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Microwave welding of polymeric-microfluidic devices

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Abstract
This paper describes a novel technique for bonding polymeric-microfluidic devices using microwave energy and a conductive polymer (polyaniline). The bonding is achieved by patterning the polyaniline features at the polymer joint interface by filling of milled microchannels. The absorbed electromagnetic energy is then converted into heat, facilitating the localized microwave bonding of two polymethylmethacrylate (PMMA) substrates. A coaxial open-ended probe was used to study the dielectric properties at 2.45 GHz of the PMMA and polyaniline at a range of temperatures up to 120 °C. The measurements confirm a difference in the dielectric loss factor of the PMMA substrate and the polyaniline, which means that differential heating using microwaves is possible. Microfluidic channels of 200 μm and 400 μm widths were sealed using a microwave power of 300 W for 15 s. The results of the interface evaluations and leak test show that strong bonding is formed at the polymer interface, and there is no fluid leak up to a pressure of 1.18 MPa. Temperature field of microwave heating was found by using direct measurement techniques. A numerical simulation was also conducted by using the finite-element method, which confirmed and validated the experimental results. These results also indicate that no global deformation of the PMMA substrate occurred during the bonding process.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Microfluidic devices are finding numerous applications in areas such as analytical chemistry [1], biological applications [2], pharmaceutical development [3] and chemical synthesis systems [4]. Microfluidic devices have many advantages over conventional macro-sized systems such as their compact size, disposability, increased functionality, potential for system automation, reduced waste streams, increased precision and accuracy, and for providing fast analytical tools. The world market for these systems and devices is expected to exceed $1 billion [5], and consequently the commercializing of these devices is gaining momentum.

A wide range of microfluidic device components such as valves and pumps [6, 7] have been reported. Most of the microfluidic devices have been fabricated in silicon, quartz or glass [8–10]. However, these materials have some drawbacks such as their cost, fabrication limitations, and lack of optical clarity; therefore they are not the first choice for many microfluidic applications, especially in biology and medicine [11]. Conversely, polymers as substrate materials offer the benefit of low cost, volume fabrication [12] with the added benefits of optical clarity and a wide variety of materials to choose from. For these reasons polymers continue to attract growing interest in the high volume production of disposable microfluidic chips. Different polymers such as polydimethylsiloxane (PDMS) [13], polymethylmethacrylate (PMMA) [14] and polycarbonate (PC) [15] have been widely discussed for microfluidic fabrication.

The bonding and sealing of polymer-based microfluidic devices, without changing or destroying the integrity of patterned microstructures, is a very challenging issue. A number of bonding techniques have been tried such as adhesive bonding [16], thermal bonding [15], solvent bonding [17] and resin-gas injection bonding [18]. However, most of these techniques have some limitations: they may either cause localized geometric deformation of the substrates or leave an interfacial layer with a significant thickness, and chemical variation. It is, therefore, necessary to develop an alternative and practical technique for sealing polymeric-microfluidic devices hermetically, which can scale from the laboratory to the manufacturing environment.

Microwave technology is an excellent method for a sealing operation because of its preferential heating capability and non-contact delivery of energy. This technology relies on the interaction of microwave energy with a microwave absorbing material such as a conductive polymer. Metaxas and Meredith [19] derived an expression, equation (1), for the rate of heating $\mathrm{d}T/\mathrm{d}t$ when an electric field is applied to a material. It is directly proportional to the loss factor, ε'', the frequency, f, and the square of the electric field, E. The rate is also inversely proportional to the density, ρ, and the specific heat, C_p. Hence, the amount of microwave energy absorbed by material is a function of the applied electric field, loss factor and frequency; while ε_0 is the permittivity of free space (constant).

$$\rho C_p \frac{\mathrm{d}T}{\mathrm{d}t} = 2\pi \varepsilon_0 \varepsilon'' f E^2. \quad (1)$$

