Low-cost and facile implementation of microfluidic colour-changing devices using dry film photoresist-based moulds

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ABSTRACT

In this work, different microfluidic colour-changing devices are implemented by using dry film photoresist-based moulds instead of standard photolithography moulds. EtertechHT-115T negative dry film photoresist is employed to realise the rapid fabrication of the moulds for colour-changing layers. The major factors that may affect the fidelity of the dry film moulds during fabrication are summarised and analysed, including the optimum exposure times and the appropriate developing times. Especially, the impacts of different concentrations of sodium carbonate (Na\textsubscript{2}CO\textsubscript{3}) solution on developing rate are investigated for 1–5 layers (50–250 \textmu m thick) of EtertechHT-115T dry film photoresists by experiments. The created dry film moulds show the advantages of low cost, high manufacturing efficiency and requiring no professional training. Each application of the microfluidic colour-changing devices presents high transparency and good colour-changing effect. The microfluidic colour-changing layers based on dry film moulds can be used in different wearable devices of human, and also can be applied for realising surface camouflage and display functions of soft machines/robotics.

1. Introduction

Microfluidics is a rapidly expanding field with applications in a number of different markets including biochemical analysis [1,2], drug delivery [3] and medical diagnose [4,5], due to the small sizes, high throughput and low cost of microfluidic systems [6]. In recent years, some studies on the colour-changing function of microfluidic systems are reported. Morin et al. reported a microfluidic network to provide both camouflage/display and movement of a soft machine [7]. In our previous reports, we proposed a microfluidic liquid colour-changing method, which was applied in different optical devices. By circulating colour liquids in microfluidic channels on the colour-changing layers, these devices can realise vision protection [8], surface camouflage and optical filtering [9]. However, the key step to fabricate these microfluidic devices is to make colour-changing layers with...
different channel structures by using microfluidic moulds, which need complex creating process and require large investments (including clean environment, well trained people, expensive photolithography equipments, etc.), and therefore become the development bottleneck for microfluidic research and application.

Dry film photosist offers many advantages such as low cost, good uniformity of photosist distribution, low exposure energy and near vertical sidewalls [10]. It has been most commonly used in the patterning of printed circuit boards (PCBs) [11] and electroplate moulds in Lithographie, Galvanoformung, and Abformung (LIGA)-type process [12]. Recently, dry film photosist also arises greater interests of the researchers in microfluidic field [13,14]. Smejkal et al. proposed a dry film photosist microfluidic chip used for analysing the isotachophoresis of lactate in human serum [15]. Horak et al. reported a disposable microfluidic immunochip, which employed photosist film to biofunctionalize the channels to enhance electrochemical detection of a cardiac biomarker [16]. Weltin et al. presented a flexible microsensor, which was fabricated on a wafer-level including a layer of dry film photosist for insulation [17]. Qu et al. provided an electrochemical micromachining technology of micro-dimple arrays on cylindrical inner surfaces using GPM200 dry film photosist [18]. Kling et al. developed a microfluidic platform enabling the electrochemical readout of up to eight enzymelinked assays (ELAs), dry film photosist served as the channel material in this system because it allowed long storage of pre-immobilized assays [19]. However, the microstructures in these devices are directly defined and machined by using dry film photosists, which are fine for most cases such as liquid/gas transferring and biochemical tests, but impossible for some optical applications, because optical transparency is not considered in these microfluidic systems.

Here, we develop the application of EtertecHT-115T dry film photosist (50 µm thick) in the moulds implementation for different microfluidic colour-changing devices, including colour-changing glasses, camouflage glasses and colour-changing starfish. The key parameters in the moulds fabrication are analysed and optimised for 1–5 layers (50–250 µm thick) of dry film photosists by experiments. Different sizes of microstructures for each application can be personal designed and rapidly realised by this low-priced way without need of professional training and expensive equipments. Two or multilayer structures of colour-changing devices can also be easily achieved for more complex functionality by using this method.

