Heat distribution and thermal analysis of joining thermoplastic composites using microwave energy

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Short title: **Thermal analysis of joining composites**

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Abstract: Industrial microwave technology for processing polymers and polymer-based composites is currently in a state of considerable flux. In this study, a microwave facility consisting of a 0.8 kW variable control power generator operating at 2.45 GHz is used to join random glass or carbon fibres reinforced thermoplastic composites. The 2.45 GHz microwave was selected for the study because the magnetron at this frequency is readily avalable at low cost. The input power can be varied in four steps. A cycle time of 30%, 50%, 80% and 100% of full power can be selected. The heat absorbed and heat flow in the sample materials are studied. The temperatures at different points of the samples are also measured using infrared thermometer. The effect of power input and cycle time on the temperature distribution in the test piece is detailed together with the underlying principles of sample material interactions with electromagnetic field. The material used for the research is 33% by weight of random glass fibre reinforced polystyrene [PS/GF (33%)]. Araldite has a high loss tangent and can absorb microwave energy and convert it into heat much better than the thermoplastic, polystyrene and the glass fibre. The samples were therefore lapped joined using Araldite as primer. The Araldite consists of 100% liquid epoxy and 8% amine. Computer software package (MATLAB 6) is utilised to analyse the heat distribution in the samples and its relation to the lap shear bond strength of the joints obtained. It is found that sufficient combination of power level and duration of exposure to microwave irradiation is required for obtaining proper bonding with good strength. It is also discovered that proper bonding will only form when the rise in temperature is significant, ie the sample temperature should be above 30 ^{o}C, at which the Araldite will cure quickly and properly. Based on these findings, the lap shear bond strength of the test pieces after microwave joining can be predicted by reading at the temperatures reached by the samples without carrying destructive testing.

Keywords: complex relative permitivity, loss tangent, random glass fibre-reinforced polystyrene, lap shear bond strength and microwaves.

1. Introduction

High energy rate joining of thermoplastic composites using microwave was studied because it was believed that the microwave/materials interactions of some of thermoplastic composites with and without fillers (Araldite) will favour the process. In microwave processing of materials, most of the heat absorbed by the test pieces is due to the absorption of microwave irradiation and then conversion of the microwaves into heat by the samples.

Ku et al. [1-3] used the equipment shown in Figure 1 for this study. Menumaster is the trademark of the microwave oven used. Its cavity dimensions are 500 mm x 700 mm x 500 mm. Referring to Figure 1, the microwaves generated from the magnetron are guided through WR340 waveguide to the test pieces. Avoiding radiation leakage is of primary concern and the joining process is enclosed within a microwave oven cavity so that microwaves will not radiate to the environment.

With reference to Figure 1, the incident waves generated by the magnetron travelled downwards through three sections of WR340 waveguide and interacted with the test pieces located in the second section before being reflected back by the top face of the adjustable plunger. The plunger was designed and manufactured to have a sliding fit contact with the waveguide. The interaction between the incident and the reflected waves set up a standing wave and as desired the maximum electric field occurred at the seam of the lapped test pieces. This was achieved by adjusting the moveable piston so that its top face was an odd multiple of $\lambda_g/4$ from the centre of the slit, λ_g being the wavelength within the waveguide. The plunger can be moved up and down by rotating the knob at the bottom of the plunger. The sliding fit between the plunger and the waveguide would permit the leakage of microwaves to the surrounding. The problem was solved by the use of short circuit plunger. After joining by microwaves with Araldite as primer, the joints of the PS/GF (33%) test pieces were lap shear tested, using a Shimadzu tensile testing machine. The lap shear strengths of the composite material, the details of the plunger used and the risks involved in the joining process can be found in a paper published earlier [4].

In a university based establishment in Queensland, Australia, Siores et al. used a ridge waveguide (shown in Figure 2) to join thermoplastics successfully; they joined PMMA (polymethyl methacrylate) and HDPE (high desity polyethylene) with professional Araldite, Araldite and RS High Strength Epoxy as primer but found that Araldite was the best primer [5].

2. Theoretical backgrounds

2.1 Materials microwaves interaction considerations

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, $\tan \delta = \varepsilon''/ \varepsilon'$. The real part of the permittivity, ε', sometimes called the dielectric constant, mostly determine 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 $\tan \delta$, to convert microwave energy into thermal energy [6].

Random GF reinforced (33%) PS was selected because the material has a similar value of loss tangent to that of random GF reinforced (33%) low-density polyethylene (LDPE) and it would be convenient to compare the results of the two materials [1, 2, 6]. The composite is not readily available in the market and it was specially manufactured in Plastic and Rubber Technical Education Centre (PARTEC) in Brisbane, Australia. The length of the reinforcing fibre was 6 mm or a little bit less as fibres longer than 6 mm will not improve the strength of injection-moulded test pieces. However, typical lengths of fibres used in reinforced injection moulding materials were 0.8 to 25 mm [7].

2.2 Test piece microwave interaction results

The two mirror image test pieces with dimensions in millimetres were cut using a band saw from a standard tensile test piece for composite materials. The lapped area was made 20 mm x 10 mm. The lapped areas were first roughened by rubbing them against coarse, grade 80, emery paper. They were then cleaned by immersing them in methanol and allowed to dry in air before applying 1.5 to 2 cubic millimeres of Araldite onto both surfaces. After applying the filler, the two pieces were tightened by a dielectric (rubber) band, which encircled the lapped areas four times as depicted in Figure 3. This is to fix the relative position between the two test pieces and to apply pressure onto the lap joint. The pressure on the lap joint was estimated to be 4 N/cm^2 and it was critical as the bond strengths of the test pieces cured by leaving them in ambient conditions for one hours with and without the rubber band pressure were 306 N/cm^2 and 168 N/cm^2 respectively [8]. 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 4. The dielectric band was made to push the two pieces when the interface was melted by microwave energy and joined them together. The test pieces were then exposed to two different power levels of 400W and 800W with varying time of microwave exposure.

