Variable Frequency Microwave Processing of Thermoplastic Composites

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Abstract: This paper extends the range of applications for Variable Frequency Microwave (2 – 18 GHz) (VFM) facilities to thermoplastic composites. Five thermoplastic polymer matrix composites are processed and discussed, including 33% by weight random carbon fibre reinforced polystyrene [PS/CF (33%)], and low density polyethylene [LDPE/CF (33%)]; 33% by weight random glass fibre reinforce polystyrene [PS/GF (33%)], low density polyethylene [LDPE/GF (33%)] and nylon 66 [Nylon 66/GF (33%)]. Bond strengths of the joints were shear tensile tested and results were compared with those obtained using fixed frequency (2.45 GHz) microwave processing. The primer or coupling agent used was 5-minute two-part adhesive containing 100% liquid epoxy and 8% amine, which was more readily microwave reactive than the composites themselves. The VFMF was operated under software control, which provided automatic data logging facilities.

Industrial applications of microwaves are relatively new technology. 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.[1] 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. [2,3,4,10] 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.[3,5,10]

INTRODUCTION

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. [6,7] 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.[7,8] 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.[8,9]

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,[8] as shown in figure 2(a), more than one thousand frequencies were launched into the cavity sequentially. 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.

FIXED FREQUENCY MICROWAVE PROCESSING OF MATERIALS

In the fixed frequency microwave facilities configuration, 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 change of temperature during the joining process was not measured. The apparatus used in the fixed frequency processing has been described in other papers and will not be discussed here.[4,5,10]

VFM PROCESSING OF MATERIALS

The VFMF are located at Industrial Research Institute Swinburne (IRIS), Swinburne University of Technology, and consist of a Microcure 2100 Model 250 with a frequency sweep range of 2 - 7 GHz operating at a nominal power of 250W, and of a VW1500 with a frequency range of 6 – 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 programme input. The dimensions of the cavity for Microcure 2100 Model 2500 are 300 mm x 275 mm x 375 mm. Two halves of tensile test piece of the sample were joined together using the VFM energy with or without araldite as primer. 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 1.5 to 2 cubic centimetres was applied to the two roughened surfaces to increase the mechanical keying or interlocking.[10] The two test pieces were then brought together and the total pressure applied was about 4 N. Programme with the required parameters was then written and input to control the VFMF via the PC. In the VFMF, 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.

Programme for LDPE/GF (33%)

The best frequency to process this material using Microcure 2100, ie frequency range between 2 GHz to 8 GHz was from 6.5 GHz to 8 GHz.[11] Since the material was processed with variable frequency sweep, it was necessary to identify the centre frequency for the sweep, which was found to be 7.25 GHz. Since the bandwidth of the sweep should be greater than 1.0 GHz, the selected bandwidth was 1.1 GHz.[12] The actual start and stop frequencies would be centre frequency $\pm \frac{bandwidth}{2}$, ie the sweep would be from 6.7 GHz to 7.8 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.[13,14,15] The processing temperature was set at 95^{o}C with a deadband (precision) of 1^{o}C and the total processing time was set at 6 minutes. The maximum permitted temperature was set at 100^{o}C, above that the machine was switched off automatically. The programme for joining LDPE/GF (33%) were as follows:

Variable Frequency = 7.25 GHz, bandwidth = 1.1 GHz, sweep time = 0.1 secs

Power Output = 200 Watts

Set Temp = 95 Degrees C, 1 Degree C, duration = 6 minutes

Maximum Temperature = 100 Degree C

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 LDPE. The reason for setting this maximum temperature was to avoid excessive temperature rise, which forms hot spots and thermal runaway. Results of the process will be given in the result section later on.

Programme for PS/GF (33%)

For this material, a fixed frequency of 2.5 GHz was chosen and in use with the VFMF. Microwave energy is launched into the cavity to enable comparison to be made between LDPE/GF (33%) and PS/GF (33%). The same primer was also used and the power level of 200 W was chosen. While the processing temperature was set at 95^{o}C and the processing time was set at 540 seconds. The maximum temperature was set at 100^{o}C to prevent overheating. Other parameters were left the same as in the case of LDPE/GF (33%). Results will also be discussed later.

Programmes for Other Materials

Three other sets of experiments were performed to process the remaining three materials, Nylon 66/GF (33%), LDPE/CF (33%) and PS/CF (33%).

Again, for Nylon 66/GF (33%) the same filler material liquid rapid araldite, was used. With this material, the centre frequency chosen was 7.25 GHz but the bandwidth of sweep was increased to 1.5 GHz for more optimum bond strength results.[15] Since the Vicat temperature[16] of nylon 66 was higher than those of polystyrene and low density polyethylene, the temperature was set at 100^{o}C to prevent the overheating of the araldite. The output power of 200 W was selected. Details of tensile shear test results and other findings are described later.

With PS/CF (33%), no araldite was used as a primer because the reinforcing carbon fibre would bring about larger loss to the material. Joining of PS/CF (33%) by VFM was also performed using centre sweep frequency of 7.25 GHz and the sweep bandwidth of 1.5 GHz. The power output was 100 W. The results are given later.

The last material to be considered was LDPE/CF (33%). In this case, the processing was also carried out using VFMF but a fixed frequency of 2.5 GHz was chosen. Because of the fixed frequency used, and the anticipated arcing of the carbon fibre, the duration of exposure to microwave energy was significantly reduced.

RESULTS

LDPE/GF (33%) Processing Results

During the processing, it was found that the temperature rose steadily with no sign of hot spots or thermal runaway as shown in Figure 3. The maximum temperature reached was 95^{o}C at time equalled to 360 seconds. For obtaining tensile shear test results, several sets of test pieces were joined at different duration and details are discussed below.