Most polymers are transparent to microwave energy, and as a result cannot raise their temperature when exposed to radiation of this wavelength. Therefore, the inclusion of microwave absorbers such as conductive polymers offers an opportunity to develop new sealing technologies by placing them at the joint interface, where they selectively absorb the microwave energy. Subsequent heat generation and diffusion makes it possible to locally heat the interface, and bulk polymer flows through the joint to form a weld as the polymer cools under pressure. Conductive polymers such as polyaniline (PAni), polypyrrole and polythiophene have been used as a new class of microwave absorbing susceptor [20]. Polyaniline has been considered as one of the most important materials due to its environmental stability. However, until recently conductive polymers such as polyaniline have been very difficult to produce in liquid form, until dispersion techniques were developed by ORMECON™ to produce a solvent-based nanoparticle suspension. The morphology of the dispersible polyaniline powder is now fully understood and explained elsewhere [21].

Recently there have been reports of polymer microfluidic sealing using microwaves and thin metal layers [22]. However, there has not been work on using selective and localized microwave heating to seal microfluidic devices using solvent-based polyaniline. The solvent-form is easier to use than the powder-form because its delivery does not promote a potentially uncontrollable spreading of the powder. The solvent-form can easily be patterned and confined to specific areas. A solvent-form also lends itself to printing processes, which are more commercially convenient compared to metal implantation or thin film techniques. Microwave bonding using polyaniline has been used successfully demonstrated for thermoplastic polymer such as high-density polyethylene (HDPE) [23]. Yarlagadda *et al* [24] reported the use of focused microwave energy in welding engineering thermoplastics, and finally Wise and Froment [25] provided an extensive review of microwave welding of thermoplastics and supplied useful information on microwave susceptible implant materials such as metals, carbon black and polyaniline.

In this paper, we report a new and promising technique for achieving precise and well-controlled bonding of PMMA microfluidic polymer substrates by using microwave radiation and a solvent-form polyaniline without causing any deformation to the patterned microstructures.

2. Experimental details

2.1. Materials

Liquid form conductive polyaniline under the trade name ORMECON™ L5006 was used for this study. It contains conductive solid nano-particles and organic solvent (xylene and toluene). The polymer substrates used here were polymethylmethacrylate (PMMA), which has dimensions of 100 mm length, 25 mm width and 2 mm thickness. PMMA is an amorphous polymer with a glass transition temperature of around 105 °C. Both polyaniline and PMMA were used as received from the supplier.

2.2. Dielectric properties measurement technique

The dielectric properties of polyaniline and PMMA were measured by using a 3.6 mm diameter open-ended coaxial cable probe (semi-rigid coaxial cable), which was connected to an automatic network analyser hp 8410 with hp sweep signal generator hp 8620. The probe was placed in contact with the sample and the dielectric permittivity was determined from the S_{11} parameters at the interface between the probe and sample. Prior to any measurements, systematic errors were eliminated by calibration with a short circuit, an open circuit, and de-ionized water at room temperature [26]. The temperature of the sample was raised from ambient to 120 °C by placing the sample in intimate contact with a large, thermally controlled mass, with a fibre-optic temperature probe placed near the sample. The microwave frequency was fixed at 2.45 GHz. Each measurement was repeated three times and the average was taken. The accuracy of this technique is ±2% for the dielectric constant and ±1% for loss factor.

2.3. Single-mode microwave system description

A multimode applicator has the disadvantage of low-energy coupling compared to a single-mode cavity. The latter is more efficiently coupled to the material, and especially to low loss materials. Furthermore, the electric field patterns inside the single-mode cavity are predictable and controllable. For these reasons a single-mode microwave cavity was used with a 1000 W, 2.45 GHz microwave generator as shown in figure 1. The microwave generator was connected to a circulator, via an auto tuner with a computer-controlled system and a three-stub manual tuner. The applicator (rectangular wave guide) with internal dimensions of 86 mm length, 43 mm width and 2 mm wall thickness was connected to the stub tuner on one side, with a solid metal plate on the other; this was done in order to

