



## A new technique for ferroelectric microfluidic channels by rolling method

Jinxing Li<sup>a</sup>, Zhaoqian Liu<sup>a</sup>, Bing-Rui Lu<sup>a</sup>, Gaoshan Huang<sup>b</sup>, Yifang Chen<sup>c,\*</sup>, Yongfeng Mei<sup>b,\*</sup>, Ran Liu<sup>a,\*</sup>

<sup>a</sup>State Key Lab of ASIC and System, Fudan University, Shanghai 200433, China

<sup>b</sup>Department of Materials Science, Fudan University, Shanghai 200433, China

<sup>c</sup>MNTC, Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire, OX11 0QX, UK

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### ABSTRACT

Microfluidic channels on the size of tens of microns are being developed for use in a variety of applications such as microreactors, DNA analysis, and micro total analysis systems. Among the fabrication techniques for microfluidic channels, the recently-developed rolled-up method provides a great opportunity to integrate tubular channels by selective underetching and release of a strained thin film from a substrate. In this work, microfluidic channels with large diameter-to-length aspect ratios have been fabricated by a rolling method. The micro-FTIR characterization demonstrated that the functional poly(VDF-TrFE) ferroelectric polymer was engineered in the micro channel. The ferroelectric properties of the microchannels were studied by piezoresponse force microscopy (PFM). Local hysteresis measurement suggests that the microchannels show good ferroelectric properties. Fluid moving and emptying of the microfluidic channels were captured by video microscopy.

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## 1. Introduction

Interests in micro- and nanofluidic devices and lab-on-a chip research have grown considerably over the last decade [1]. Devices, with channels on the size of tens of microns, are being developed for use in a variety of applications such as microreactors, DNA analysis, and micro total analysis systems. With current techniques, round channel designs, which are of especially attractive functions, are still difficult to realize. Recently, a fabrication technique of micro- and nano- tubes in several material systems by selective underetching and release of a strained thin film from a substrate has been developed [2–5]. The simplicity of the fabrication process provides a great opportunity to integrate three-dimensional structures into an available semiconductor technology. In this work, transparent microfluidic channels have been fabricated by a rolled-up method with large diameter-to-length aspect ratios. The fluid flows in the microchannels can be clearly visualized under an optical microscope. Significantly, we have successfully integrated a poly(VDF-TrFE) ferroelectric polymer in the micro-channel by the rolled-up technology. The ferroelectric nature and the excellent biocompatibility of the polymer inside the channels provide a unique opportunity for the applications in protein analysis, cell encapsulation, bio actuators, drug delivery, and biosensors.

## 2. Experiments, results and discussions

The fabrication process of rolled-up ferroelectric-polymer-comprised microtubes is schematically displayed in Fig. 1. Initially, a uniform lift-off resist (LOR) layer [6], which was used as a sacrificial layer in the roll-up process, was spin-coated on a Si wafer at a speed of 3000 rpm and then pre-baked at 180 °C for 20 min. The active layer (SiO/SiO<sub>2</sub> bi-layer structure) was deposited on the LOR layer in good vacuum (<10<sup>-3</sup> Pa) by electron beam heating evaporation. The thickness ratio of 1:4 between SiO and SiO<sub>2</sub> is found to be the optimal one for the roll-up process. Then the poly(VDF-TrFE) butanone solution (4 wt.%), was deposited on the active layer by spin-coating at a speed of 2000 rpm. To facilitate the rolling-up process, the starting edge of the underetching was defined by mechanical scratching. The underetching step was conducted by putting the samples in a diluted alkali KOH solution (5 wt.%). The lift off resist, which is based on polymethylglutarimide (PMGI), was dissolved in the alkali chemicals, releasing the upper tri-layer. The intrinsic stress in the SiO/SiO<sub>2</sub> bilayer structure made the free-standing films bend up and self-assemble into a microtubular structure.

In order to facilitate the following characterization process, the rolled-up nanomembranes were then dried in the critical point dryer by using liquid CO<sub>2</sub> as inter-media to avoid the collapse of the rolled-up structures when directly moved from etchant solution to a dry environment. It is worth noting that the inner diameter of the microfluidic channels can be tuned by the total thickness of the SiO/SiO<sub>2</sub> bi-layer and the length of the microchannel can be as long as several centimeters. Fig. 2a shows an optical

\* Corresponding authors.

E-mail addresses: [y.chen@rl.ac.uk](mailto:y.chen@rl.ac.uk) (Y. Chen), [yfm@fudan.edu.cn](mailto:yfm@fudan.edu.cn) (Y. Mei), [rliu@fudan.edu.cn](mailto:rliu@fudan.edu.cn) (R. Liu).