3. Results

3.1 Temperature distribution

In most cases, only the parts smeared with filler were warmed or heated depending on the power level used and the time of microwave exposure. The parts not smeared with Araldite had much lower loss and did not absorb much of the microwave irradiation. After bonding, the temperatures at different locations, noted by E_{L4}, E_{L3}, E_{L2}, E_{L1}, E, E_{R1}, E_{R2}, E_{R3}, E_{R4} (Figure 5) were measured using infrared thermometer. E is the mid point of the lapped test pieces with E_{L1} and E_{R1} are at 10 mm from left and right of E respectively. Similarly, E_{L2} and E_{R2} are at 20 mm from left and right from E respectively and so on for E_{L3} and E_{R3} and E_{L4} and E_{R4} respectively. Referring to Figure 5, microwaves travelled from the top of the test pieces. The hottest spots of the sample were expected to be on the lapped area and along points, E_{L2}, E_{L1}, E_{R1} and E_{R2}. This is because the lapped area contained the applied Araldite, which absorbed the microwave energy more and converted it into heat. Figure 5 shows the temperature distribution of samples exposed to different duration of microwave irradiation of 800 W. The distribution of electric fields along the cross-section of the WR 340 rectangular waveguide had a similar shape to that of the curves in Figure 6 [13] . The test pieces were plunged into the curve with the highest electric field in line with the samples. The electric field along the test pieces was therefore strongest. At an exposure time of 60 seconds, the recorded temperatures for points E_{L1}, E and E_{R1} were 32 ^{o}C, 33 ^{o}C and 31.5 ^{o}C respectively. The ambient temperature was 21 ^{o}C. The oven cavity temperature after bonding for 60 seconds of microwave exposure was 27 ^{o}C. The mid-point of the sample, point E, was hottest and it was 12 ^{o}C higher than the room temperature. The longer the duration of exposure to microwave energy, the higher the temperatures of the points as depicted in Figure 5. The two points adjacent to the midpoint, E, ie, E_{L1}, E_{R1} also recorded significant temperature rise. Furthermore, the longer the time of exposure of the sample to microwave energy, the greater the temperature difference between E and E_{L1}, and E and E_{R1} respectively. This is illustrated by the more acute the angle $E_{L1}EE_{R1}$; at shorter duration of microwave irradiation, the angle was obtuse, ie, there was not much temperature difference between E and E_{L1}, and E and E_{R1} respectively (see the 15 second-exposure in Figure 6) but the temperatures were much higher than the ambient temperature. The temperature of points outside the lapped area, ie, E_{L4}, E_{L3}, E_{L2} on the left and E_{R2}, E_{R3}, E_{R4} on the right were also much higher than the ambient temperature. The rise in temperature might have been due to the heat conducted from the lapped area where the Araldite had absorbed more microwave energy. It can also be noted that the temperatures of the points to the left of the lapped area were more or less the same to those on the right because the test pieces were inserted into the rectangular waveguide through the slit symmetrically and the electric fileds inside the waveguide were also symmetric.

Figure 7 shows the temperature distribution of samples exposed to different duration of microwave irradiation of 400 W. The temperature distribution along the points considered is similar to that of the 800 W microwave power exposure in Figure 6. The temperature of points outside the lapped area, ie, E_{L4}, E_{L3}, E_{L2} on the left and E_{R2}, E_{R3}, E_{R4} on the right were also much higher than the ambient temperature. This is expectable and the reason is the same as in the case of 800 W microwave irradiation mentioned above.

3.2 Heat flow and temperature gradient

Figure 8 shows heat flow lines, which spread out from the centre of the test pieces. The temperature did not change uniformly because the ends were not insulated. Bisect the test pieces along the point E and consider the right hand side of them, for two positions along the sample separated by distance dx, the average temperature gradient between the two positions is $\frac{d\theta}{dx}$ where dθ is the temperature difference between the two positions. The heat flow, $\frac{Q}{t}$, along the sample depends on [9]:

i) the temperature gradient $\frac{\theta_1 - \theta_2}{L}$ along the sample;

ii) the cross-sectional area of the sample and

iii) the material of the test piece.

To measure heat flow, the heat energy Q conducted along the test piece in time t must be measured.

The specific heat capacity of PS/GF (33%), using the techniques mentioned in a text, was measured to be 1086 $Jkg^{-1}K^{-1}$ [9]. By referring to Figure 4, the total energy, Q, absorbed by the test pieces during their exposure to microwave irradiation can be estimated by dividing the test pieces into sections of different temperatures. Consider the section of E and E_{R1} of PS/GF (33%), the temperature of E and E_{R1} after exposing to microwaves of 800 W for 60 seconds were 33.5 ^{o}C and 31.5 ^{o}C respectively. Their average temperature was $\frac{33^{o}C + 31.5^{o}C}{2}$ =32.25 ^{o}C. The volume of the section = 10 mm x 10 mm x 3 mm x 2 (lapped area) = 600 mm^3. The volumes and average temperatures of the other sections of the test pieces were similarly calculated and were tabled in Table 1. The mass of the test pieces was 9.80 g. Since the total volume of the test pieces was 6200 mm^3 or 6.2 cm^3, the density of LDPE/GF (33%) = $\frac{mass}{volume} = \frac{9.80g}{6.2cm^3}$ = 1.58 g/cm^3.

The mass of section E and E_{R1} = volume x density = 0.6 cm^3 x 1.58 g/ cm^3 = 0.95 g.

