



# Direct assembly of cyclic olefin copolymer microfluidic devices helped by dry photoresist



Lamia El Fissi<sup>a,\*</sup>, Denis Vandormael<sup>b</sup>, Laurent A. Francis<sup>a</sup>

<sup>a</sup> ICTEAM Institute, Université catholique de Louvain, Louvain-la-Neuve, Belgium

<sup>b</sup> SIRRIS – Liege Science Park, Seraing, Belgium

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## ABSTRACT

A generic method for rapid, reproducible, and robust selective bonding of microfluidic chips made of Cyclic Olefin Copolymer (COC) has been developed and optimized. In this work we propose an adhesive bonding technique using ORDYL negative dry film photo-resist as glue to perform the packaging of COC micro-patterned structures. The ORDYL resist is qualified in terms of resolution, biocompatibility and fluidic sealing. The adhesive bonding is achieved by laminating a thin layer of ORDYL SY300 (<17 μm) on top of the microfluidic part and then bonded to the other COC part. In this research, an oxygen plasma treatment for adhesion improvement was performed on COC surfaces at various plasma times. The bonding method is described in detail and the bonding quality of the chips was evaluated by a shear strength testing procedure and a leak test by pressurizing a microfluidic channel with an aqueous solution using an external peristaltic pump. Results are reported emphasizing the efficiency of the proposed approach and the developed process features high yields (>70%).

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## 1. Introduction

The impact of microfluidic technologies has dramatically increased during the last few years since microfluidic is considered as a key technology within the field of life science [1]. Polymer based Lab-on-a-chip (LOC) devices or micro-total analysis systems (μTAS) are currently hot research topics in the field of microfluidics and BioMEMS [2]. The main advantages of microfluidic systems are that important biological operations such as pathogen detection and genotyping can be accelerated and amplified by integrating a variety of functions like sample preparation, DNA amplification, cell capture, pumping, mixing, and detection onto a single platform with minimal or no human intervention. The advantages of polymers over glass and silicon as a substrate for microfluidic devices include [3] their biocompatibility, disposability, good chemical resistance, optical properties, low cost, and capacity for high volume production using established manufacturing techniques such as hot embossing and injection molding.

Several polymeric materials including polymethylmethacrylate (PMMA) [4], polycarbonate (PC) [5] polyester [6], fluorinated ethylene propylene [7], and poly(ethylene terephthalate) [8] and cyclic olefin copolymer (COC) [9] have been investigated for the

microfluidics fabrication [10]. COC (Topas) which is a thermoplastic copolymer has been used because of its excellent properties, such as high glass transition temperatures, excellent transparency [11].

Bonding between polymer substrates is an essential fabrication step to seal and form micro-channels or micro-chambers in plastic LOC or μTAS system since the micro-channels on the polymer layers are normally opened after the micro-machining step. Today, several bonding techniques for plastic substrates have been developed such as a thermal lamination [12] the adhesive bonding [13], the thermal bonding [14,15], the laser welding [16,17], and the conventional solvent bonding [18,19].

The solvent bonding of thermoplastics takes advantage of the polymer solubility in selected solvent systems to achieve entanglement of polymer chains across the interface. When a thermoplastic surface is solvated, the polymer chains become mobile and can readily diffuse across the solvated layer, leading to an extensive intertwining of chains between the surfaces and resulting in exceptionally strong bonds. The solvent bonding is carried out at room temperature or decreased temperature levels which comply with the reagent pre-storage. Furthermore, the chips are usually stored at high temperatures for several hours to accelerate the solvent evaporation after the bonding. The temperature stability of the bonds depends significantly on the complete evaporation of the solvents, since the presence of solvents causes stress cracks and cloudiness.

\* Corresponding author. Tel.: +32 010472174.

E-mail address: [lamia.elfissi@uclouvain.be](mailto:lamia.elfissi@uclouvain.be) (L. El Fissi).

The laser welding is an interesting approach which can be used for bonding of contours, planes and at local spots [16,17]. This is particularly interesting for applications with pre-stored reagents as the heat affected zone is very narrow in laser welding. But since the welding happens at the abutting surfaces, one of the two layers has to allow the laser light transmittance while the other one absorbs the light and melts. Transparent materials can be equipped with absorbing additives like carbon particles or semi-translucent absorbers.

Such absorbers increase significantly the absorbance within the NIR range, while the transmission within UV and the visible bandwidth is hardly affected.

Most polymers also show a natural absorbance within the IR-spectrum which makes them weldable without the need for additional absorbers. Compared to the intermediate absorber layer, this approach can cause increased stress cracks. The selective bonding by laser requires a very precise alignment of substrate, lid foil and laser head. When foils are laser bonded, the depth of focus must be very precise.

During thermal bonding [13–15], substrates are heated at temperature near or above the glass transition temperature ( $T_g$ ), and a pressure is applied to increase mating contact forces. The combined temperature and pressure can generate sufficient a flow of polymer at the interface to achieve an intimate contact, with inter diffusion of polymer chains between the surfaces leading to a strong bond. One major challenge of the thermal bonding is the channel deformation caused by un-optimized temperature and pressure. Thus, properly controlling temperature, pressure, and time is critical to achieve high bond strength while limiting deformation of the embedded microchannels due to the bulk polymer flow.

