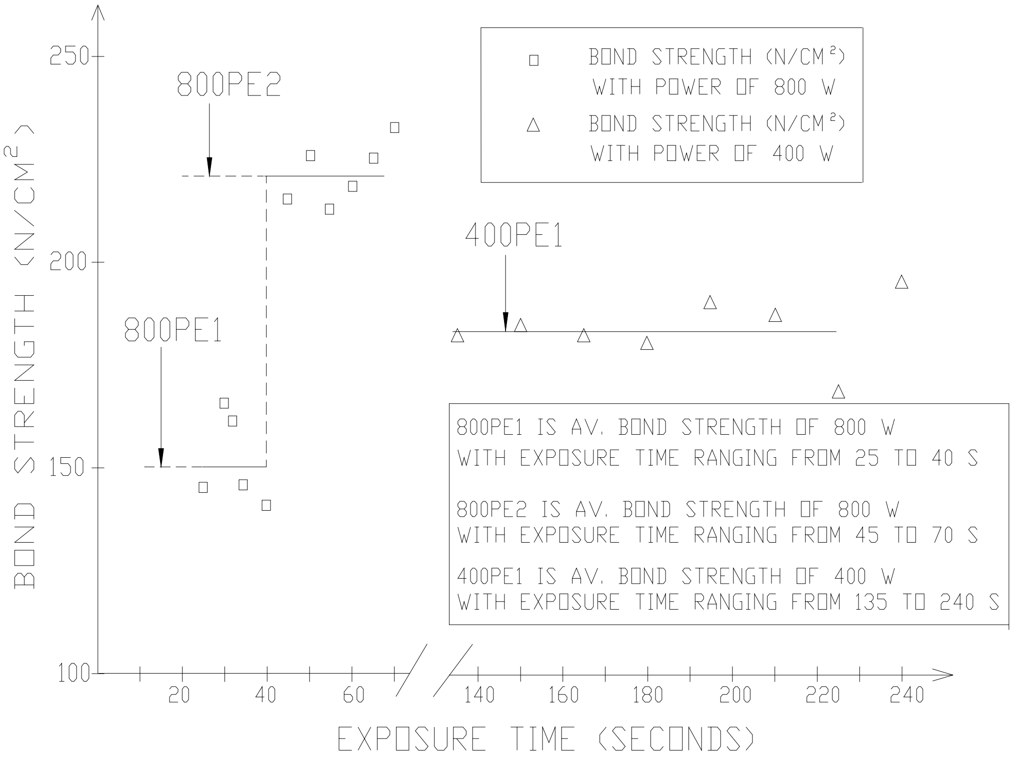


Figure 8: Lap shear strength of LDPE/GF (33%) joined by fixed frequency microwave (2.45 GHz) in a slotted rectangular waveguide using 5-minute two parts Araldite

Figure 9: Cavity of Wari-WaveVW 1500

Figure 10: Cavity of Microcure 2100 model 250

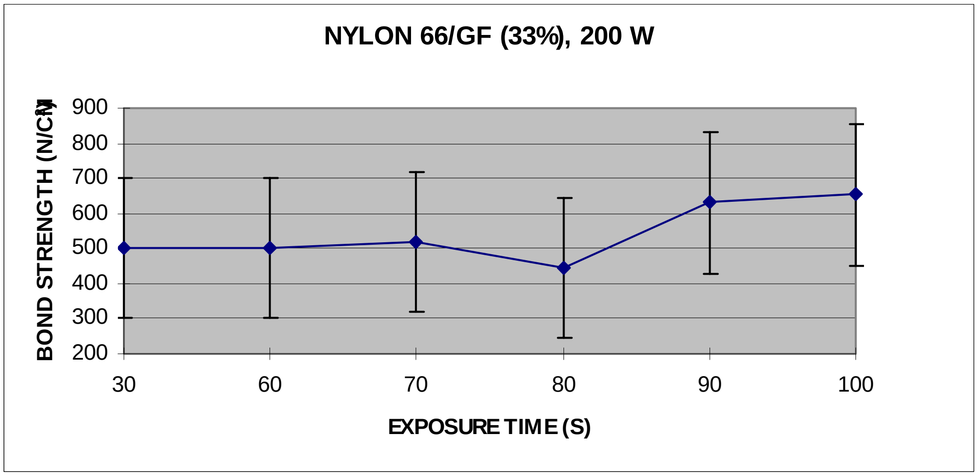


Figure 11: Bond strength of nylon 66/GF (33%) with Araldite using variable microwave frequency