Fixed Frequency Results for LDPE/GF (33%)

At the fixed frequency of 2.45 GHz and a power level of 800 W, the cluster of experimental bond strength results, at microwave exposure times ranging from 25 to 40 seconds, were best represented by their average value of 302 N (line 800PE1 in Figure 4); while those resulting from microwave energy exposure in a range of 45 to 65 seconds was represented by their average value of 437N (line 800PE2 in Figure 4). In both cases, the results obtained were similar with the work of another researcher in Australia using high density polyethylene.[17] A step change in behaviour was also noted but the reasons for it has to be explored through more thorough study of the materials. These were illustrated in Figure 4. At shorter exposure times, the recorded average bond strength was only 97% of that cured in ambient conditions and it could be argued that no diffusion of parent material to the primer had taken place.[10] When longer exposure times were used, the average bond strength was found to be 41% higher. The processing times were also merely 0.06% and 0.1% respectively of the ambient cured ones. At the power level of 400 W, the cluster of bond strengths, obtained by exposing to fixed frequency microwaves from 135 to 240 seconds were best represented by their average value of 369N (line 400PE1) as depicted in Figure 4. It was 18% higher than that cured in ambient conditions and the processing time was only 0.33% of the ambient cured one.

Variable Frequency Results for LDPE/GF (33%)

With VFM, 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 a tensile shear stress test, failure clearly occurred at the parent material. Figure 5 shows that bond strengths obtained had the same characteristics as in the case of fixed frequency facilities, ie the bond strength would not improve much with increasing duration of microwave irradiation and was found to be 378 N.

PS/GF (33%) Processing Results

As a temperature of 101^{o}C (precision set was 1^{o}C) was attained at time equalled to 290 seconds, the machine was automatically stopped. It was found that temperature rose steadily and slowly in the first one hundred seconds as shown in Figure 6. The steep rise in temperature was observed when time equalled to 101 seconds. Heat conductivity was still very good at the beginning of the process and the heat was conducted to the surrounding area quickly as the temperature reached below 80^{o}C. Above this temperature and in time duration of 210 seconds, hot spot(s) developed into thermal runaway and the machine was shut down when the temperature of 101^{o}C was reached at time equalled to 290 seconds. Since the power level of 200 W caused hot spots and possibly thermal runaway, the power level was reduced to 150 W and several sets of test pieces were processed at different time intervals. Figure 7 shows the temperature versus time diagram for PS/GF (33%) joined at 150W and for a period of 570 seconds. The maximum temperature of 95^{o}C was recorded in running this experiment and the optimum tensile shear test results are detailed in later paragraphs.

Fixed Frequency Results for PS/GF (33%)

The primer used for joining this material was also five minute two part adhesive. Simple lap joints were selected for the connection of the two half test pieces. It was found that with 400 W power level, peak bond strength was achieved by exposing the test pieces to microwaves for 2 minutes; the bond strength (651N) and hence the shear stress (3.255N/mm^2) at this exposure duration exceeded that obtained by ambient conditions curing by 17%, but the time required was a mere of 0.2% of its counterpart.[4,5,13] For exposure times of one and a half to four and a half minutes, the shear stresses obtained using microwave-cured filler were higher than those obtained by allowing the adhesive to set under ambient conditions. With a power level of 800 W, the maximum bond strength (661N) and hence the maximum shear stress (3.305 N/mm^2) were achieved when the exposure time was 45 seconds and it exceeded the ambient conditions cured bond strength by 19 %, but the time required was only 0.08% of its rival.[4,5,10] The lower bond strength obtained, for test pieces exposed to microwaves for over 2 minutes and 45 seconds for power levels of 400 W and 800 W respectively, might be due to over-curing of the adhesive.

Fixed Frequency Results for PS/GF (33%) Using VFMF

Figure 8 shows the apparent bond strength of PS/GF (33%) bonded with two-part five-minute araldite as primer, using VFMF. The word apparent was used because all test pieces failed at the parent material and not at the bondline. This meant that the bondline was stronger than the parent material. Using VFM, the fixed frequency of 2.5 GHz was selected to enable comparisons to be made. It was found that the apparent peak bond strength of 532 N was observed at an exposure time of 480 seconds. Values for other exposure times were just above 500 N. These values including the peak bond strength were lower than those obtained using the fixed frequency facility. The apparent peak bond strength was only 82 % of that obtained with a power level of 400 W, using the fixed frequency facilities. It was only 80.5% of that obtained with a power level of 800 W and was marginally higher (3.5%) than its ambient cured rival.[5,10] In addition, the exposure times in VFM were much longer than its counterpart. At peak bond strength, the exposure time using VFMF was 4 times longer than that of its counterpart with a power level of 400 W. With the power level of 800 W, the value increased to 10.7 times. This was mainly due to the fact that the output power used in the VFM was only 150 W. Even if the maximum power output of 250 W was employed, the results did not improve significantly. Since the low power output made the processing time long, hence in microwave processing the power output played a significant role. Since the failure of the test pieces were at the parent material, it could be argued that the bondline strength should be more than 532 N.

Nylon 66/GF (33%) Processing Results

With this material, the processing was smooth as in the case of PS/GF (33%) and nothing special was noted.

Fixed Frequency Results of Nylon 66/GF (33%)

With glass fibre reinforced nylon 66, the peak bond strengths obtained were at exposure times of 35 and 55 seconds with power levels of 400 W and 240 W respectively. They were 32% and 28% respectively higher than those obtained by curing the adhesive at room temperature conditions but the times required were only 0.06% and 0.1% of their counterparts.[4,5,10] This material together with the adhesive seemed to couple with microwaves better than PS/GF (33%).