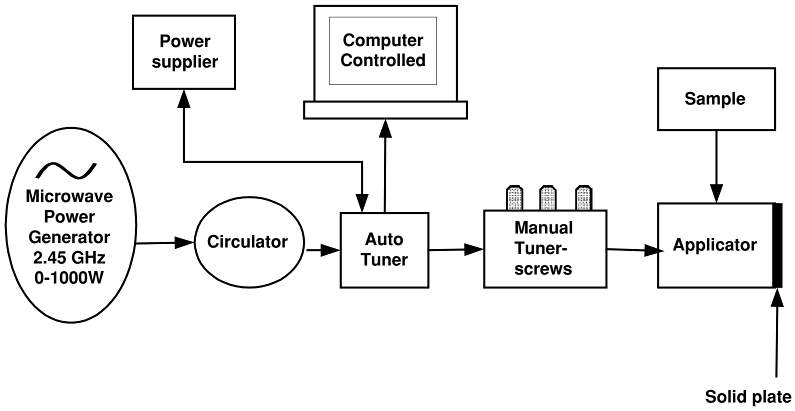


Figure 1. Schematic drawing of a single-mode microwave system.

generate a standing wave. Focusing of the microwave energy onto the polyaniline-patterned strips was complicated by the tiny volume of absorbing material. To overcome this problem it was essential to use a pre-tuned three-stub tuner with an impedance auto-tuner to achieve a perfect match.

2.4. Sample fabrication

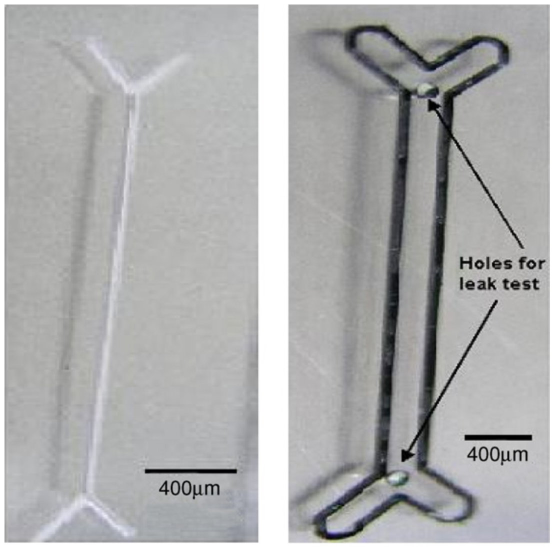
Substrates of 100 mm length and 25 mm width (PMMA) with different sized microfluidic and polyaniline channels were fabricated using a CNC-controlled micro-milling machine (ISEL model No. CPM 3020) which is a stepper motor driven with a 10 μm step size. For temperature field measurements, a single channel of 2 mm width, 300 μm depth and 60 mm length was milled into 4 mm thick PMMA substrate. The dimensions of the microfluidic channels were 200 μm or 400 μm wide, 200–300 μm deep and 24 mm long, in 2 mm thick PMMA. Since polyaniline was in liquid form, it was necessary to make channels to contain it around the fluidic structures. Therefore polyaniline channels with dimensions of 400 μm width, 200 μm depth and 25 mm length were fabricated in the substrate. Two via holes were also drilled in each layer to facilitate alignment. After micro-machining, the debris in the channels was removed by cleaning with isopropyl alcohol (IPA). The separation between the microfluidic channel and the polyaniline channel was 500 μm.

2.5. Techniques of patterning conductive polymer at the interface

For polyaniline patterning, the previously fabricated channels were filled with polyaniline using a syringe, and any excess on the surface of the substrate was removed by dragging a microscope slide over the surface. Excess polyaniline on the surface of the substrate was cleaned using IPA. Figure 2 shows a microfluidic channel to be sealed (*a*) on one PMMA substrate and polyaniline patterned locally on another PMMA substrate (*b*), with holes that were used for fluidic connections during leak and pressure testing.

