

# PRESSURE TOLERANT MULTILAYERED POLYMER FILM MICROFLUIDICS BY ONE-STEP BONDING PROCESS FOR HIGH THROUGHPUT EMULSION GENERATION

Kyoung-Ik Min<sup>1</sup>, Dong-Pyo Kim<sup>1</sup>

<sup>1</sup>Pohang University of Science and Technology, Korea

## ABSTRACT

We present a simple method for fabrication of a pressure tolerant multilayered 3D polyimide film microfluidic device for generating a high throughput oil-in-water emulsion. The device fabricated can withstand pressure up to approximately 70 bars and temperature up to 200 °C, and polyimide is inert to solvents and solutions, not causing problems such as swelling as with PDMS. The triple-layered microchannels fabricated by one-step bonding process of patterned and assembled thin films are demonstrated to produce monodisperse n-hexane droplets upto a generation rate of 200 kHz. Moreover, highly uniform PLGA micro- and nanoparticles were obtained using 3D flow focusing device without channel clogging.

## KEYWORDS

Droplet, polyimide film, three dimension, high pressure, multilayer

## INTRODUCTION

In microsystem technology, the development of cost effective and efficient fabrication technique is important to make the devices readily available for various commercial and disciplinary applications.[1] In particular, droplet-based microfluidics is very useful because it enables continuous synthesis of monodisperse microcapsules in one step for applications in the areas of drug delivery, synthesis of biomolecules, and diagnostic testing.[2] In the 2D planar microfluidic devices for the preparation of monodisperse droplets often caused clogging within channel and fouling of the reactant along the channel walls. Although it has been shown that 3D fluidic geometries can overcome the fouling problems, multilayered polymer devices require laborious step-wise fabrication and suffer the instability with common organic solvents. [3, 4] Particularly, low elastic modulus of PDMS limits the working flow rates to low. Therefore, there is a strong demand for developing a simple technique for fabrication of pressure tolerant 3D microfluidic device which enables oil-in-water emulsion at high flow rate with durability.

## EXPERIMENT

Figure 1-a illustrates fabrication of the proposed 3D polyimide microfluidic device for generating microdroplets. Typically, two pieces of polyimide films each 125 μm thick (Kapton HN film, Dupont, USA) as top and bottom layers were ablated by UV laser (355 nm, ESI, USA) to form Y-shaped microchannel (200 μm wide and 60 μm deep) with in/outlet holes (1 mm diameter). A 25 μm thick adhesive polyimide film (Kapton EKJ film, Dupont, USA) that is gluey on both sides as an internal layer were punched using laser to form Ψ-shaped open-channel (60 μm wide for inner channel and 200 μm wide for main channel) with a through-hole (1 mm diameter). The 4-corners of each film were holed (1 mm diameter) to align the film patterns. For the proposed triple-layer 3D microfluidic device, the internal layer film (25 μm thick) with open channel was sandwiched between two pieces of the ablated 125 μm thick polyimide films by inserting metal pins through the holes at the film corners to be aligned. Finally the polyimide-based microchannel were bonded by pressing a set of the stacked films between two glass slides at 300 °C under a pressure of 10 kPa for at least 1 hr. The simple alignment and thermal bonding step allows multilayer stacking process.

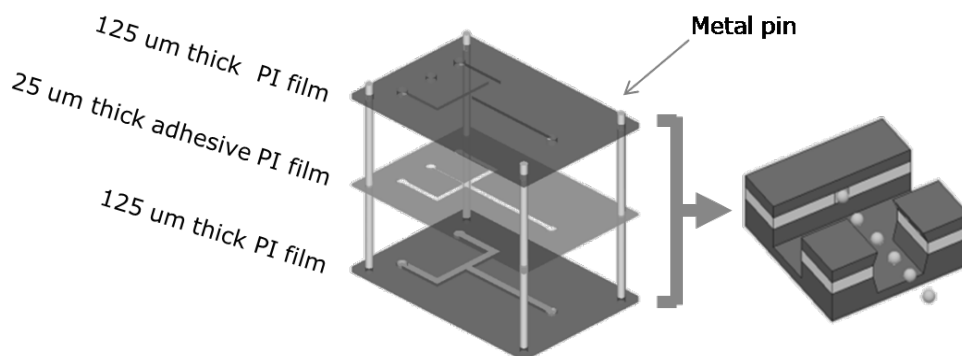


Figure 1. Schemes for triple-layered 3D microfluidic device.

To evaluate a bonding strength of the sealed microchannel, the triple-layered microfluidic device was increasingly pressurized by pumping water using HPLC pump into the inlet. The triple-layered microfluidic device with open outlet could endure the pressure even at a continuous flow rate 10 mL/min for 10 min with no

delamination. Particularly, when the device with closed outlet was subjected at pumping rate 10 mL/min, excellent pressure resistance of the sealed microchannel was also demonstrated by rendering the burst pressure of 1000 psi (~70 bar). It is worth noting that the striking pressure endurance up to ~70 bar of the polymer-based device is useful for high pressure involving applications that was required to use metal- or glass-based devices.