Variable Frequency Results of Nylon 66/GF (33%)

Figure 9 illustrates the bond strength of nylon 66/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. During most of the exposure period, the bond strengths of the test pieces were found to be above 1000N or shear strength of over 5 N/mm^2 and test pieces failed at bondline. At an exposure time of 35 seconds, the bond strength of the test piece joined by VFM was around 1005 N, 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 55 seconds, the bond strength using VFM was 1050 N, which was 9 % higher than that procured from its rival operating at 240 W. Figure 9 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 1305 N. Since the tensile strength of nylon 66/GF (33%) is 172.17 N/mm^2, there was plenty of room for improving its tensile shear strength without weakening the parent material due to the selective heating of VFM.[5]

Fixed Frequency Results for PS/CF (33%)

Test pieces of PS/CF (33%) were joined using the fixed frequency facilities without using primer.[18] With the power level of 240 W, the peak bond strength was 342 N and its time of exposure to microwave irradiation was 15 seconds. With the power level of 400 W, the peak bond strength obtained was 444 N with an exposure of 6 seconds to microwave irradiation. With the power level of 640 W, the graphite arced within a very short time of 7 seconds. Bond strengths with shorter time of joining were lower than those obtained from the 400 W power level. From the results of the two power levels, it could be argued that a power level of 400 W best joined the material because higher bond strengths could be achieved. The time taken to obtain those bond strengths was also shorter.

Variable Frequency Results for PS/CF (33%)

Figure 10 shows the bond strengths of PS/CF (33%) versus time of exposure. In tensile shear tests, all samples failed at the parent materials but their values were low as compared to the tensile strength of the original material.

The load required to break the original materials was 1108 N. Since the strength of the joined material was significantly reduced, it appeared that the main reason for its weakness was due to the excessive exposure to microwave irradiation. It could therefore be argued that up to certain limits, a better strength of the joined material could be achieved by reducing the time of its exposure to microwave energy.

Bond strength results of PS/CF (33%) versus power levels at 100 seconds of exposure to variable microwave energy are shown in Figure 11. Under tensile shear tests, all failures were at parent material. Their values were also very low as compared to the strength of original material. It seemed that the high level and long duration of microwave irradiation altered the parent material properties. Since 50 W was the minimum power that VFM could launch into the cavity, a better strength of the joined material could only be achieved by reducing the duration of exposure. However, there was also limit to this because a too short duration of exposure would result in incomplete welding and weak joint strength.

Fixed Frequency Results for LDPE/CF (33%)

With the power level of 240 W, it was found that the peak bond strength of LDPE/CF (33%) was 299 N and was at 10 seconds of exposure.[19] The failure was at the parent material. The strength was very low as compared with the strength of original material of 878 N. With the power level of 400 W, it was again found that the peak bond strength of 257 N was obtained at an exposure time of 7 seconds. Again, the strength of the parent material was weakened by the microwave energy.

Fixed Frequency Results for LDPE/CF (33%) Using VFMF

Joining tests of LDPE/CF (33%) using VFMF were performed. A fixed frequency of 2.5 GHz was intentionally chosen to make comparisons easier. From Figure 12 it can be found that the peak bond strength is 432 N at an exposure time of 18 seconds. The failures for the first two points were at bondline. The failures for the last two points were at the parent material. In the first two cases, incomplete bonding gave rise to weak bonds. On the other side of the coin, the parent material was weakened by excessive exposure to microwave energy. If the exposure time was made above 20 seconds, arcing of graphite took place and the test pieces were deformed.

Conclusions

The dielectric properties of PS and LDPE were not much different.[4,5,10,20,21] Since both materials had the same percentage of weight of carbon fibres as their reinforcements, it could be argued that PS/CF (33%) and LDPE/CF (33%) has similar dielectric properties as well. Neither the bond strength of PS/CF (33%) processed at variable frequency, nor that of LDPE/CF (33%) processed at fixed frequency in VFM seemed to reach the strengths of their parent materials respectively. Now consider the quality of bond brought about by processing them using different microwave facilities. Hot spots were found on the joint of LDPE/CF (33%) joined using the fixed frequency of 2.5 GHz chosen from VFM facility. The joint of PS/CF (33%) processed by variable frequency was perfect. It could therefore be argued that VFM could produce stronger bonds for the two materials, PS and LDPE, with excellent quality of joint properties.

Another noteworthy point with carbon fibre reinforced thermoplastic materials was that fixed frequency joining would not be pursued since the carbon fibre would arc and can give rise to thermal runaway thus resulting in deformed or even burnt samples.

The study also discovered that the power level was vital in joining thermoplastic composites irrespective of whether fixed or variable frequency microwave irradiation is used, and that for LDPE/GF (33%) the bond strength does not improve much with increasing the duration of microwave irradiation, irrespective of whether fixed or variable frequency microwave energy is employed.