The microwave power absorbed =

(mass) x (specific heat capacity) x (rise in temperature)

= 0.95g x 1086 $Jkg^{-1}K^{-1}$ x [(32.25 +273) K – (21 +273) K] = 11.583 J

The mass and energy absorbed of other sections can be similarly calculated and are shown in Table 2. The total energy absorbed by the test pieces was the sum of energy absorbed by each section and was 86 J.

The heat energy stored in the section E_{R4} and the end of the test piece on the right hand side was 17.845 J, this rise in temperature in the samples was due to heat energy flow from E to the same end of the test piece. Despite the low loss of the composite material, PS/GF (33%), the heat stored in the test pieces came overwhelmingly from the microwave absorption of the samples smeared with Araldite.

The temperature distribution along the half test piece for 60 seconds exposure to microwaves of 800 W (in Figure 6) was split into two parts. One to the left of point E and the other to the right of it. Two polynomials were obtained using MATLAB 6 to represent temperature distribution to the left and right of point E. MATLAB 6 was chosen for obtaining the three equations because it is readily available in the university.

To do this in MATLAB (for Equation 1) first define the variables l and T, using MATLAB's automatic linear interpolation. The Langrange polynomial will be called P and it will be constructed piecemeal, beginning with P=0;. Then for each data point the coefficients for the corresponding L_k (base) polynomial will be constructed as follow:

```
l=[-66 –40 –30 –20 –10 0];
T=[28 28 28.5 31 32 33];
```

```
P=0
for k=1:6
u = l(l~=l(k));              % pick pout the values of $l$ other than $l_k$
p = [1, -u(1)];              % first factor in the polynomial product
q = l(k) –u(1);              % first factor in the denominator
for j=2:5
p = conv (p, [1, –u(j)]);    %  polynomial multiply by each successive factor
q = q* (l(k) –u(j));         % multiply denominator by each successive factor
 r = p/g;                    % coefficients of the base component polynomial
end
 P = P + T(k) *r;            % The Lagrange polynomial
end
 fprintf('%1.8f',P)
```

The coefficients of one of the polynomial (Equation 1) are 0. 00000052, 0.00007325, 0.0034, 0.056623, 0.40079 and 33. The polynomial is therefore,

$$T(l) = 0.00000052x^5 + 0.00007325x^4 + 0.0034x^3 + 0.056623x^2 + 0.40079x + 33$$

Similarly, Equation 2 is

$$T(l) = -0.00000052x^5 + 0.00007325x^4 - 0.0034x^3 + 0.056623x^2 - 0.40079x + 33$$

With reference to Figure 8, Equation 1 and Equation 2 are not linear and they represent the change in temperature with positions along the test pieces to the left of the centre point, E and to the right of it respectively. The slope of polynomials at a particular point along the sample represents the temperature gradiant on that location.

From these two equations, the temperature of a particular location along the samples can be easily computed. Equations 1 and 2 for other duration of exposure to microwaves at power levels of 400 W and 800 W can be similarly obtained. In addition, by substituting the values of maximum temperature at positions E (see Figure 5) for each duration of exposure to *CONV* function in MATLAB, a polynomial, Equation 3 = $-0.0000087x^5 + 0.000160x^4 - 0.01115x^3 + 0.3633x^2 - 5.4x + 0.955$ for finding the temperature at location E in the test pieces and at a particular duration of exposure can be obtained. By using Equation 3, and Equations 1 and 2 for different duration of exposure, the temperatures along the samples at a particular time of exposure can be estimated.

3.3 Lap shear bond strengths

A load range of 2000 N and a load rate of 600 N per minute were selected for the lap shear test [10]. Figure 9 shows the lap shear strength of PS/GF (33%) joined by a fixed frequency microwave facility in a slotted rectangular waveguide. The primer used for joining this material was also five minute two part adhesive. 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 lap shear strength, 326 N/cm^2, at this exposure duration exceeded that obtained by ambient conditions curing by 17%, but the time required was a mere of 3.2% of its counterpart [1, 3]. For exposure times of one and a half to four and a half minutes, the lap shear strengths 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 lap shear strength was 331 N/cm^2 and was achieved when the exposure time was 45 seconds and it exceeded the ambient conditions cured lap shear strength by 19 %, but the time required was only 1.28 % of its rival [11, 12]. 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 [3].

Figure 10 shows the relationship of lap shear bond strengths and temperatures of the centre points of the test pieces with respect to the duration of exposure to 800 W microwave irradiation. At all duration of exposure to microwaves, the temperatures of the centre points of the test pieces increased steadily with the increase in time of microwave exposure; the lap shear strength of them showed the same trend initially but when the duration of exposure was over 50 seconds, the values of the lap shear strength fell and this was due to over-cured of the Araldite [1].

Figure 11 shows the relationship of lap shear bond strengths and temperatures of the centre points of the test pieces with respect to the duration of exposure to 400 W microwave irradiation. The temperatures of the centre points of the test pieces increased steadily with increase in microwave exposure and the lap shear strength showed the same trend but flattened and declined slightly when the exposure time was over 120 seconds. Exposure times of over 300 seconds will deform the samples. The selected cycle times were the most optimum ones because they gave good quality bond with high strength [1-4].

4. Conclusions

The potential benefits of the technology will speed up the replacement of thermosetting resins by advanced thermoplastic composites in the structural parts of aeronautical, military and recreational industries. The constituent elements of the composite, PS with loss tangent at

2.45 GHz = 5.3 x 10^{-4}, and GF with loss tangent = 5.3 x 10^{-5} are low loss material and it is therefore expected that the composite itself, PS/GF (33%) with loss tangent at 2.45 GHz = 3.73 x 10^{-4} is a low loss material as well [3, 13]. The Araldite with loss tangent at 2.45 GHz = 0.244, therefore plays a vital role in absorbing microwave energy and converted it into heat and cures itself rapidly [14]. The absorption of microwaves and hence the rise in the temperature of the samples did promote the curing of the Araldite.