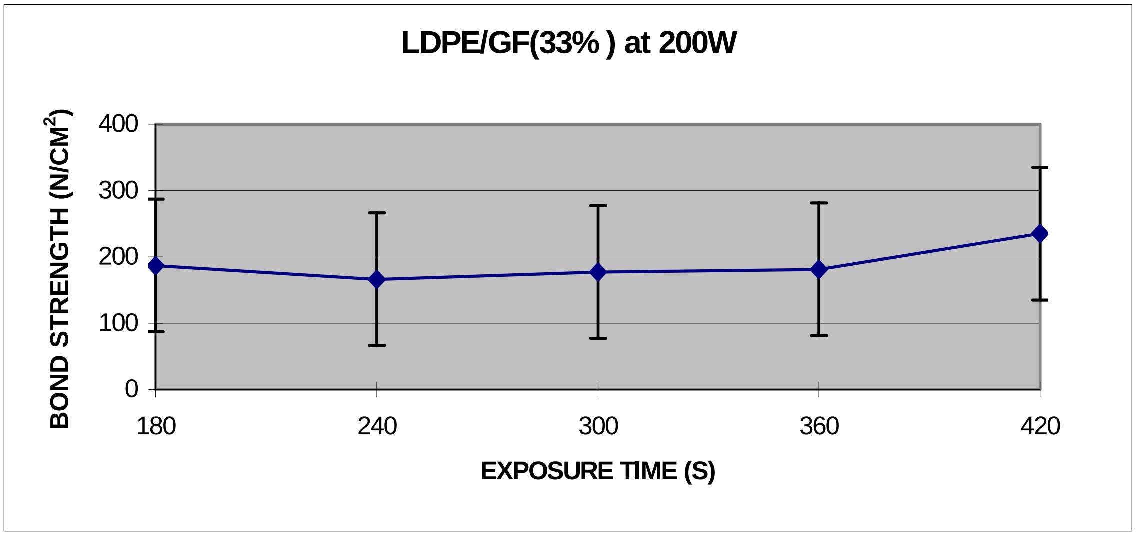


Figure 12: Lap Shear Strength of LDPE/GF (33%) Bonds Joined by VFMF

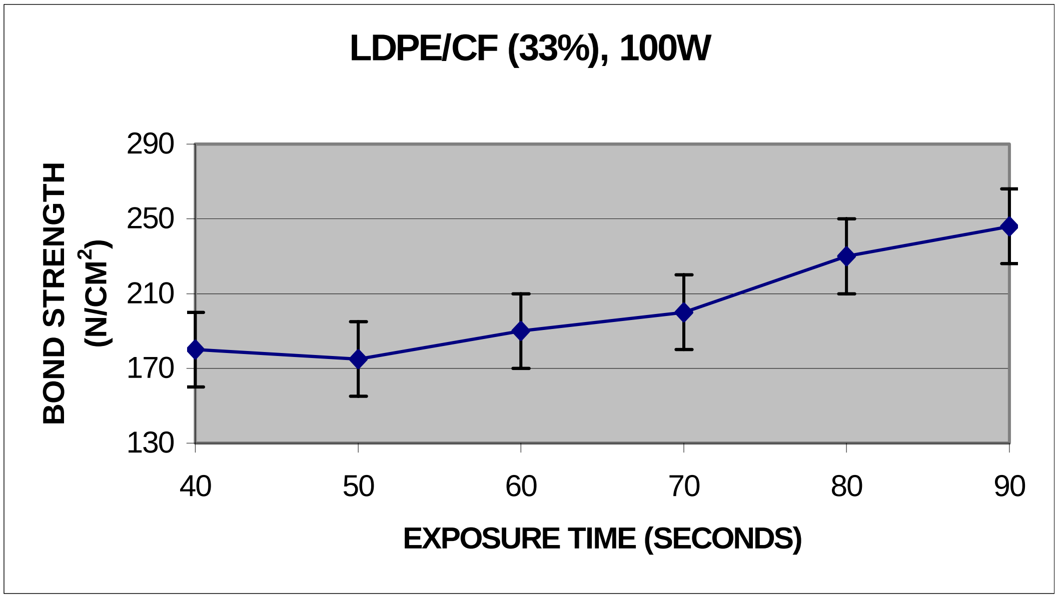


Figure 13: Lap Shear Strength of LDPE/CF (33%) Bonds Bonded by VFMF

Table 1: Optimum Frequency Bands to Process the 5 Materials in the Frequency Range of 2 GHz to 18 GHz.

Materials	Optimum Frequency Band (GHz)
LDPE/GF (33%)	9.0 - 12.5
LDPE/CF (33%)	8.5 - 9.0 and 10.7 - 12.0
Nylon 66/GF (33%)	8.3 - 9.0 and 10.8 - 12.0