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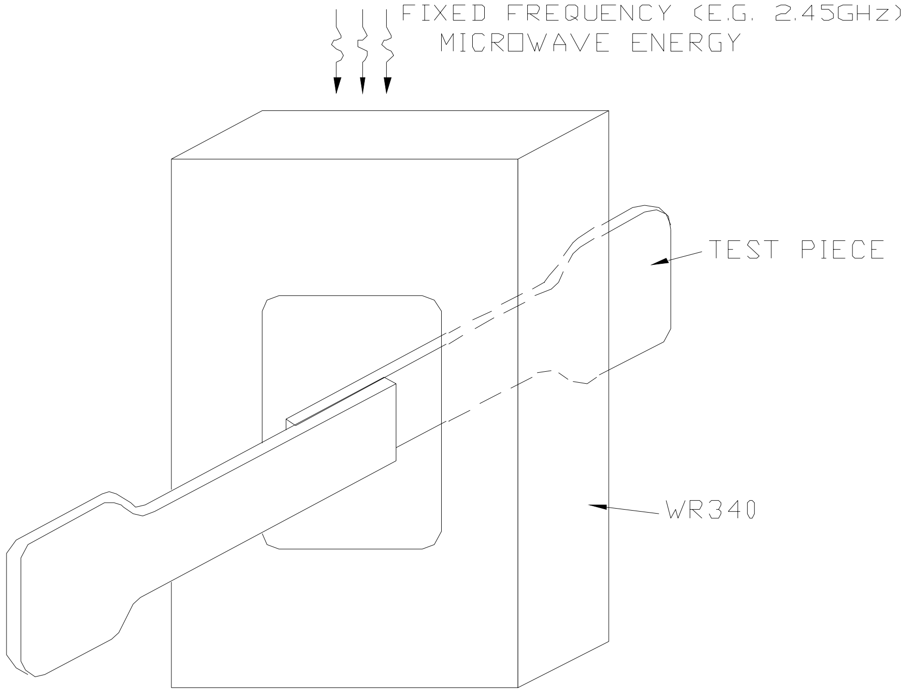
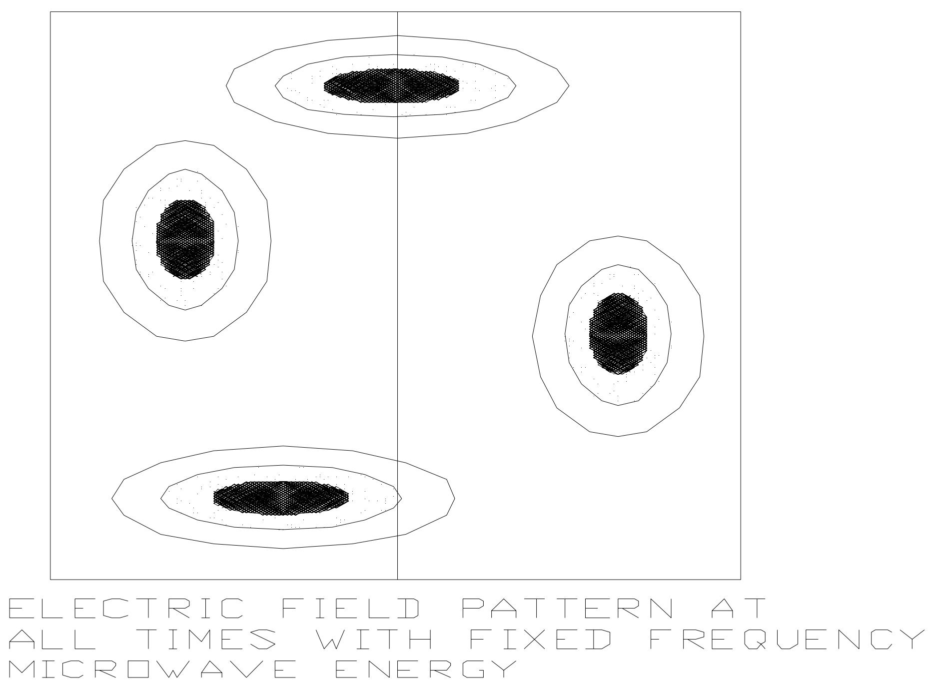
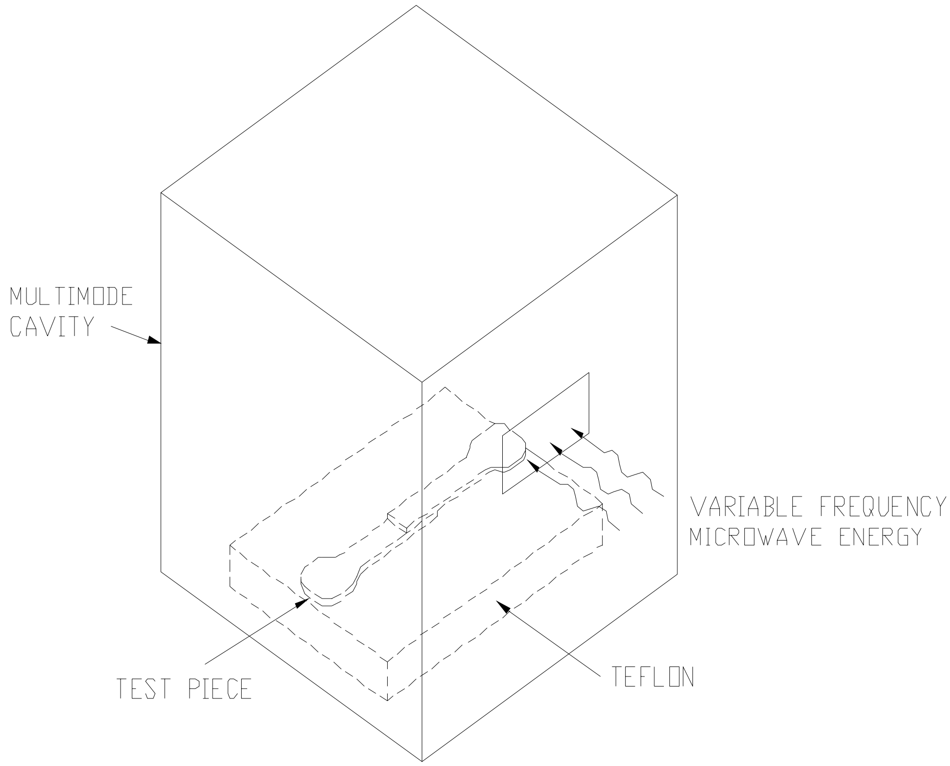


