Single mode microwave sealing of polymer-based microfluidic devices using conductive polymer

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ABSTRACT
Polymer based microfluidic devices have an important potential use in BioMEMs applications due to the low cost and biocompatibility. However, sealing the devices hermetically without blocking the channels, altering their dimensions or changing the surface properties is a challenging issue in their fabrication. In this paper a microwave-based sealing technique using a polymethylmethacrylate (PMMA) substrate and conductive polymer (polyaniline) is presented. The developed novel bonding technique has achieved precise, well-controlled and selective heating, which causes localized melting of the polymer substrates. At the joint interface, patterned polyaniline features absorb electromagnetic radiation and convert it into heat, which facilitates the microwave bonding of two PMMA substrates. This new approach can easily seal microfluidic devices with micron-sized channels without blocking or destroying the integrity of the channel. Microfluidic channels of 400 μm and 200 μm wide were sealed using a microwave power of 300 Watts, in less than 20 seconds. The microfluidic channel fabrication techniques, polyaniline patterning method at the interface and bonding evaluation such as sample cross section and leak test are discussed. The dielectric properties of polyaniline and PMMA at 2.45 GHz frequency are also evaluated by using the open probe technique, which shows PMMA is essentially transparent to microwave energy.

Keywords: Microfluidic, sealing, microwave, microfabrication and polyaniline

1. INTRODUCTION
The field of microfluidics is undergoing rapid growth in terms of new device developments. Microfluidic devices have enjoyed success in certain niche applications such as biotechnology, pharmaceuticals, public health and life sciences, each of which has its own applications. Hence, the field is experiencing changes in terms of material selection, device development techniques and sealing methods. Generally, microfluidics refers to devices or flow of configurations that have the smallest design feature on the scale of a micron, typically with rectangular channels with cross-sectional dimensions on the order of tens or hundreds of microns. A wide range of microfluidic devices such as valves, pumps and flow sensors have been reviewed and presented [1].

Most of the microfluidic devices reported in the literature have been fabricated in silicon, quartz, and especially glass [2,3] has been the dominant substrate material for fabrication. However, these materials are often too expensive and lack some properties such as optical clarity and poor biocompatibility. On the other hand, polymers as substrate materials offer a possible solution to these fabrication challenges with the additional benefit of mass fabrication of microfluidic devices. Their wide range of material properties, their low costs and the development of suitable polymer microfabrication techniques in the past years have attracted enormous interest, especially as this opens up the road to a high volume production of disposable microfluidic devices.

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In the literature various polymers such as polydimethylsiloxane (PDMS), polymethy methacrylate (PMMA), polycarbonate (PC), polystyrene (PS) and polyethylene (PE) have been reported for microfluidic fabrications [4-6].

One of the important and challenging issues in polymer microfluidic devices is bonding and sealing together different components without blocking the channel. Currently a number of bonding techniques have been reported in the literature such as adhesive bonding [6], thermal bonding [7,8], solvent bonding [9], resin-gas injection technique [10], laser welding [11] and microwave bonding using a thin metal gold as a microwave absorber [12]. However, most existing bonding techniques in microfluidic devices have certain limitations. For example the presence of an adhesive can change the channel size and channel properties in adhesive bonding. Thermal bonding demands close tolerances to ensure a hermetic seal and necessitate high moulding cost. Therefore it is necessary to develop a better and more practical technique for sealing microfluidic devices.

In this paper, we report a new and promising technique for achieving precise and well-controlled sealing of polymethylmethacrylate (PMMA) microfluidic polymer substrates by using microwave radiation and a conductive polymer (polyaniline) without causing any global deformation to the substrates.

2. MICROWAVE BONDING TECHNOLOGY

Microwave sealing relies on the nature of the interaction of high frequency energies with a microwave absorber such as conductive polymer. Most polymers are transparent to microwaves, which means they will not absorb microwave radiation and as a result it cannot raise their temperature. Therefore, microwave absorbers such as conductive polymers offer an opportunity in developing new sealing technologies by placing them at the joint interface, where they absorb selectively the microwave energy. Subsequent heat generation makes it possible to locally heat the interface, and bulk polymer begins to flow across the joint to form a weld as the polymer cools under pressure. This makes microwave bonding an attractive alternative in view of its advantages over conventional sealing processes. One advantage of microwave bonding is the ability to flood expose large areas heat generate extremely localized heating.

Conductive polymers such as polyaniline, polypyrrole and polyalkylthiophenes have been used as a new class of microwave absorbing susceptor. Among the conductive polymers, polyaniline has been considered as one of the most important materials because the electromagnetic parameters can be adjusted by changing both the oxidation and the protonation state [13].