Power level of microwaves used and the duration of exposure of the samples to microwave irradiation also play important role in the heating and curing of the Araldite. With reference to Figure 10, it can be noticed that if the power level of microwaves used is relatively low, say, 400W, the exposure duration must be long, otherwise no bonding will take place. In this case, no proper joint could be obtained if the exposure time was less than 65 seconds [2]. On the other hand, if the power level used is relatively high, say, 800 W, the exposure duration can be shorter, but it must still be up to certain value, otherwise, bonding will not occur properly. This is clearly illustrated in Figure 9. When the exposure duration was less than 23 seconds, the Araldite could not be fully cured even if a rise in temperature in the sample was recorded. The resulting bond strength was weak, even weaker than test pieces with Araldite cured under ambient conditions. From the above observation, it can be argued that the bond in the lapped area is going to form properly only if the rise in temperature is significant enough to cause the complete curing of the Araldite in a short time. By observing the relationship of lap shear strengths, temperature at centre points and duration of exposure to microwave irradiation in Figures 10 and 11, one can deduce that when the temperature on the centre of the sample is over 30 ^{o}C, the Araldite will have been cured properly and quickly, and the resulting bond strength will be good. However, it must be noted that too high a temperature is not welcomed because the primer will be over-cured and the lap shear strength will be weakened. In addition, the dielectric band used to apply pressure on the lapped area of the test pieces will also deform them because at higher temperatures the samples will be very soft and can be deformed with ease [1, 2]. This study enables one to predict the quality of the bond strength obtained in joing two pieces of thermoplastic composite samples by microwave irradiation without destructing the test pieces but by measuring the temperatures reached by the samples.

It should, however, be noted that the combination of cycle times and power levels obtained in the experiments gives only an indication on how to apply this knowledge universally. The effective combination of the two factors depend on the magnitude of power source as well as the effeciency of the applicators. In addition, the loss tangent of the materials will also play an important part in the cycle times as well as the power levels used in carrying out the bonding.

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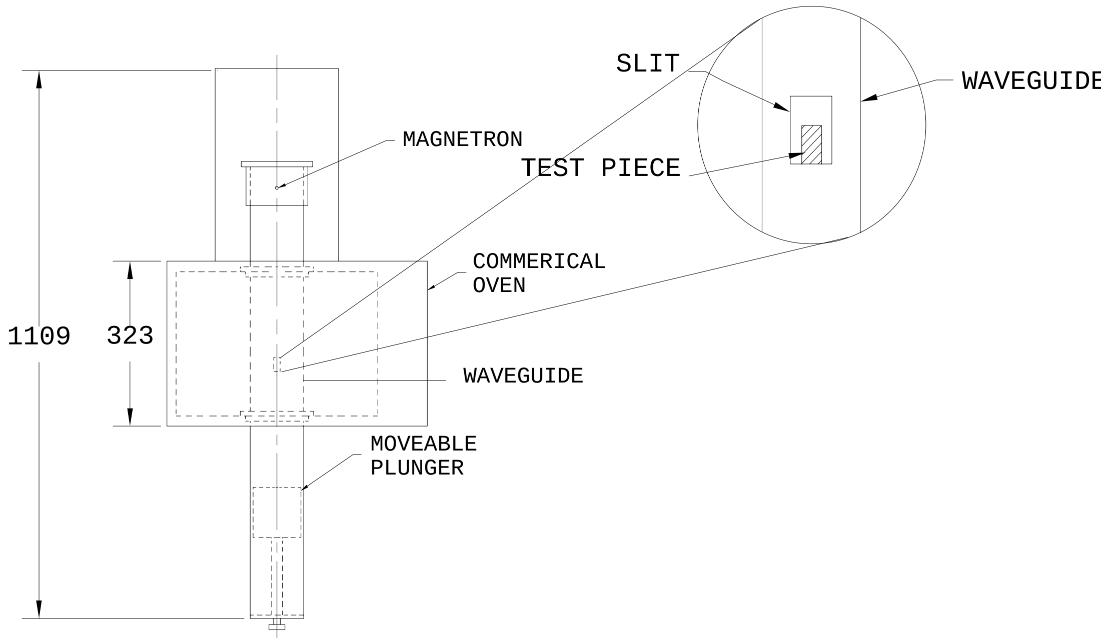