a) 2.45 GHz Microwave Energy Launched into a Single Mode Applicator

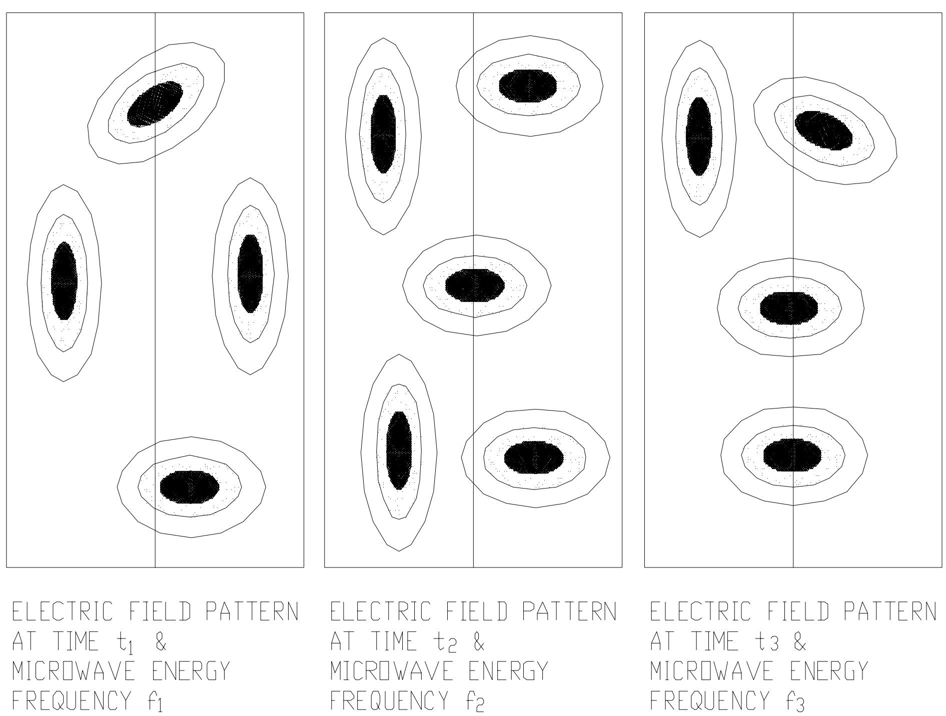


b) Electric Field Pattern for (a)

Figure 1: Fixed Frequency Microwave Heating – Nonuniform Heating



a) Variable Frequency Microwave Energy Launched into Multi Mode Cavity



b) Electric Field Pattern at Different Times in (a)

Figure 2: Variable Frequency Microwave Heating – Time-Averaged Uniform Heating

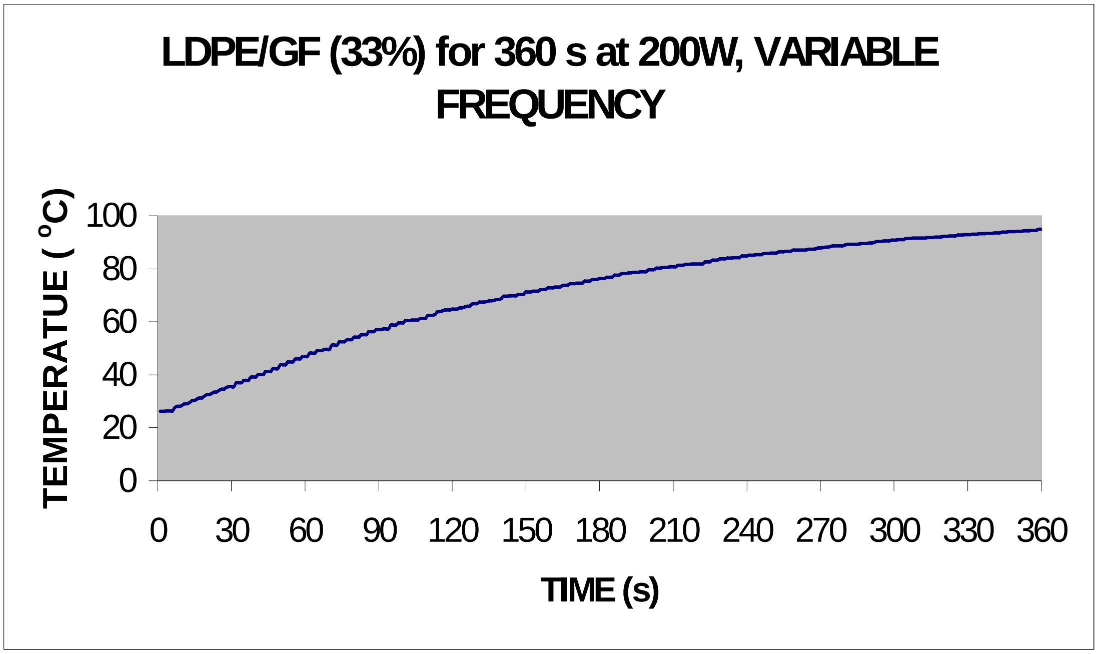


Figure 3: Temperature versus Time for LDPE/GF (33%)