The adhesive bonding uses an additional intermediate layer, organic or inorganic; which joins the two bond partners. Especially if bond materials do not suit for the direct bonding, they still can be linked by adhesives. The adhesive material deforms and flows, so that it can make a sufficiently close contact with the wafer surface to create a bond. In the adhesive wafer bonding the adhesive layer is deposited on one or on both the wafers, e.g. by spin coating, laminating, spraying or other suitable deposition techniques. The wafers are brought into contact and the intermediate adhesive layer is cured, typically by applying heat and pressure. The adhesive bonding may be used to join a large variety of materials combinations including metal–metal, metal–plastic, metal–composite, composite–composite, plastic–plastic, metal–ceramic systems. Adhesives are usually based on chemical effects like polymerization (e.g. acrylics), polycondensation (e.g. silicones) or polyaddition (e.g. epoxies) and can often be cured at room temperature, at elevated temperatures or by using UV light.

For successfully thermal bonding microfluidic chips, a high number of influence parameters have to be taken into account [19]. However, both the chip design [21] and the adhesive layer thickness [22] are major influence parameters for bonding, i.e., chips featuring large ( $>1$  mm) and deep ( $>500$   $\mu\text{m}$ ) channels, only, will require less bonding process development due to the reduced risk of clogging than chips comprising small (500  $\mu\text{m}$ ) and shallow (200  $\mu\text{m}$ ) channels as well as isolated features. In addition, the bonding layer transferred has to form a thin and smooth layer on the microfluidic chip for a strong adhesive bonding.

Established materials for adhesive bonding in micro-machining are dry film photoresists [19–21]. Examples for dry film photoresists used in plastic LOC fabrication are ORDYL [22–25], e-NIT215 [26], Poly-ether-ether-ketone (PEEK) [27], TMMF [28] or self-made SU-8 foils [29,30]. ORDYL provides a set of interesting properties, such as its chemical stability, low cost and availability in a wide range of thicknesses. Particularly, ORDYL SY300 shows compatibility with biological fluids [32]. Also it has a strong adhesion to different materials such as glass, silicon, epoxy, resin and polymer

and presents an excellent solvent resistance and a good acid/base resistance [33].

In general, the lamination of dry film resist yields layers with low thickness deviation. Also, the dry film photoresist can easily be laminated onto structured wafers with a high topography profile with good planarity, no liquid handling required, good adhesion to almost any substrate, and simple fabrication process.

Recently, dry film photoresists have moved away from their original purpose of providing sacrificial layers for the fabrication of printed circuit boards and were used to manufacture electroplating molds [34], replication tools [35], for wafer bonding [36] and sensor packaging [47], as an etch mask for silicon-DRIE [38] and as a permanent material for microfluidic applications [39].

In this work, we present the development of a new use for ORDYL dry film photoresist. Our approach is based on a new bonding technique to fabricate microfluidic structures containing embedded channels with precisely defined geometries in COC substrate using ORDYL dry film photoresist as glue. The ORDYL photoresist will be transferred only on the patterned areas unlike common transfer approaches. The fabrication process is based on the ORDYL SY300 series. We optimize the bonding temperature and time of the soft-, and post exposure bake to ensure both; the mechanical stability of the resist as well as certain adhesiveness which is required for the low-temperature bond.

The COC embedded channels (microfluidic part) is fabricated using an injection molding technique. Different channel sizes will be tested: channels width (30, 50, 100 and 250  $\mu\text{m}$ ), and height (100, 250 and 500  $\mu\text{m}$ ).

The key features of the technique include: (a) COC embedded channels were at first subjected to oxygen plasma treatment, (b) manually lamination of ORDYL dry film through a COC embedded channel. (c) The ORDYL is imprinted on the COC channels and the patterns are obtained when the protection polyester film is removed. Unlike the liquid photo resist coating, in this case an indirect coating method is used that leaves all channel walls in their original state. It is achieved by the ORDYL's lamination selectively on the contact surfaces of the microfluidic part and not inside the channels. Following a detailed description of the bonding procedure steps, the bonding quality of the chips was evaluated by the shear strength testing procedure and a leak test by pressurizing a microfluidic channel with an aqueous solution using a peristaltic pump and a pressure test.

## 2. Experimental

### 2.1. Materials

A negative-type permanent photoresist ORDYL SY300, manufactured by Tokyo Ohka Kogyo Co, is used. The structure of the dry film resist has to have a chemical resist formulation that is very viscous, in order to sandwich it between a polyethylene (PE) sheet and a polyester (PET) basis (Fig. 1). Although the exact composition and functioning is a trade secret, it is known that it contains roughly 25% acrylic esters and 60% acrylic polymers [32]. The acrylic esters contain epoxy groups that give the dry-film resist a high chemical and mechanical resistivity after cross-linking and establish bonds to the substrate surface. The dry film is available in thicknesses ranging from 17  $\mu\text{m}$  to 50  $\mu\text{m}$  and is delivered in rolls of 30 m long with standard widths of 200 mm, 250 mm or 330 mm. The recommended processing parameters of ORDYL SY300 film are listed in Table 1.

Cyclic olefin copolymer (COC), Topas 5013 (Ticona, Florence, KY, USA), with glass transition temperatures ( $T_g$ ) of 130 °C was selected as the substrate material. The polymer pieces were injection molded (BOY XS V injection molding machine) at 285 °C and 750 bar pressure from the COC pellets.

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