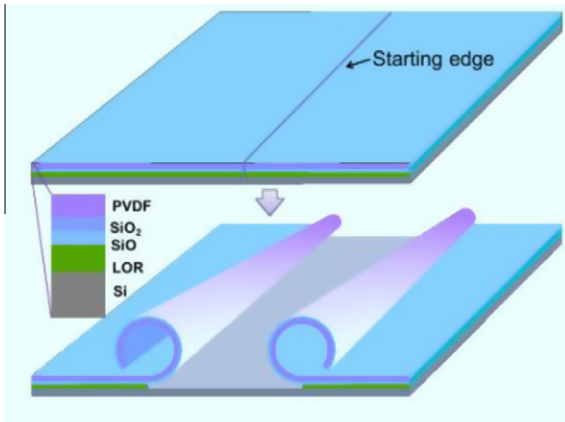


Fig. 1. Schematic representation of the rolled-up process for fabricating ferroelectric microfluidic channels.

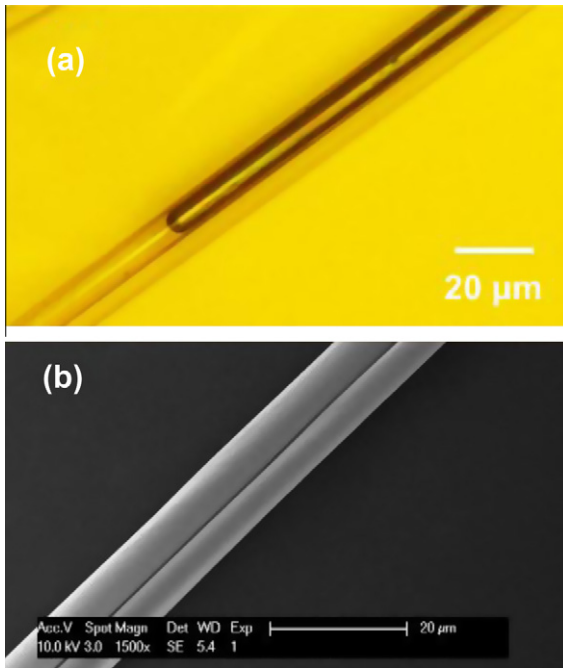


Fig. 2. (a) An optical image of a rolled-up microchannel. (b) A SEM image of dual-channel consisting of two parallel tubes rolling from opposite sides.

image of the rolled-up microchannel with a SiO/SiO<sub>2</sub> bi-layer thickness of 30 nm and a poly(VDF-TrFE) thickness of ~200 nm. The transparency of the microchannel was demonstrated by the clear observation of a micro bubble inside the channel. Fig. 2b clearly displays a SEM image of dual-channel consisting of two parallel tubes rolling from opposite sides.

To confirm the existence of ferroelectric polymer poly(VDF-TrFE) in the microchannel, micro-Fourier Transform Infrared Spectroscopy ( $\mu$ -FTIR) was used to measure the absorbance of the microchannel. The micro-FTIR Spectroscopy cooperated with a microscope has a small detection area of  $50 \times 50 \mu\text{m}^2$  can effectively exclude the influence of the polymer next to the microchannel. The FTIR spectrum of the microchannel is shown in Fig. 3. It can be seen that even though the polymer was in the inner surface of micro channel, surrounded by 30 nm thick SiO/SiO<sub>2</sub> bi-layer, all the three bands in the spectrum with dipole moments along the polar *a*, *b*, and *c* axis ( $1190$ ,  $1290$  and  $1400 \text{ cm}^{-1}$ , respectively) of the ferroelectric orthorhombic phase appear in the spectrum [7], indicating that

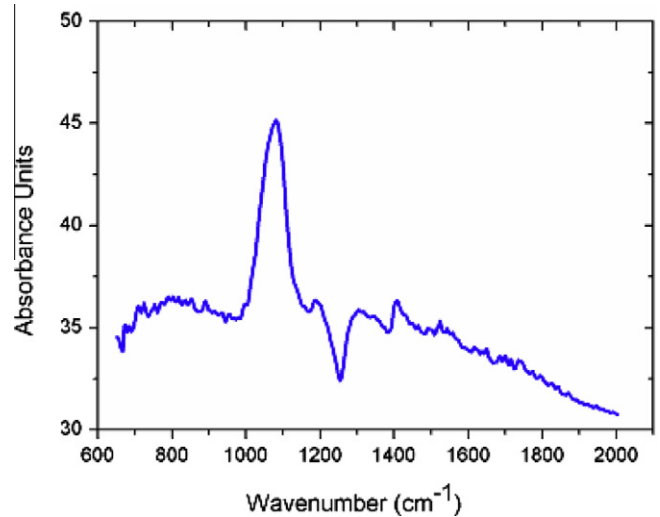


Fig. 3. Micro-FTIR spectrum of microfluidic channel with poly(VDF-TrFE) integrated.

the ferroelectric polymer poly(VDF-TrFE) is successfully integrated into the microchannel.

The rolled up channel with integrated ferroelectric polymer may have unique microfluidic applications different from conventional ones. Fig. 4 displays successive images of a microscopy video of a bubble flow in the micro channel. The flow of the bubble is believed to be caused by the different capillary pressure in the two ends of the channel [8]. The unique feature indicates that the specially designed microchannels could be integrated into microfluidic system [9] to automatically propel the flow inside the system.