Figure 1. Microwave Facilities Configuration

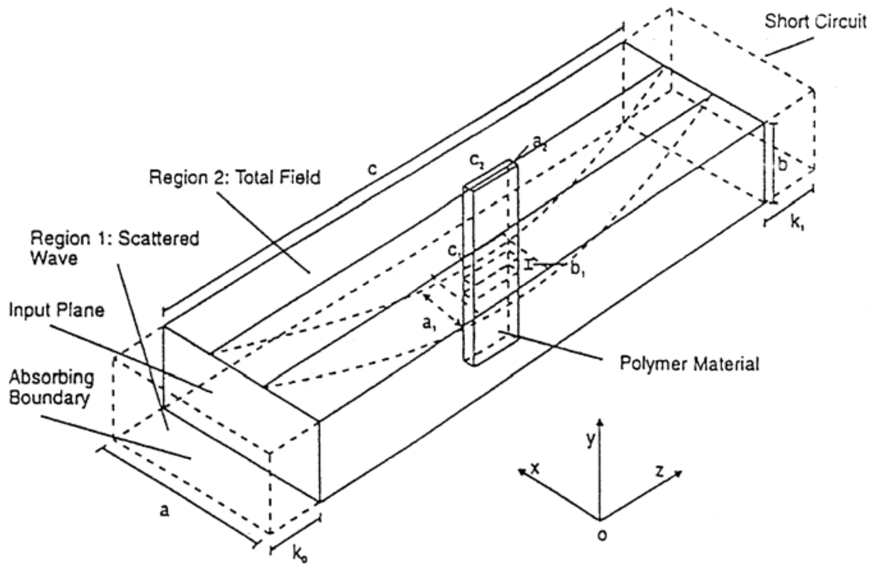


Figure 2: A Ridge Waveguide for Microwave Heating of Polymer Materials

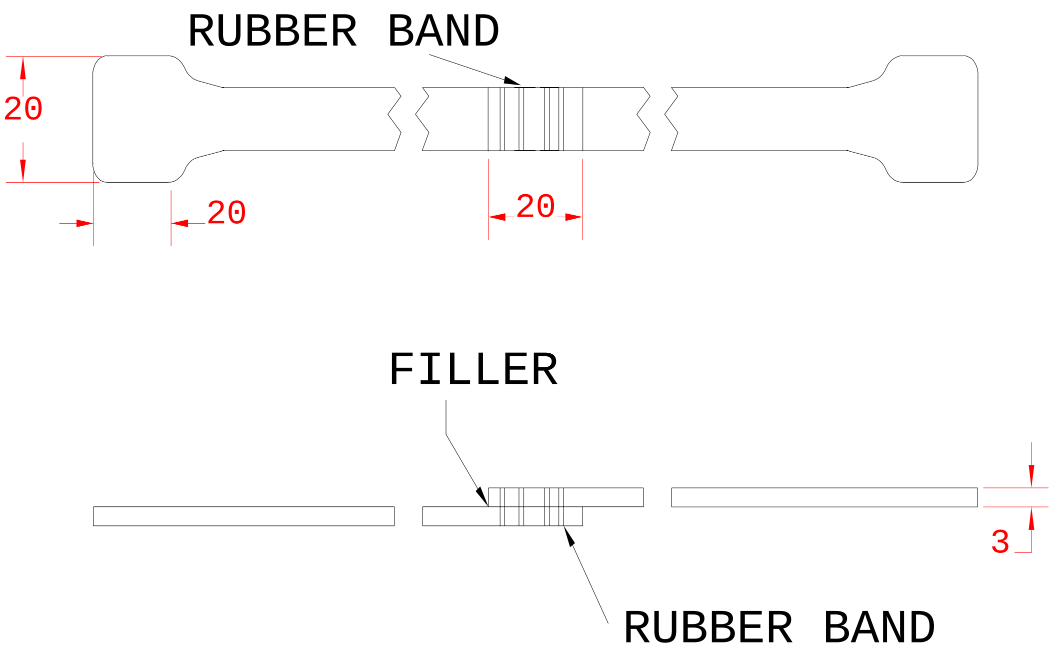


Figure 3: Test Pieces tightened by a Dielectric Band

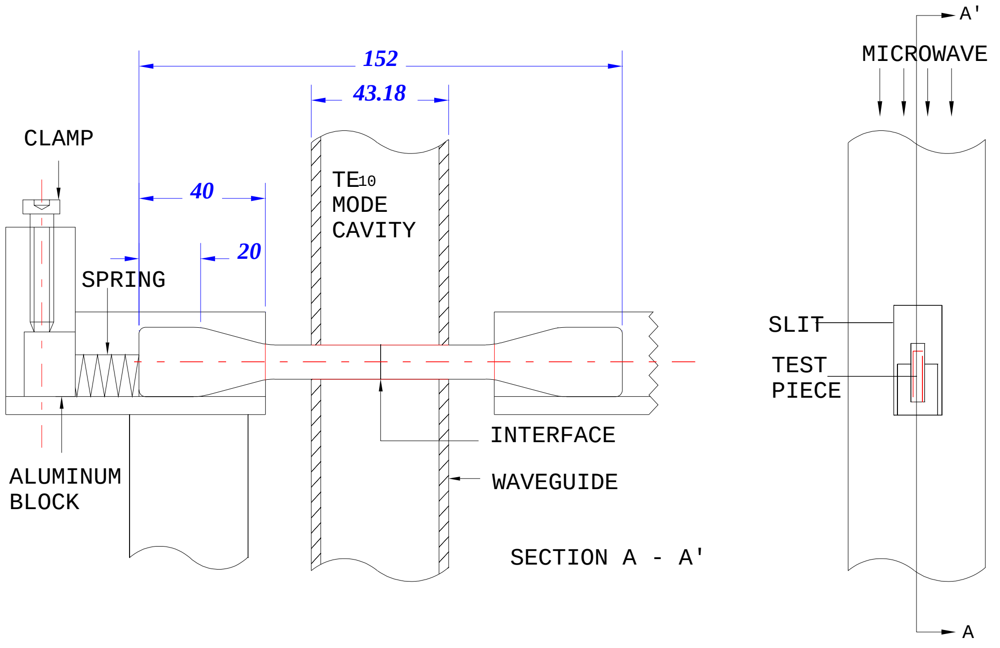