3. EXPERIMENTAL

3.1 Microwave system description

A single mode cavity was connected to a 2.45 GHz microwave generator (see Figure 1) with 1000-Watts maximum power. The microwave generator is 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 dimensions of 86 mm length, 43 mm width, and 2 mm thickness was used. Finally, a solid metal plate was attached at the end of the waveguide to generate a standing wave. It is essential that a pre-tune with manual tuning is applied so that the auto tuner is able to quickly achieve a perfect tune.

A multimode applicator has the disadvantage of low energy coupling efficiency compared to a single mode cavity. The latter is more efficiently coupled into the materials, especially low loss material. Furthermore, the electric field patterns inside the single mode cavity are controllable and predictable. Therefore, maximum heating efficiency can be achieved by using single mode cavity.
3.2 Materials

Liquid form conductive polymer (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 used here is polymethylmethacrylate (PMMA), which has dimensions of 100 mm length, 25 mm wide, and 2 mm thickness. PMMA is an amorphous polymer with a glass transition temperature of around 109°C. Both polyaniline and PMMA were used as received from the supplier. Some properties of the polyaniline are tabulated in Table 1.

Table 1: Polyaniline properties [14].

<table>
<thead>
<tr>
<th>Properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ignition temperature</td>
<td>535°C</td>
</tr>
<tr>
<td>Boiling point</td>
<td>111°C</td>
</tr>
<tr>
<td>Steam pressure at 20°C</td>
<td>29 mbar</td>
</tr>
<tr>
<td>Solid content</td>
<td>16%</td>
</tr>
<tr>
<td>Organic content</td>
<td>84%</td>
</tr>
</tbody>
</table>

3.3 Dielectric properties measurements

The dielectric properties of polyaniline and PMMA were measured by using a 3 mm open-ended probe, which was connected to an automatic network analyzer [15]. The probe was placed in contact with sample and the dielectric permittivity was determined from the $S_{11}$ parameters at the interface between the probe/sample. Prior to any measurements, systematic errors were eliminated by calibration with a short circuit, an open circuit, and distilled water. The temperature of the measurement was started from room temperature to 110°C, and a fibre optic probe, placed on top of the block of aluminum, recorded temperature. The block was located inside a heater. The frequency was fixed at 2.45 GHz during measurements.

3.4 Substrate fabrication and polyaniline patterning techniques

The substrates (PMMA) with different micro-sized features for microfluidic channel and polyaniline channel were fabricated by using a CNC controlled micro-milling machine. The dimension of the microfluidic channel was 200 µm or 400 µm wide, 200 µm deep and 24 mm long.
Since polyaniline is in a liquid form it is necessary to make channels to pattern it, therefore channels with dimensions of 400 µm wide, 200 µm deep and 25 mm long were fabricated on PMMA substrate for polyaniline patterning. After micro machining, the debris in the channels was removed by cleaning the channel with isopropyl alcohol (IPA). The separation between the microfluidic channel and polyaniline channel was 500 µm. For polyaniline patterning, the channels were filled with polyaniline and any excess on the surface of the substrate was removed by dragging a microscope slide on the surface and all polyaniline remains were cleaned by using IPA. Figure 2 shows microfluidic device to be sealed (A) and polyaniline patterned locally on the PMMA substrate (B) showing holes for leak and pressure test.

3.5 Microwave bonding experiment set up

Two PMMA substrates, one containing a microfluidic channel and other one patterned polyaniline were then placed on top of each other with proper alignment (two holes on each substrate) in order to make sure that the microfluidic channel was placed between the polyaniline patterns.

A Teflon sample holder was used to hold the sample in position. The required pressure was applied on the substrate by using a torque meter. The torque meter was calibrated by using a pressure sensor device, which was placed between the substrates in order to convert the applied torque to pressure. The constant pressure of 0.3 PMa was applied on the sample before placing it inside the cavity and no additional pressure was applied during welding in order not to damage the microstructure. Since the position of the sample in the wave-guide will affect absorption of electromagnetic energy during welding, the sample with Teflon holder is placed at the center of the cavity and in parallel with the incoming electric field; this will maintain a higher heat generation. The microwave power of 300 Watts was applied to the sample with a period of 15 seconds.

3.6 Characterization and sealing evaluation

In order to test the quality of sealing, several experiments such as interface evaluation by using SEM and leak test have been conducted. For interface evaluation, the sealed microfluidic channels were taken and samples for interface evaluation were prepared by cutting cross-sections using a low speed diamond saw.