2. Experimental procedures

2.1. Experimental materials

In this paper, 10:1 mixture of sylgard 184 silicone elastomer and curing agent (Dow Corning Corporation, USA) is employed to fabricate the PDMS colour-changing layers. EtertecHT-115T (Eternal Chemical Industry Limited Company, China), a negative dry film photosist with 50 µm thick, serve as the material of the moulds patterns. In order to improve the flatness and accuracy of the moulds, 10 cm × 10 cm × 2.5 cm mirror stainless steel plates (Xingyang Stainless Steel Company, China) with very smooth surfaces are selected as the substrates of dry film moulds. 365 nm UV lamp (Shunying Photo Electricity Limited Company, China) is used for exposure. Anhydrous sodium carbonate, deionised water and nitrogen (Zhongjia Chemical Company, China) are used for developing, cleaning and drying during the experiments. In order to realise irreversible bonding, air plasma cleaner (Wenchang Chip Technology Limited Company, China) is prepared for surfaces treatment.
2.2. Fabrication of the microfluidic moulds by using EtertecHT-115T dry film photoresist

The fabrication process of the microfluidic moulds by using EtertecHT-115T negative dry film photoresist is shown in Figure 1. The photomask is prepared by printing the designed micropatterns on a transparency film at 2400 dpi. Compared with conventional glass/metal material, the film photomask can not only meet resolution requirement, but also show the advantages of low-cost, fast and convenient for manufacturing, thus makes the dry film etch technology more accessible to an out-of-clean room environment.

Then, transfer the designed microfluidic patterns from the photomask to the dry film photoresist sheet through the following process: Clean the mirror stainless steel plate with deionised water, dry it in nitrogen stream, and used as the substrate of the mould. Cut the dry film photoresist into 10 cm × 10 cm square. Peel off one of the translucent substrates pre-coated over the two surfaces of the photoresist (Figure 1a). Lamine the dry film photoresist (10 cm × 10 cm × 2.5 cm) by using an office laminator at 110°C. Overlap the three sheets (PMMA cover plate, prepared photomask sheet and mirror stainless steel substrate) as shown. Fix around with clips to avoid light leaking. Irradiate under 365 nm UV lamp at a distance of 15 cm. Peel off the other translucent substrate from the dry film photoresist. Immerse in 1% (mass concentration) Na₂CO₃ solution for developing. Clean the developed structure with deionised water and dry it in nitrogen stream.

**Figure 1.** The moulds fabrication for microfluidic colour-changing devices by using EtertecHT-115T dry film photoresist. (a) Peel off one of the translucent substrates from dry film photoresist. (b) Lamine the dry film photoresist (10 cm × 10 cm) on the cleaned mirror stainless steel substrate (10 cm × 10 cm × 2.5 cm) by using an office laminator at 110°C. (c) Overlap the three sheets (PMMA cover plate, prepared photomask sheet and mirror stainless steel substrate) as shown. (d) Fix around with clips to avoid light leaking. (e) Irradiate under 365 nm UV lamp at a distance of 15 cm. (f) Peel off the other translucent substrate from the dry film photoresist. (g) Immerse in 1% (mass concentration) Na₂CO₃ solution for developing. (h) Clean the developed structure with deionised water and dry it in nitrogen stream.

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Finally, clean the developed structure with deionised water to wash away the unexposed portion and appear the engraved patterns as designed, then dry it in nitrogen stream (Figure 1h). It can be used as the microfluidic colour-changing mould now.
2.3. Fabrication of the microfluidic colour-changing devices using dry film moulds

In this paper, polymer PDMS is selected to make the colour-changing layer of the microfluidic devices due to its characters [20,21], such as high optical transparency in clean environments, less fragile and less expensive. More importantly, it is very convenient to make microfluidic devices with PDMS by using dry film moulds. The fabrication of the microfluidic colour-changing devices is based on soft lithography technology [22–24]. It mainly includes two processes: fabrication of the PDMS film with microstructures by using dry film mould and irreversible bonding with substrate [25–27].

The fabrication process of the PDMS film by using dry film mould is depicted in Figure 2a. Appropriate (according to the thickness of PDMS film to be made) mixture of liquid PDMS and curing agent at 10:1 in weight is prepared. Pour the mixture over the dry film mould and degas in vacuum environment for 1 h. Then put the whole structure into a vacuum oven at 80°C and cure it for 20 min. Peel off the partial cured PDMS film and microstructures from the dry film mould, and poke two holes at the inlet and outlet of the microfluidic patterns respectively with a 1 mm diameter puncher. One is used as liquid inlet and the other is air outlet.