2.6. Microwave bonding experimental set-up

Two PMMA substrates, one containing a microfluidic channel and the other a polyaniline-filled channel, were placed on top of each other with manual alignment using two holes in each



(*a*) (*b*)

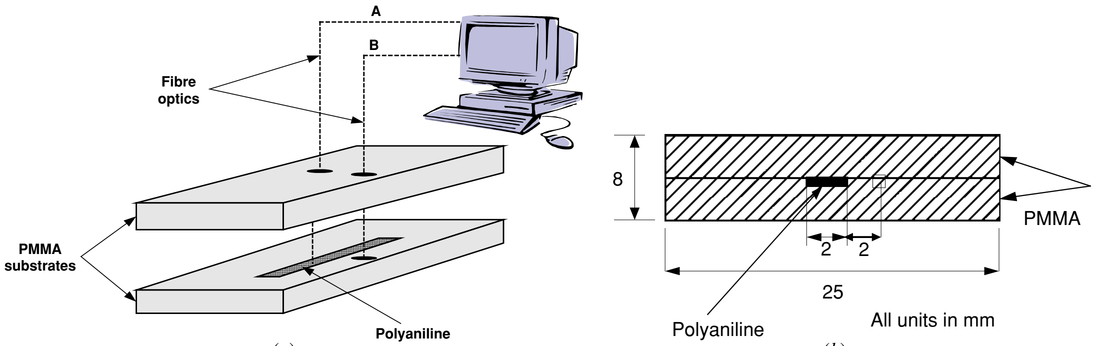
Figure 2. (*a*) Microfluidic and (*b*) polyaniline channels in a PMMA substrate.

substrate in order to make sure that the microfluidic channel was placed between the polyaniline patterns.

A Teflon sample holder was used to compress the sample during microwave exposure. The required pressure was applied on the substrate by using a torque meter and the torque value used for this study was 300 N mm. The torque value was converted to a force value by using equation (2) [27], which shows the relation between torque and force, then converted to a pressure value by dividing by the total area.

$$F = \frac{T}{r\mu + \frac{d}{2}\tan(\alpha + \rho)}, \quad (2)$$

where F is the force, T is the applied torque, r is the average radius of the screw head, μ is the friction constant (0.25), d is the diameter of the screw, α is the pitch angle of the screw, and ρ is $\tan^{-1}\mu$. Therefore, according to above equation, the pressure value for the torque used for this study was 0.12 MPa. However, due to the viscoelastic behaviour of Teflon sheets the applied pressure might not be constant. This pressure was applied over the sample before placing it inside the microwave cavity and no additional



(*a*) (*b*)

Figure 3. Schematic drawing of the temperature field measurements experiment showing (*a*) locations of the temperature probes with respect to the polyaniline channel and (*b*) cross-section of the sample.

pressure was applied during welding in order not to damage the microstructures. Since the position of the sample in the wave-guide determines the amount of electromagnetic energy absorbed during welding, the sample with Teflon holder is placed at the centre of the cavity and parallel to the incoming electric field; this will ensure maximum energy absorption, which maintains a higher temperature. A microwave power of 300 W was applied to the sample for a period of 15 s.

2.7. Sealing evaluation techniques

2.7.1. Interface evaluation. In order to test the quality of sealing, several techniques were employed in order to evaluate the interface properties. For SEM analysis, sealed microfluidic channels were prepared by cutting cross-sections using a low speed, about 0.2 m s^{-1}, diamond saw. Cooling liquid (UC-50 cutting oil) was used during cutting to avoid heat build-up between the sample and the cutting saw. After cutting, the samples were cleaned by using an ultrasonic bath with IPA and finally gold coated. The cross-section of the substrate was examined using a scanning electron microscope (JEOL JSM-840) and optical microscope.