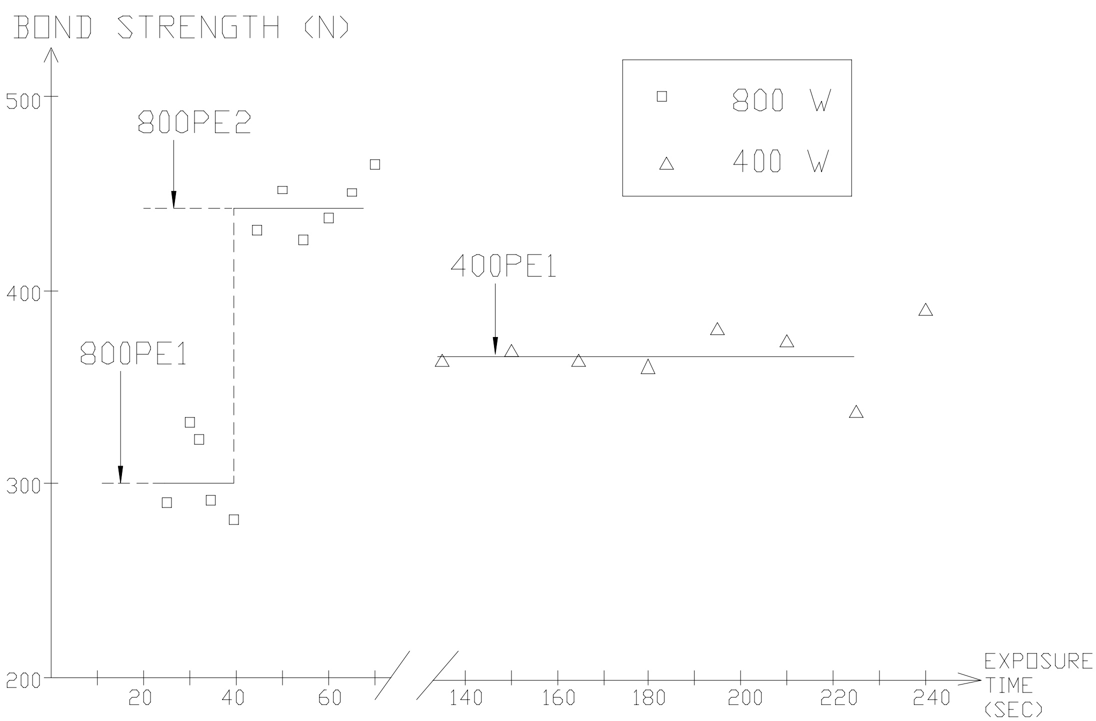


Figure 4: Bond Strengths of LDPE/GF (33%) and Five Minute Two Part Adhesive at 2.45 GHz

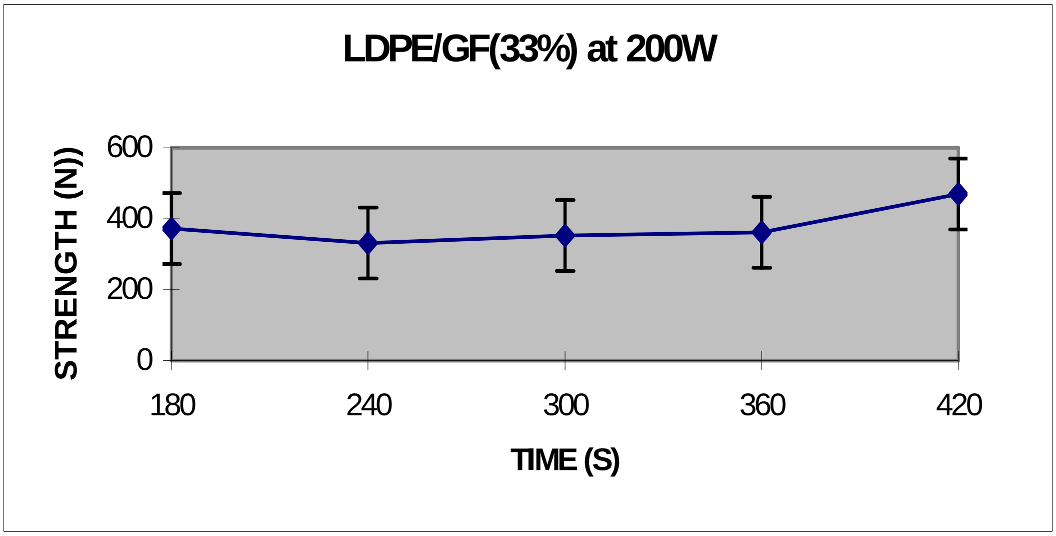


Figure 5: Bond Strength of LDPE/GF (33%) with Araldite Using Variable Microwave Frequency