The fabricated PDMS film with microstructures is bonded on the substrate (glass, resin or plastic) to form closed microfluidic channels for colour liquids circulation. In order to achieve irreversible bonding (strong enough to withstand a high supply pressure), the two surfaces to be bonded are firstly modified by using air plasma cleaner at 80 W for 35 s, which can increase the number of surface oxide sites and improve their hydrophily [28–30]. Then place the two treated surfaces together within 1 min and press gently so as to be a full contact. Finally, cure the bonding structure in a vacuum oven at 80°C for 1 h to make sure of that the bonding strength is strong enough. The specific irreversible bonding process is described in Figure 2b.
3. Results and discussions

3.1. Fidelity of the microstructures on dry film moulds

The fidelity of the microstructures on dry film moulds can directly affect the performance of the microfluidic colour-changing devices after fabrication. The major factors that affect the fidelity of the dry film moulds are investigated, including bubble, exposure time, developing time, and developer concentration.

When laminating dry film photoresist on the substrate, bubbles will be easily formed between the photoresist and substrate, resulting in irregular channel boundaries. Figure 3a shows two formed bubbles between single-layer EtertecHT-115T dry film photoresist and mirror stainless steel substrate. One of the bubbles is located on the microfluidic channel exactly, thus irregular channel boundary is formed, as demonstrated in Figure 3b. So, bubble examination after lamination is very necessary. In this research, bubble examination is carried out through micro-observations by using light microscope XSP-63B (Shanghai Optical Instrument Company, China). If the measured bubble diameter is less
than 1/5 of the minimum channel width, the bubble influence can be neglected. Otherwise, dry film lamination and bubble examination must be repeated.

Among the factors that influence the moulds fidelity, exposure time is most crucial. Figure 4 presents the microscope images of the microfluidic channels with different photoresist thickness under overexposure, underexposure and appropriate exposure, respectively. It is obvious that overexposure makes the extra photoresist around the channels unwashable, resulting in wider channel structures than mask graphics, whereas underexposure makes the photoresist unable to be polymerised completely and easily to be washed off, resulting in obscurer and narrower channel structures than mask graphics. Through a large number of experiments, the general exposure time ranges for 1–5 layers (50 µm–250 µm thick) of EtertecHT-115T dry film photoresists are obtained. Another six measurements are conducted for each obtained time range, then the optimum exposure time ranges for different thickness dry film photoresists are summarised and proposed, as illustrated in Figure 5a.

Developing process is also an important part in the mould fabrication with dry film photoresist. Underdevelopment leads to insufficient depths of channels, overdevelopment makes the dry film microstructures weak and easily to be destroyed. Commonly, 1% (mass concentration) Na₂CO₃ solution is used for development. In this paper, in order to

Figure 4. Microscope images of the microfluidic channels under different exposure times for different dry film photoresist thickness. (a) 50 µm thick, (b) 100 µm thick, (c) 150 µm thick.
investigate the influence of developer concentration on developing rate, different concentrations of Na$_2$CO$_3$ solutions are tested by experiments, as shown in Figure 5b.

Figure 5b indicates that the developing rate is not stable for the same concentration of developer solution, the longer the developing time lasts, the slower the developing rate becomes. The mainly reason is that the photoresist dissolved in the developer gradually increases as the growth of the developing time, resulting in the decreases of the developer effective concentration.