Figure 4: Test Pieces in Position

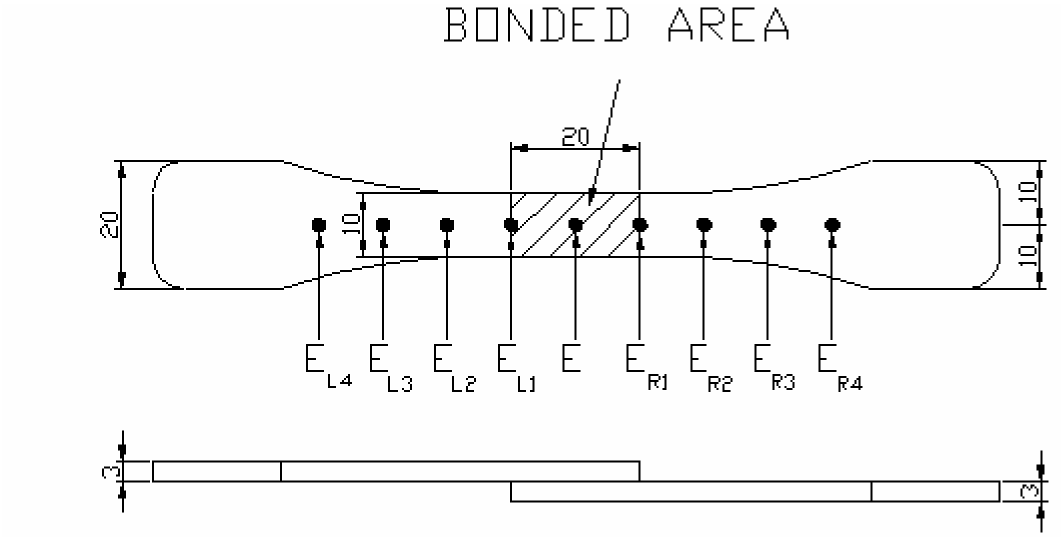


Figure 5: Locations at which temperature measurements (in the sample) are taken

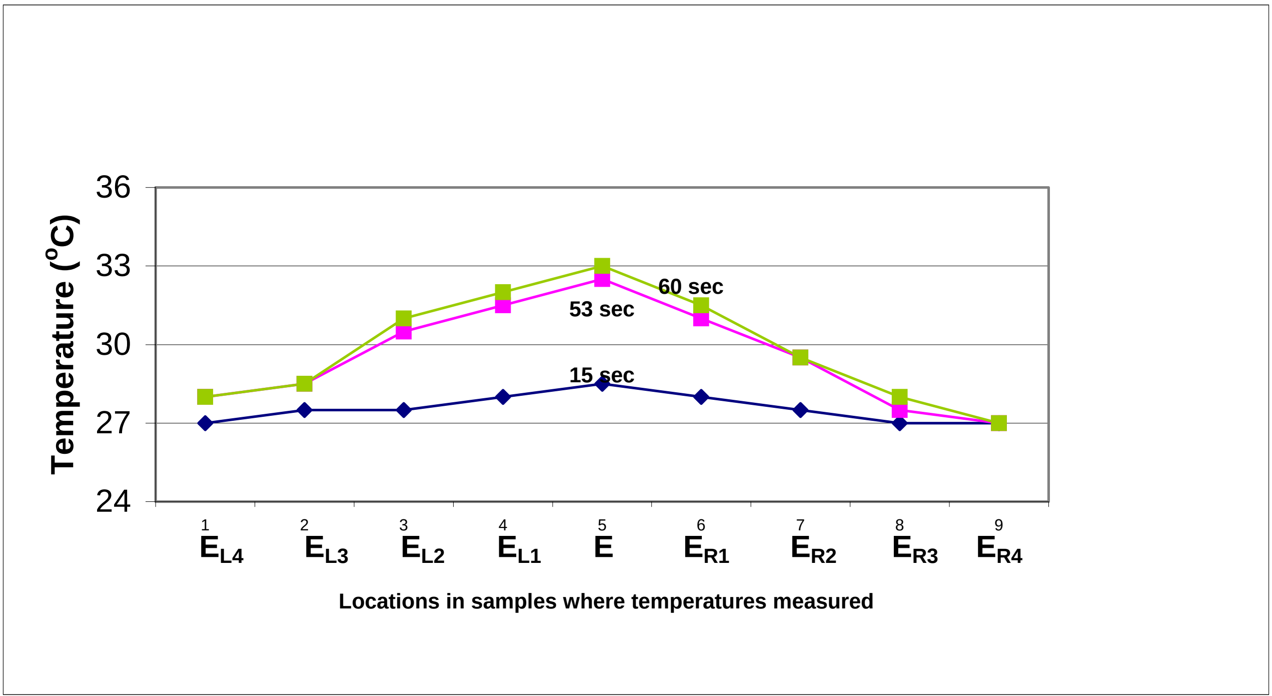


Figure 6: Temperature at different locations in samples with different exposure duration to 800 W microwave irradiation