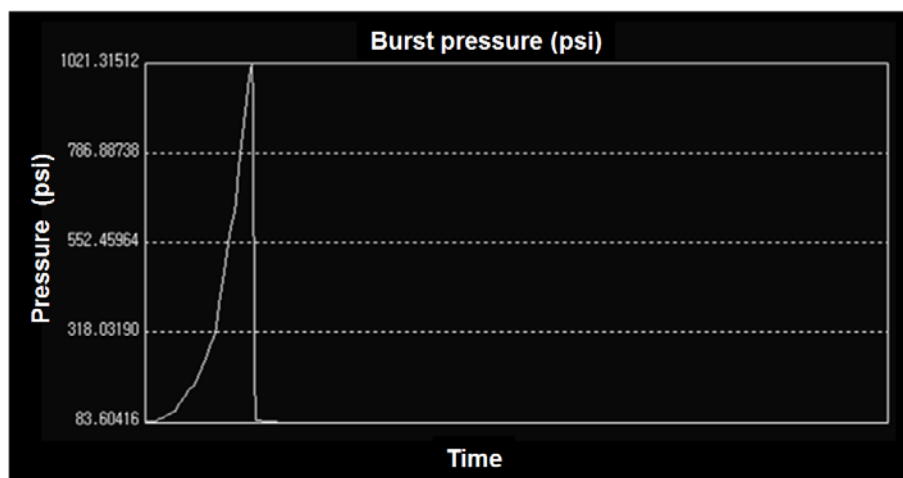


Figure 2. Actual pressure tolerance test of triple-layered polyimide film microfluidic device measured by HPLC pump. Burst pressure by pumping water at 10 mL/min into one-end open microchannel (closed outlet)

Wettability of the channel wall is important for reliable generation of water-in-oil or oil-in-water emulsions by 3D focusing flow method. Water-in-oil droplets require hydrophobic surface property as in PDMS microfluidics, while oil-in-water emulsions require hydrophilic channels as in glass microfluidics. The original polyimide film has a water contact angle of 70°, but repeated laser ablations led to a rough surface of undulations of a few tenth of micrometer, which lowered the contact angle to 50°. It is interpreted that the photochemical oxidation with UV irradiation produced hydrophilic groups on the polyimide surface such as –OH and –COOH. Therefore, the hydrophilic polyimide channel is suitable for producing stable oil-in-water emulsions. To evaluate the capability of the 3D flow focusing microfluidic device as a high throughput emulsion generator, a triple-layered microfluidic device was used to prepare hexane-in-water emulsion and PLGA particles. When the flow rate of hexane as a dispersion phase was controlled at 500  $\mu$ L/min through the inner channel, and the water as a continuous phase at 4 mL/min through the two inlets, highly monodisperse hexane droplets with 43  $\mu$ m diameter were efficiently produced without wetting problem at a high throughput generation frequency of 200 kHz. The pressure resistant polyimide film device is suitable for high throughput generation of homogeneous hexane droplets at high flow rates that were assembled into hexagonal close-packed layers. (Fig. 3-a) This throughput frequency is approximately 10 times higher in productivity than ~20 kHz of oil-in-water drop generation frequency using glass capillary. It is noted that no delamination of film microfluidic device was observed at the total flow rate of 4.5 mL/min in the main channel from junction to outlet.

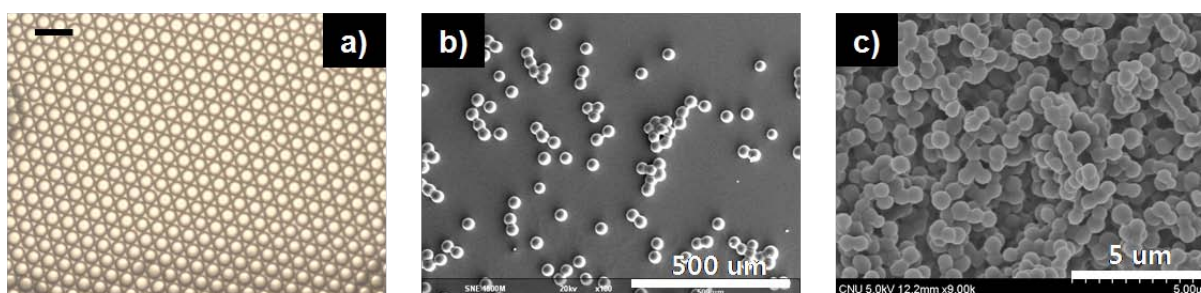


Figure 3. a) Optical image of hexane drop in water with 2 wt% SDS (scale bar is 100  $\mu$ m), SEM images of PLGA b) micro and c) nanoparticles produced in the 3D flow focusing device.

A major advantage of this 3D flow focusing geometry is that all the droplets are created at the end of the inner channel, and the formed droplets pass along the main channel surrounded by continuous phase without contacting the channel surface, resulting in no clogging and fouling along the channel. In order to confirm no occurrence of surface fouling and clogging problem, PLGA microparticles were prepared by the triple-layered microfluidic device using 3% PLGA in dichloromethane as a dispersion phase and 1% PVA aqueous solution as a continuous phase. Figures 3-b show highly uniform PLGA microparticles 35  $\mu$ m in diameter that were obtained when the flow rate of PLGA and PVA solution were 300  $\mu$ L/min and 500  $\mu$ L/min, respectively. Meanwhile, mostly 2D planar devices are

caused clogging of the channels during fabrication of nanoparticles from PLGA polymer solution, thus resulting in broad size distribution of particles. As shown figure 3-c, uniform PLGA nanoparticles were obtained by solvent extraction at the 1% PLGA in acetonitrile velocity of 300  $\mu\text{L}/\text{min}$  and continuous phase velocity of 500  $\mu\text{L}/\text{min}$ , respectively. It is noted that the film microfluidic device could be used due to 3D geometry without additional hydrodynamic flow.

The fabricated devices are quite attractive because of its ability to withstand a pressure up to 70 bars, and inertness to any chemical unlike PDMS. This simple and economical fabrication technique significantly facilitates the mass production of multilayered film devices that opens for various microfluidic applications in the areas of chemistry as well as biology.

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#### **CONTACT**

Dong-Pyo Kim 82-54-279-2272 or dpkim@postech.ac.kr