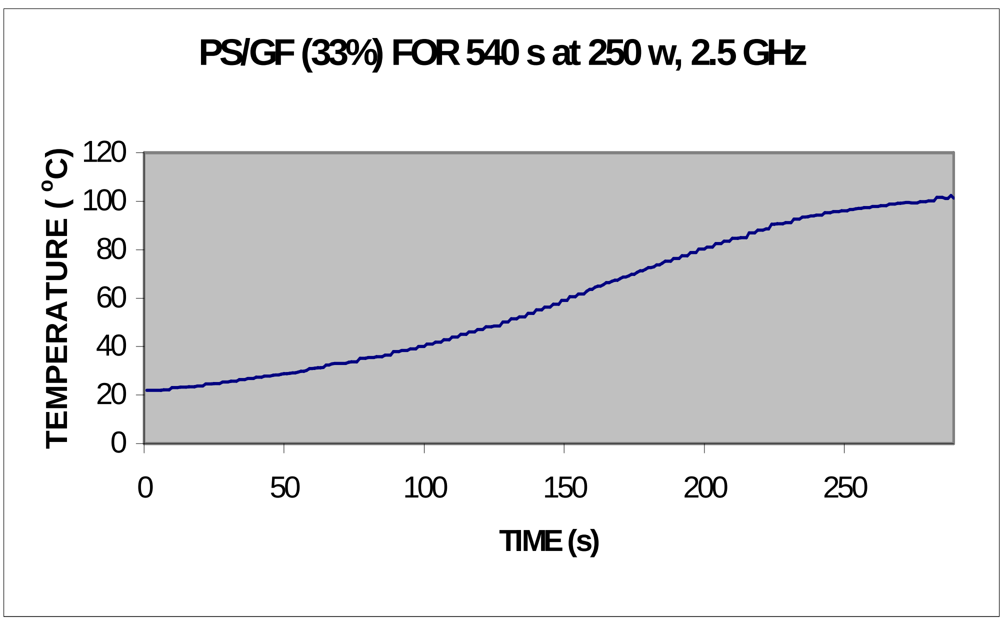


Figure 6: Temperature versus Time for PS/GF (33%) at 200 W

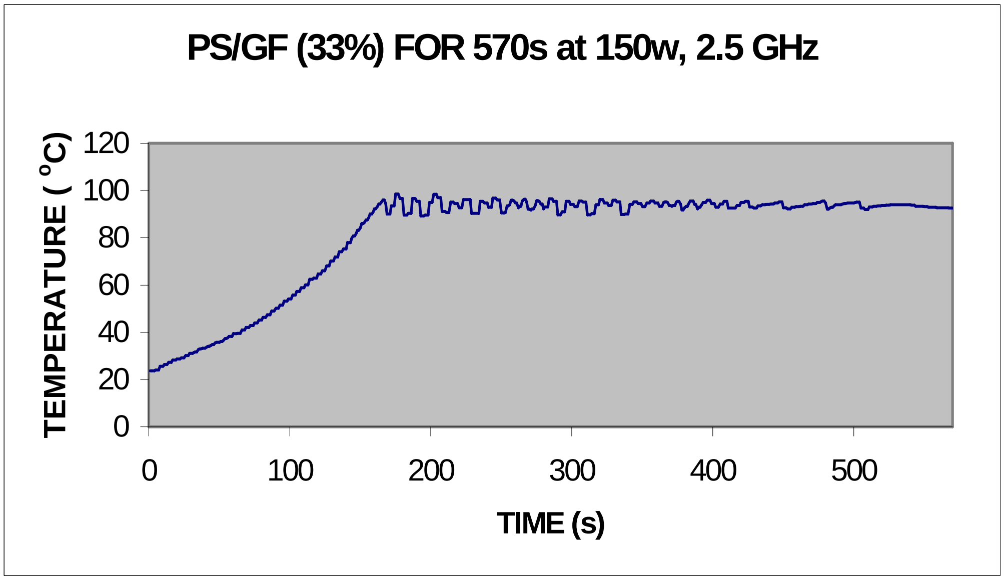


Figure 7: Temperature versus Time for PS/GF (33%) at 150 W using fixed Microwave Frequency

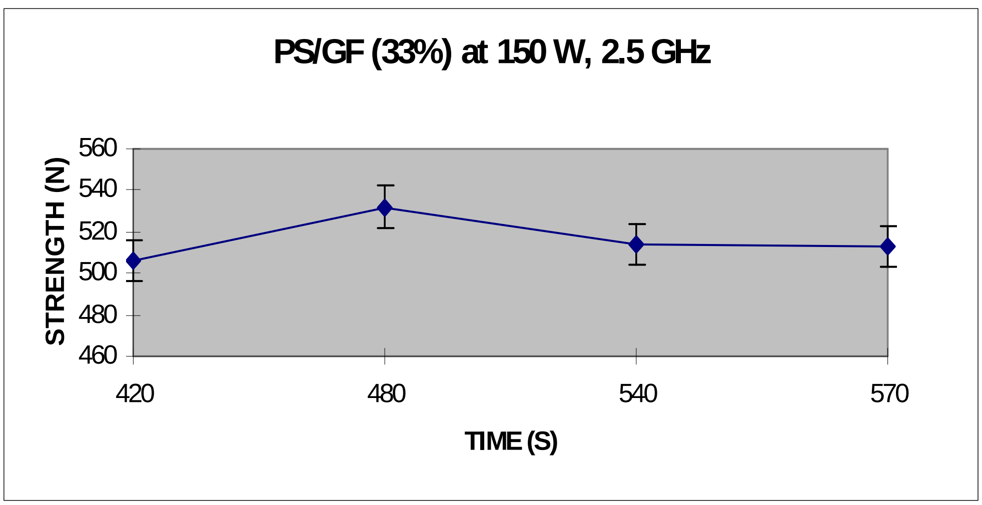


Figure 8: Bond Strength of PS/GF (33%) with Araldite Using Fixed Microwave Frequency

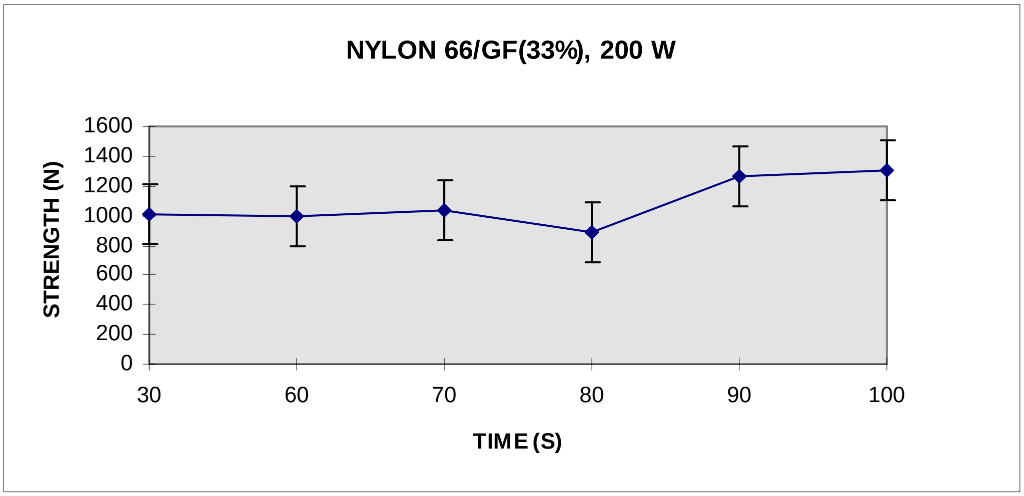


Figure 9: Bond Strength of Nylon 66/GF (33%) with Araldite Using Variable Microwave Frequency