Ferroelectric property of the rolled up channel could be clearly demonstrated by piezoresponse force microscopy (PFM) as shown in Fig. 5. Instead of the insulated bi-layer SiO/SiO<sub>2</sub> 50 nm platinum was used to form the electrodes required by the PFM measurement. Therefore, the planar structure before rolling process has to be altered in the first place to expose the ferroelectric polymer on the outer surface of the rolled up channel as presented in the up schematic in Fig. 5. After spin-coating LOR on the Si substrate, poly(VDF-TrFE) was first spin-coated instead of SiO/SiO<sub>2</sub> bilayer deposition on the LOR in the original structure, followed by 50 nm platinum deposition via electron beam evaporation. The Pt/poly(VDF-TrFE) bi-layer can also roll upward into micro channel once released from the substrate by etching the LOR with diluted KOH, as we can see from the AFM image of the micro channel's upper part in the inset of Fig. 5a.

Based on the newly designed PFM setup, hysteresis loop of the rolled up poly(VDF-TrFE) was obtained as shown in the bottom image in Fig. 5b, indicating that the rolled up micro channel consists of the ferroelectric polymer poly(VDF-TrFE). The hysteresis loop of

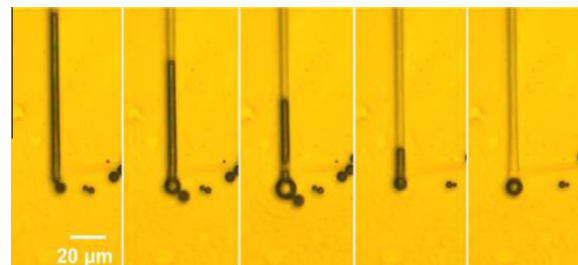
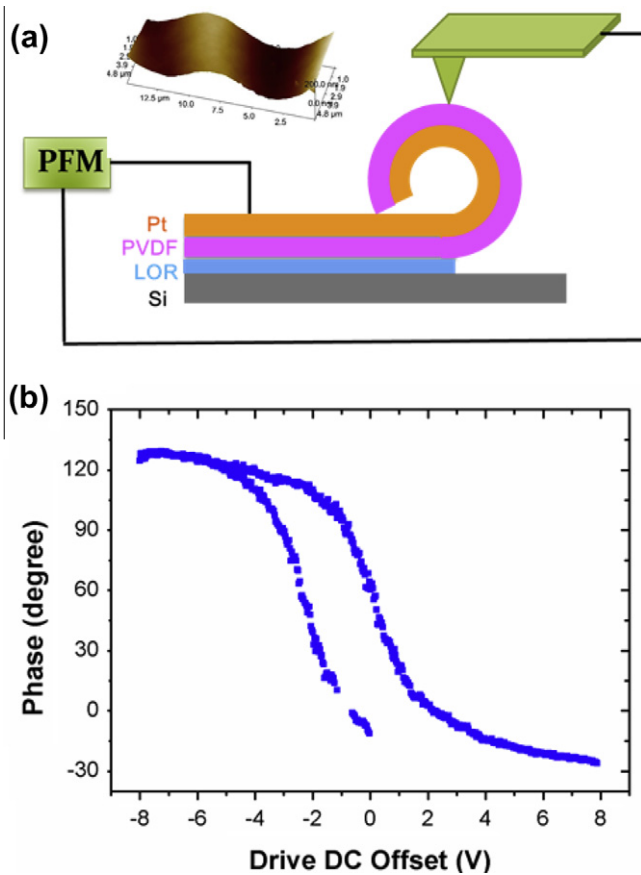


Fig. 4. Successive images of a microscopy video of a bubble flow in the microfluidic channel.



**Fig. 5.** AFM and PFM characterization for the microchannels. (a) Schematic illustration of experimental AFM & PFM set-up. The inset displays an AFM image of the upper part of a rolled-up microchannel. (b) Hysteresis loop of an integrated PVDF layer in Pt/PVDF ferroelectric microchannel.

the channel also shows good ferroelectric properties [10], which means that the fabrication of ferroelectric microchannel could be realized through this novel rolling method and further be inte-

grated into microfluidic system or lab on chip. What's more, this ferroelectric microchannel may be applied as a micro pump or micro motor since the ferroelectric tube wall could contract or dilate when positive or negative voltage was applied.

### 3. Conclusions

To sum up, we have successfully integrated the ferroelectric polymer poly(VDF-TrFE) in the microfluidic channel simply by rolled-up method. The micro-FTIR characterization demonstrated that the functional poly(VDF-TrFE) ferroelectric polymer was engineered in the microchannel. In addition, the rolled up ferroelectric polymer shows good ferroelectricity in the PFM measurement. The microfluidic application is demonstrated in our work, showing that the fluid will flow automatically due to the different capillary pressure in the channel. It is anticipated that the good uniformity and biocompatibility of this ferroelectric microchannel may have huge potentials in bio researches, like cell analysis, biochemical sensors, etc.

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