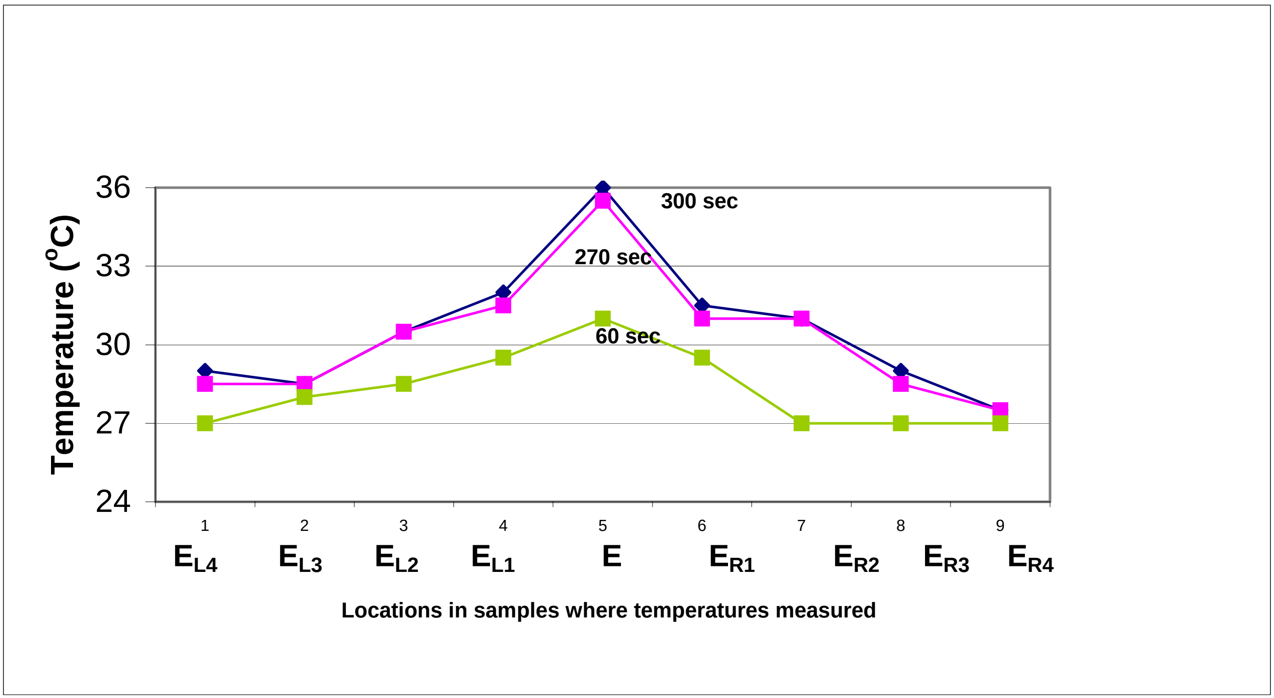
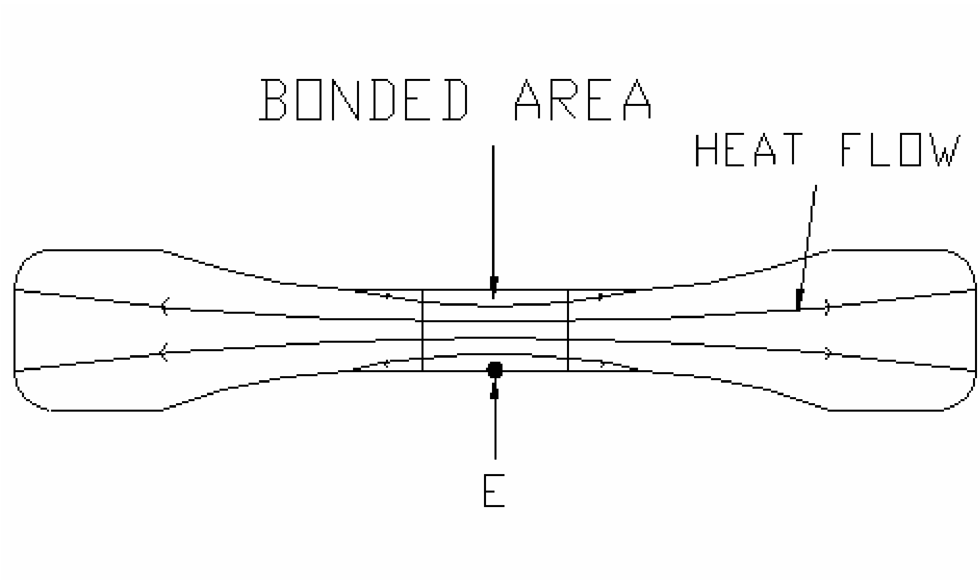


Figure 7: Temperature at different locations in samples with different exposure duration to 400 W microwave irradiation



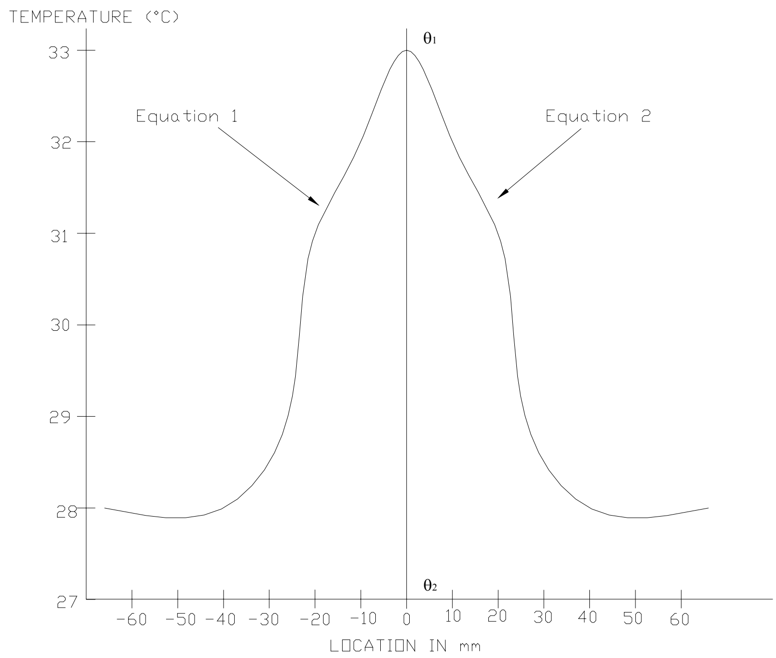


Figure 8: Heat flow and temperature gradient of test pieces exposed to a power level of 800 W and a duration of 60 seconds.

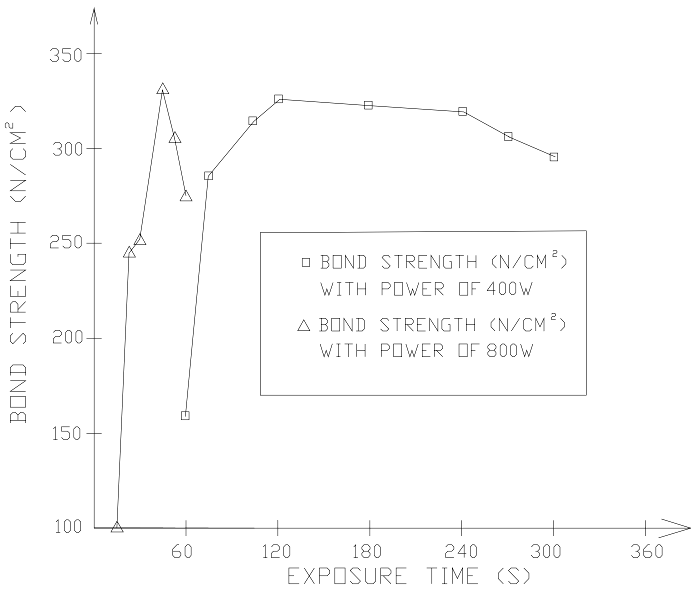


Figure 9: Lap Shear Strength of PS/GF (33%) Joined by Fixed Frequency Microwave (2.45 GHz) in a Slotted Rectangular Waveguide using Rapid Araldite