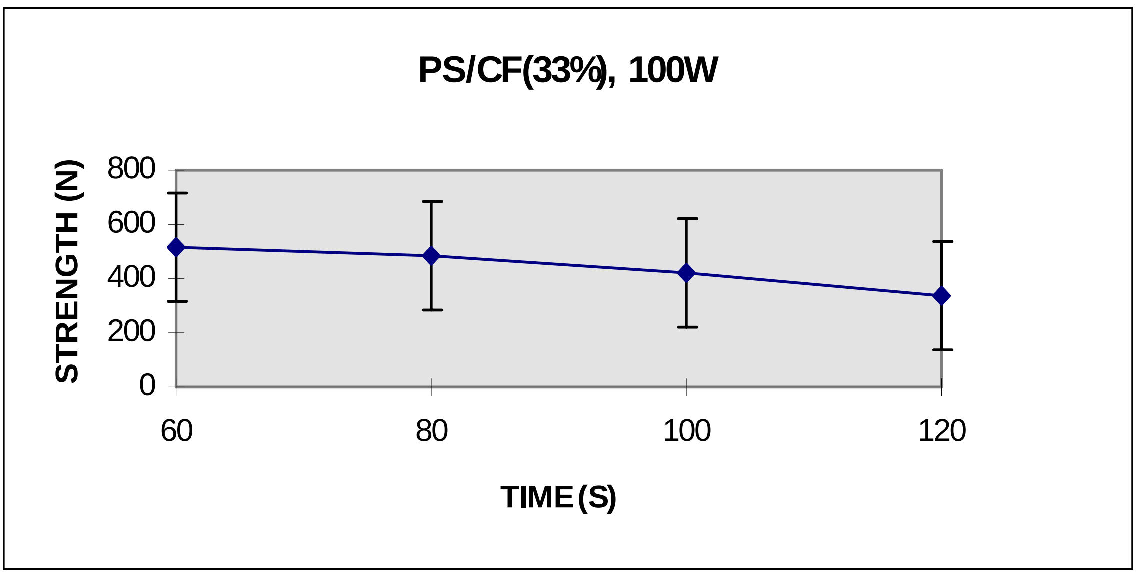


Figure 10: Bond Strength of PS/CF (33%) with No Primer Using Variable Microwave Frequency

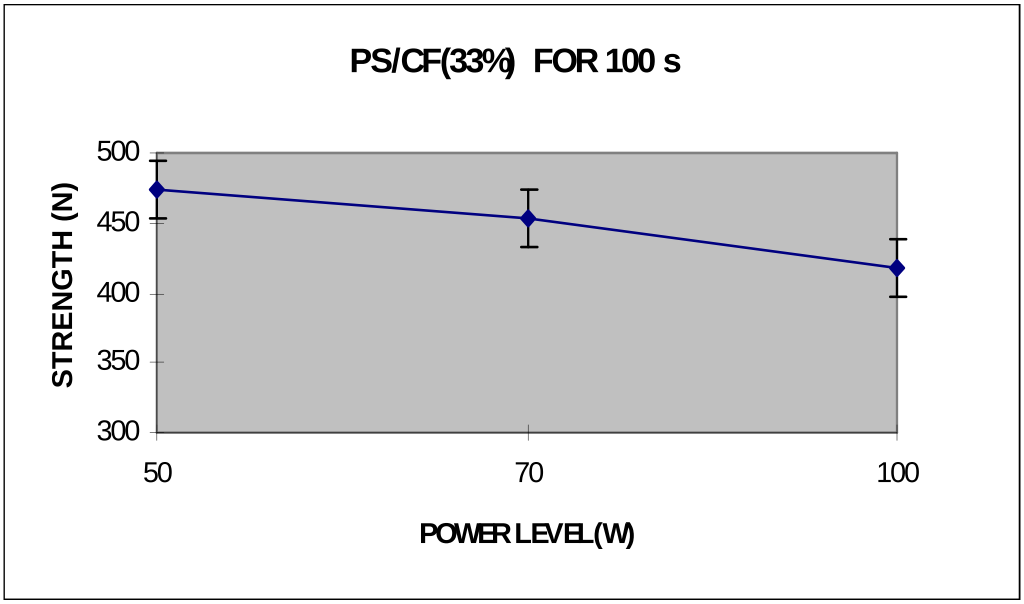


Figure 11: Bond Strength of PS/CF (33%) Using Variable Microwave Frequency and Different Power Levels

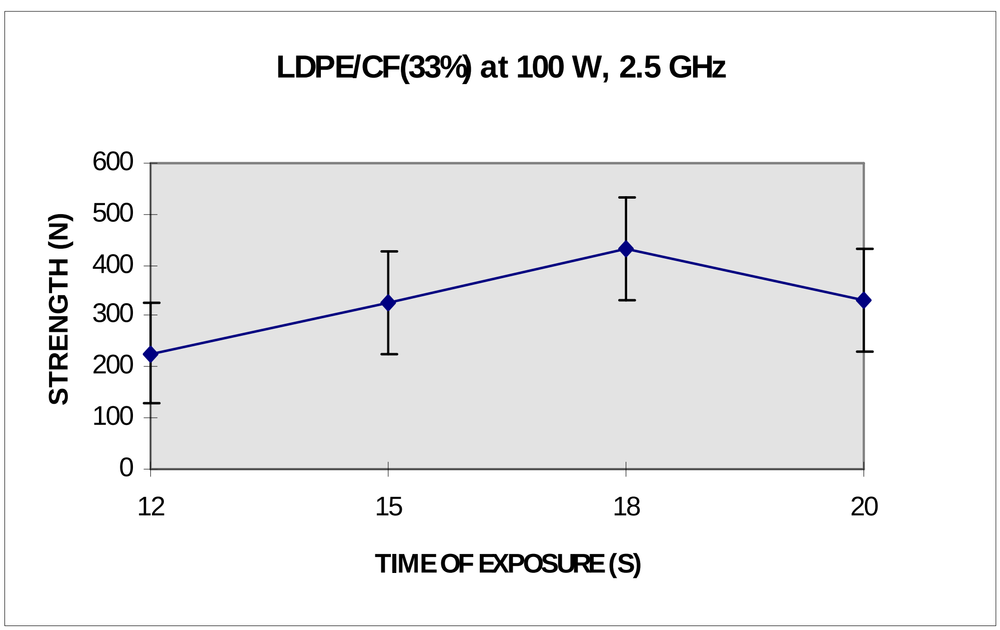


Figure 12: Bond Strength of LDPE/CF (33%) with No Primer Using Fixed Microwave Frequency