Leak and pressure tests of the sealed channels were performed by filling the bonded microfluidic devices with dye-coloured water. The fluid was pumped through the microfluidic channel by a syringe pump with the help of a computer-controlled system. At the outlet of the channel a pressure sensor was connected at the termination, which recorded the pressure.

2.8. Temperature field measurements for microwave heating

During microwave heating a fibre-optic temperature measurement system (FISO technologies) with a temperature range of −40 °C to +350 °C was used to record the temperature rise during microwave welding of two PMMA substrates. This is a direct temperature measurement technique; the advantage of using this probe is that it is not disturbed by the electric field. The polyaniline channel was made 2 mm wide so that a 1.45 mm diameter fibre-optic temperature probe could be placed within the polyaniline. Two two-millimetre diameter holes were drilled through the microwave cavity, and also into the upper substrate, in order to insert the fibre optic. Two fibre-optic probes were used: one inserted into the channel containing polyaniline, while the other probe was placed 2 mm away from the channel; the details of experimental arrangement and probe locations are shown in figure 3. This will give us the temperature field distribution across the substrate. A microwave power of 300 W was used. The accuracy of the probe is specified at ±1 °C with a resolution of 0.1 °C.

3. Results and discussion

3.1. Dielectric properties

The dielectric properties (complex relative permittivity) of a material are generally defined as a measure of the ability of a material to absorb and to store electrical energy. The loss factor ε_r'' indicates the ability of the material to absorb while the dielectric constant ε_r' is the ability to store electrical energy. Another important factor in microwave heating is the loss tangent, $\tan\delta$, which indicates the ability of a material to convert stored energy into heat. Therefore, for optimum absorption, a high loss factor and loss tangent is required. The expression of the loss tangent is shown in equation (3) [19].

$$\tan\delta = \frac{\varepsilon_r''}{\varepsilon_r'}, \qquad (3)$$

where ε_r'' is the relative loss factor and ε_r' is the relative dielectric constant.

Figure 4(*a*) shows the loss factor versus temperature for polyaniline and PMMA. It can be seen that the loss factor of polyaniline increases with temperature. This means that the material absorbs more microwave energy as the temperature increases to around 115 °C, where it then starts to decrease. The reason for this decrease in loss factor is a decrease in the conductivity of polyaniline at higher temperatures [28]. The loss factor of PMMA remains almost constantly low for all temperatures; this means that the PMMA is essentially transparent to microwave energy even at elevated temperatures. A similar trend is observed for the loss tangent as shown in figure 4(*b*). Conveniently, there is a significant difference in both the loss factor and loss tangent between polyaniline and PMMA. For instance at 100 °C, the value of loss factors for polyaniline and PMMA are 5.4 and 0.056, respectively, which is a ratio of approximately 1:100.

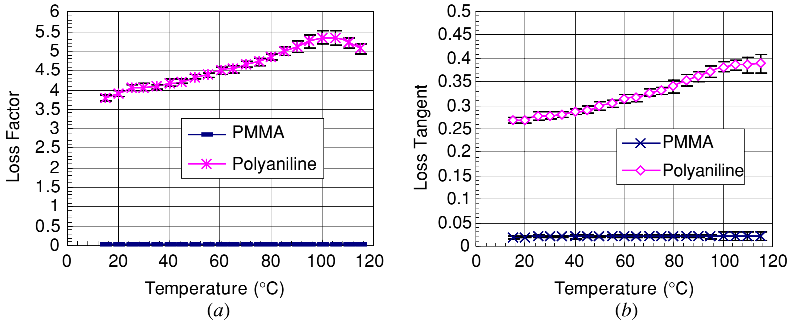


Figure 4. (*a*) Loss factor versus temperature for polyaniline and PMMA and (*b*) loss tangent versus temperature for polyaniline and PMMA.