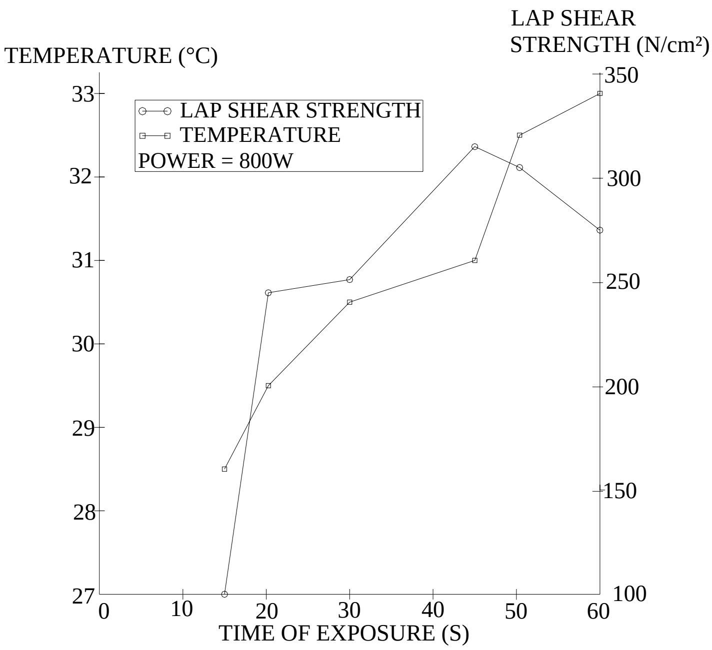


Figure 10: Lap shear bond strength and temperature against time of exposure to microwaves of 800W in the samples of PS/GF (33%)

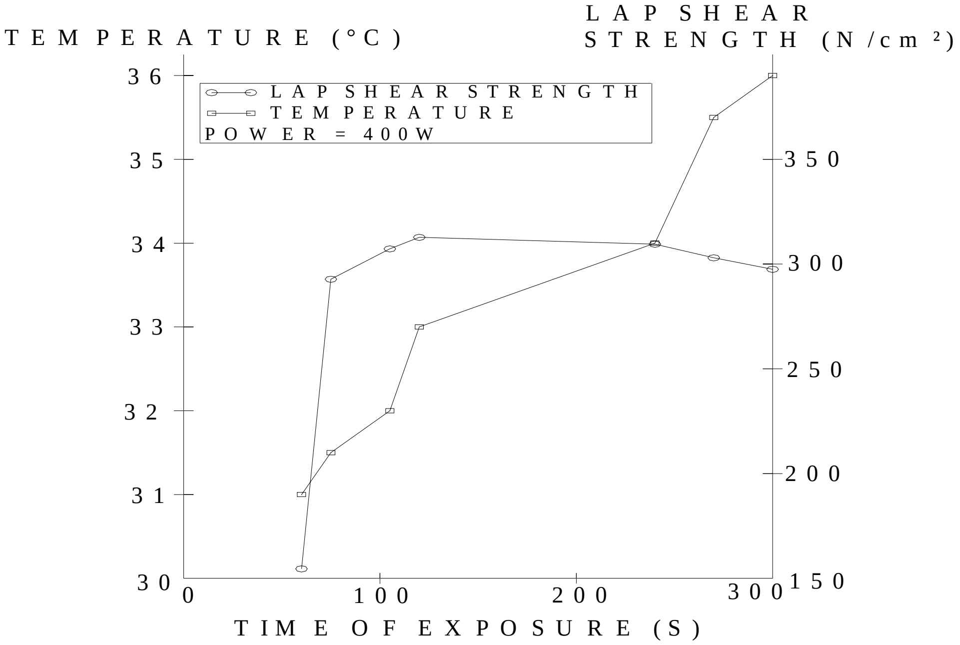


Figure 11: Lap shear bond strength and temperature against time of Exposure to microwaves of 400W in the samples of PS/GF (33%)

Table 1: Volume and Average Temperature of Different Sections of Test Pieces

Sections	Volume (mm^3)	Average temperature (^oC)
Rest on left	1600	27.5
E_{L4} and E_{L3}	300	28.25
E_{L3} and E_{L2}	300	29.75
E_{L2} and E_{L1}	300	31.5
E_{L1} and E	600	32.25
E and E_{R1}	600	32.25
E_{R1} and E_{R2}	300	31.5
E_{R2} and E_{R3}	300	29.75
E_{R3} and E_{R4}	300	28.25
Rest on right	1600	27.5

Table 2: Mass and Energy of Different Sections of Test Pieces

Sections	Mass (g)	Energy absorbed (J)
Rest on left	2.53	17.845
E_{L4} and E_{L3}	0.47	3.700
E_{L3} and E_{L2}	0.47	4.466
E_{L2} and E_{L1}	0.47	5.405
E_{L1} and E	0.95	11.583
E and E_{R1}	0.95	11.583
E_{R1} and E_{R2}	0.47	5.405
E_{R2} and E_{R3}	0.47	4.466
E_{R3} and E_{R4}	0.47	3.700
Rest on right	2.53	17.845