Figure 5b also provides the developing times for various dry film thickness by using different concentrations of Na$_2$CO$_3$ solutions. When the thickness is 100 µm, the developing time of 1% Na$_2$CO$_3$ is 102 s. For 0.5%, 2% and 5% Na$_2$CO$_3$, they are 184 s, 111 s and 105 s, respectively. And when the thickness is 250 µm, the developing time of 1% Na$_2$CO$_3$ is 344 s. For 0.5%, 2% and 5% Na$_2$CO$_3$, they are 490 s, 270 s and 351 s, respectively. Therefore, it is not that the higher the concentration is, the faster the developing rate becomes. The experimental results indicate that when the dry film thickness is less the 100 µm, 1% Na$_2$CO$_3$ solution has almost the same developing rate with 2% and 5% Na$_2$CO$_3$, this is the reason why it is widely used in the industry. However, when the thickness is greater than 100 µm, the developing rate of 2% Na$_2$CO$_3$ solution is obviously faster than 1% and 5% Na$_2$CO$_3$.

Table 1 presents the time-consuming comparison by using different moulds making methods. Compared with conventional photolithography way, the moulds fabrication based on dry film photoresist shows high efficiency, and no expensive equipments and professionals are
needed. However, the channels resolution is lower for dry film photoresist moulds, which are suitable for wider channels (>20 μm) prototyping or beginners in this field.

### 3.2. The applications of the colour-changing devices based on dry film photoresist moulds

Figure 6 presents the microfluidic colour-changing devices with different dimensions fabricated by using dry film photoresist moulds, including colour-changing glasses, camouflage glasses and colour-changing starfish. Figure 6a depicts the application effects, Figure 6b and 6c reveal the specific designs of the microstructures for each application. The microfluidic colour-changing glasses can be used to comfort and protect human eyes in an environment with strong lights by circulating colour liquids through the colour-changing layer. By designing disruptive microstructures on the lens and filling with proper colour liquids matched to the background environment, simple camouflage effect of the

| Depth (μm) | **Standard photolithography** | **Dry film photoresist** |
|-----------|-----------------------------|-------------------------|
| 50        | ~20                         | ~8                      |
| 100       | ~50                         | ~15                     |
| 150       | ~80                         | ~30                     |

*a*Standard photolithography: mainly consider the time spent in the steps (a)–(h) of Figure 1.

*b*Dry film photoresist: mainly consider the time spent in pre-baking, irradiation and post-baking, without including the cooling time after baking.

![Figure 6](image.png)

**Figure 6.** The applications of microfluidic colour-changing devices fabricated by dry film moulds. (a) The colour-changing effects of different microfluidic devices: (i) and (ii) colour-changing glasses, (iii) and (iv) camouflage glasses, (v)–(viii) colour-changing starfish. (b) Designs of the microstructures in Figure 6a. (c) Cross-sectional schematic of the regions indicated by the dotted yellow lines in Figure 6b.
glasses can be achieved for concealing needs in the wilderness. Inspired by some coloured animals with special colour-changing organisms [31] (such as chameleon), a colour-changing device similar to starfish is designed in this paper. With the simple liquid colour-changing layers matched to the environment around, the starfishes can camouflage and protect themselves from hunting. Each application presents good colour-changing effect and high transparency.

Other colour-changing and camouflage effects can be realised by changing the designs of microstructure on the dry film moulds. Two or multilayer structures of colour-changing devices can also be easily fabricated by this method to meet different requirements. Through targeted designing and making, the microfluidic colour-changing layers based on dry film moulds can be used in other various wearable devices for human. Meantime, they also can be applied for realising surface camouflage and display functions of soft machines/robotics, which have widespread usage in military.

4. Conclusions

From the fabrication and applications of the microfluidic colour-changing systems based on the dry film photoresists moulds, it can be concluded that the microfluidic moulds for different colour-changing devices can be rapidly realised by using EtertecHT-115T dry film photoresist and high-resolution masks. Proper selection of exposure times, developing time and concentration of developer solution for different dry film thickness can achieve good moulds performances. Different designs of the micropatterns and multilayer structures can be easily and quickly implemented by using dry film moulds. Each design and application of the microfluidic colour-changing device shows good colour-changing effect and high optical transparency. This fabrication and colour-changing ways also present new appearance modifying opportunities for other wearable devices of human and machines.

Disclosure statement

The authors declared no competing financial interests.

Author contributions

S.L. conceived and designed the research. M.Z. and S.L. wrote the main manuscript text. M.Z. did all the experiments and prepared all the figures. All the authors contributed to the discussion of the results and reviewed the manuscript.

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