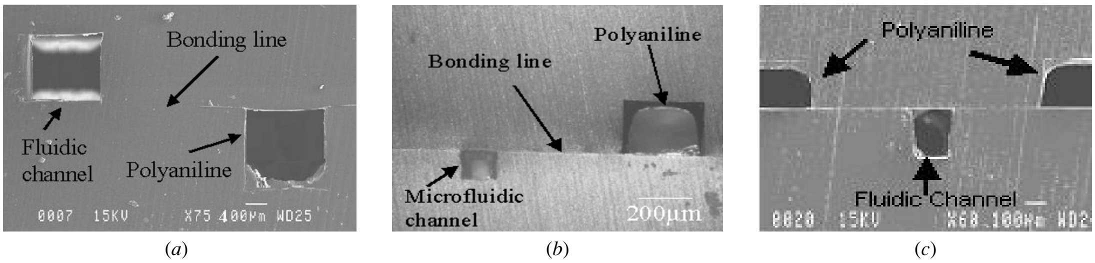


Figure 5. SEM images showing cross-section views of the sealed channels: (*a*) fluidic channel of 400 μm × 400 μm (width × depth), (*b*) 200 μm × 200 μm and (*c*) 200 μm × 300 μm.

3.2. Interface characterization

SEM images of the cross-section of sealed channels are shown in figure 5 for microfluidic channels with widths of 400 μm and 200 μm. These figures show the sealed microfluidic channel surrounded by a polyaniline channel, illustrating that a good bond is achieved without blocking or destroying the integrity of the microfluidic channel. In order to avoid any detrimental effect of microwave heating on the microfluidic channels, the location of the microfluidic channels away from the polyaniline channel was optimized. It was found that 500 μm is the optimum separation distance between microfluidic channel and polyaniline channel to prevent deformation. For a leak test, the coloured fluids flowed along the microchannel to the outlet and yielding an average failure pressure of 1.18 MPa. The absence of any colour outside the sealed channel proved that there was no leak from the welded structures.

3.3. Temperature field results

The temperature rise and temperature field measurement during heating are important parameters in bonding and welding processes. Figure 8 (experimental) shows the temperature history of the two bonded PMMA substrates with a power input of 300 W at two locations in the substrate, as shown in figure 3. It is clear that the probe in the polyaniline (PAni) channel has experienced a higher and faster heating rate, $\mathrm{d}T/\mathrm{d}t$, which is around 10.9 °C s^{-1}, when exposed to the electromagnetic field. It is noted that in less than 10 s the temperature of the polyaniline exceeds 125 °C, which is well above the glass transition temperature (T_g = 100–105 °C) of PMMA and is sufficient to weld the polymer substrates. It must also be noted that in 7 s the temperature reaches 100 °C. On the other hand the probe, which is located 2 mm away from the polyaniline channel, observes a temperature in excess of 85 °C in less than 10 s. This is 40 °C cooler compared to the other channel probe, and confirms that heating is localized to the polyaniline channel. At around 125 °C, the rate of increase of temperature reduces, consistent with the reduced loss factor at this elevated temperature.

3.4. Heat transfer numerical simulation results

In order to be able to model the heat transfer mechanism during the microwave bonding process and validate the experimental temperature field results, the finite-element method (FEM) was used. Commercial software, MAYA's I-DEAS-9 TMG thermal analysis, was utilized for this purpose. By using a three-dimensional finite-element heat conduction model assuming that heat convection to the surroundings is negligible. Due to the larger thickness of the substrate relative to the thermal source and lower thermal conductivity of the PMMA, the heat dissipation through the top and bottom of the substrate was considered to be negligible and the temperature of the outer part of the substrate was close to the ambient conditions. The following three-dimensional heat transfer equation was used.

$$\rho C_p \frac{\partial T}{\partial t} = (\nabla \cdot q) + Q, \tag{4}$$

where T is the temperature at a point inside the material, ρ and C_p are the density and heat capacity, respectively, ∇ is a differential operator, q is the heat flux and Q is the heat generated. The relationship between the heat flux and the temperature gradient is expressed according to Fourier's law as a linear function; $q = k\nabla T$ where k is the thermal conductivity of materials. A summary of the materials relevant physical parameters used in the simulation is shown in table 1.