Cooling liquid was used during cutting to avoid heat build up between the sample and saw. After cutting the samples were collected, cleaned and prepared for evaluation. The cross section of the substrate was examined using a Scanning Electron Microscope (JEOL JSM - 840).

Leak and pressure tests of the sealed channels were performed in order to characterize the seal of the microfluidic device; the bonded microfluidic device was filled with color dyes in order to detect any leak. Prior to sealing, two holes were drilled by using a micro-milling machine on the cover substrate (substrate with polyaniline), these holes were positioned at the end of microfluidic channel in order to provide access of the color dyes for leak test.
4. RESULTS AND DISCUSSION

4.1 Dielectric properties

The dielectric properties of materials are generally defined as a measure of the ability of a material to absorb and to store electrical energy. The loss factor indicates the ability of the material to absorb the electrical energy. Therefore for optimum absorption a high loss factor or a high conductivity is required.

Figure 3 shows the loss factor versus temperature for polyaniline and PMMA. This graph shows that the loss factor of polyaniline increases with temperature. This trend of increase means the material is absorbing more microwave energy at a higher temperature. However, at around 115°C the loss factor decreases. The reason is a phase change of the material at that temperature due to the evaporation of the organic solvent. On the other hand the loss factor of PMMA remains constantly low for all temperatures, this means that the PMMA is not absorbing microwave energy even at higher temperatures. All energy goes to the polyaniline, which generates heat for welding. Similarly, Figure 4 shows the conductivity of the polyaniline and PMMA at different temperature and it is observed that the polyaniline exhibits high conductivity, which increases with temperature. However, the conductivity of PMMA remains constant at different temperatures. Both of these results indicate that polyaniline is absorbing microwave energy, while PMMA is essentially transparent to microwave energy.

![Figure 3: Loss factor vs temperature for polyaniline and PMMA.](image_url)
4.2 Interface and cross section evaluation results

The SEM images of the cross-section of the sealed channels are shown in Figures 5 and 6, for different microfluidic channel widths (400 µm and 200 µm respectively). Figure 5 shows the sealed microfluidic channel and surrounded polyaniline channels (A) with dimensions of 400 µm x 200 µm for both microfluidic channel and polyaniline channel and also an enlarged view of the sealed microfluidic channel (B). Similarly in Figure 6 the microfluidic channel and polyaniline channels are shown with smaller dimensions of 200 µm x 200 µm for microfluidic channel and 400 µm x 200 µm for polyaniline channels (A) and an enlarged microfluidic channel view (B). The results show that good sealing or bonding was achieved without blocking or destroying the integrity of the microfluidic channels.

![Figure 5: SEM images of the cross-section view of (A) sealed microfluidic channel surrounded by polyaniline channels, (B) enlarged view of the microfluidic channel (400 µm x 200 µm).](image)
4.3 Leak and pressure test results

Color dyes pass through the previously fabricated holes on the polyaniline substrate directly to the microchannel, reaching the other hole at the end of the microchannel and filling completely the microfluidic channel, while the other end of the channel was connected to the pressure sensor and pressure was recorded. The sealed channel (400 µm wide and 200 µm deep) filled with color dye is shown in Figure 7. The microfluidic was filled completely and pressure was applied, the absence of any color outside the sealed channel indicates no leak and results showed no leakage up to 172 psi, and then the only leak was detected at the inlet connections.

5. CONCLUSIONS

A new method of microfluidic sealing with conductive polymers has been discussed. The microfluidic devices with a channel width of 400 µm and 200 µm have been successfully sealed using microwave parameters of 300 Watts and 15 seconds expose. This localized sealing technique has achieved precise, well-controlled and selective heating.

The results of the dielectric properties shows that the polyaniline absorbs microwave energy, while PMMA is essentially transparent to microwave energy.
Two investigations of sealing at the interface using SEM and leakage tests have been conducted to characterize the quality of the sealing. All these results indicate that strong sealing is formed at the interface. Experimentally, up to 172 psi of pressure without leakage has been achieved.

The developed polymeric microfluidic sealing technique provides an alternative approach to seal microfluidic devices without changing the structure of the micro-featured channels. Furthermore, these results demonstrate the capability of microwave sealing technology for sealing and bonding applications in polymer microfluidic systems and other polymeric bio-MEMS. However, further work is necessary to closely fit the liquid conductive polymer towards use in screen-printing techniques for mass manufacturing processes.

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