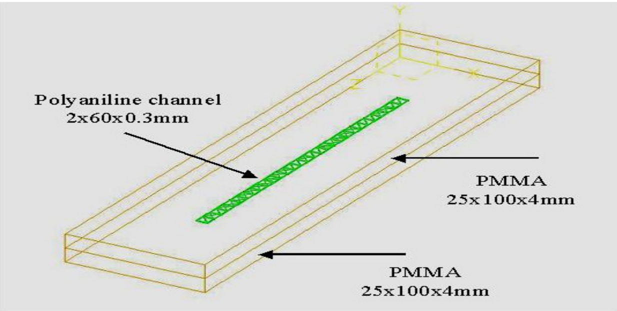


Figure 6. An illustration showing PMMA substrates and the polyaniline channel used for simulation.

In the FEM, two PMMA substrates, one with a polyaniline channel exactly the same as the fabricated structure, were used as illustrated in figure 6, while figure 7 shows the simulation result for 12 s of microwave irradiation at 300 W. Heat is mainly generated inside the channel carrying the polyaniline and then is transferred to the surrounding PMMA substrate by thermal conduction.

In order to obtain a temperature history for a point of interest in the sample, the heat (Q) was placed into the FEM analysis. In this simulation, the heat value of 2.038 $\times$ 10^7 W m^{-3} was input to match the experimental power of 300 W.

Table 1. Physical properties of the materials used for simulation.

Materials	Density (kg m^{-3})	Specific heat (J kg^{-1} K^{-1})	Thermal conductivity (W m^{-1} K^{-1})
PMMA	1300	1200	0.1
Polyaniline	1700	1100	1

Figure 8(a) shows the experimental and simulated temperature histories at the polyaniline channel, while figure 8(b) shows the temperature history of 2 mm away from the polyaniline channel and simulation results. The experimental and numerical simulations match well for the given power input and heating time of 12 s.

Figure 9 shows the FEM-predicted temperature history and distribution across the PMMA sample at various times during the period of 12 s that power was applied; the plot is based on half of the sample due to symmetry. At the centre of the substrate (and the polyaniline channel) the temperature exceeds 120 °C after 12 s, which is high enough to melt PMMA, and 105 °C after 7 s, which is almost the glass transition temperature of the PMMA. This means with this

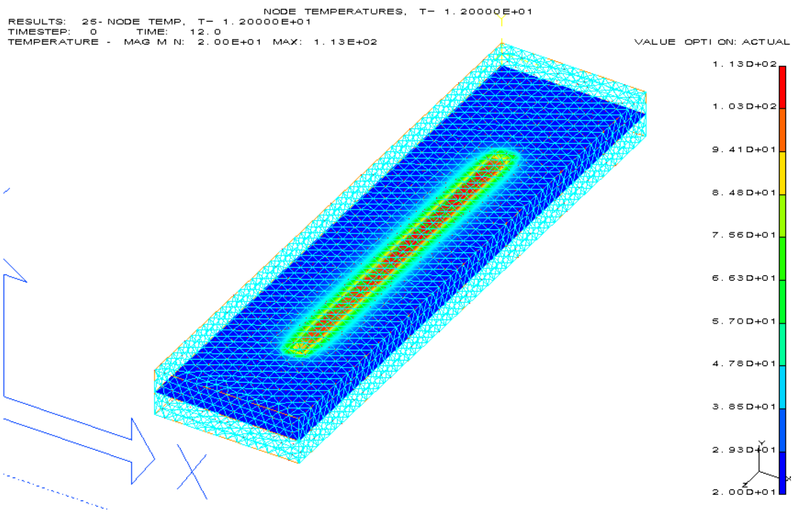
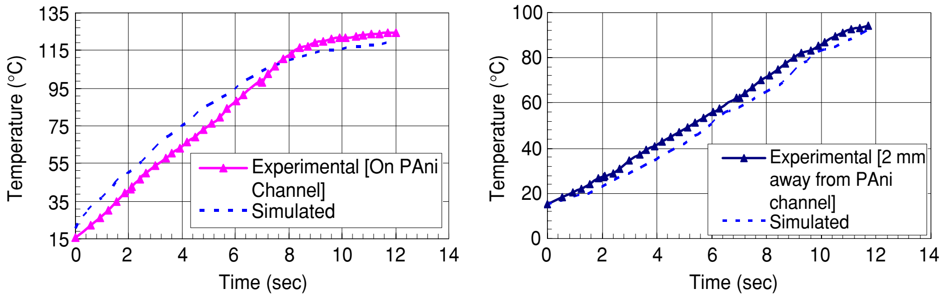


Figure 7. The simulation result for 12 s of microwave heating at 300 W is shown. This figure uses a colour scale that is best viewed in the electronic Web version of this article.



(a) (b)

Figure 8. Correlation of simulation and experimental results for (a) the polyaniline channel and (b) 2 mm away from the polyaniline channel.

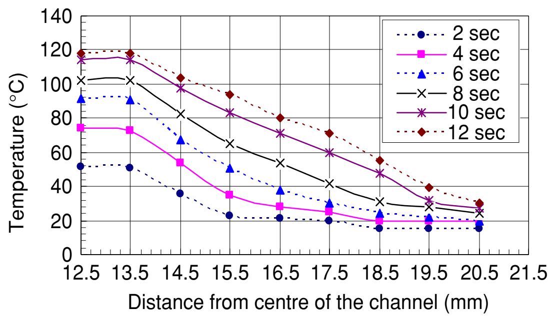


Figure 9. FEM-predicted temperature profile across the PMMA substrate, in the *x*-axis, for a range of irradiation durations.

amount of polyaniline, 7 s of exposure is enough for bonding; therefore, it is notable that more polyaniline is required to achieve a shorter time of sealing. However, the temperature is predicted to be lower in the other regions of the substrate as we move away from the polyaniline channel, and mostly remains below the glass transition temperature due to the localized nature of the heating.

This result provides information about how the temperature varies across the substrate, which will assist us to manipulate the location and volume of polyaniline in future work to achieve the desired thermal profile or heating pattern and optimize the relative location of the microfluidic channel.

4. Conclusions

A new approach for sealing polymer-based microfluidic devices has been demonstrated. Microfluidic devices with channel widths of 400 μm and 200 μm have been successfully sealed using a microwave power 300 W for 15 s. The developed bonding technique has achieved short bonding times with selective and well-controlled heating which causes localized melting of the polymer substrates without altering or changing the integrity of the microfluidic channel.

The measurement of the dielectric properties is useful, in that it helps us to show that the polyaniline absorbs microwave energy at 2.54 GHz and PMMA does not. The above experiments confirm that differential heating is unique to microwave energy and that a strong bonding line is formed at the interface to withstand a pressure up to 1.18 MPa.

Direct measurements of the temperature at the polyaniline channel are considerably higher than the temperature 2 mm away, further supporting the fact that heating is generated by selective absorption of the microwave energy by the polyaniline. The temperature at the polyaniline exceeds 120 °C in less than 10 s, which is sufficient to weld the polymer substrate. FEM predictions were in good agreement with the measured temperatures for both the polyaniline channel and away from the channel. The simulation results strongly support the conclusion that microwave heating can be used for welding and bonding polymeric microfluidic devices without any global deformation of the substrate due to the localized and selective heating.

This technique provides an alternative for sealing polymer-based microfluidic devices, without any structural deformation and demonstrates the capability of microwave technology for welding and bonding applications in polymer microfluidic systems, bio-MEMS and other micro-system packaging applications.

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