NDnano
Center for Nano Science and Technology

NDnano Summer Undergraduate Research 2017 Project Summary

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Project title: Generation of polymeric shell encapsulating microdroplets by AC Electrospray and fabrication of Silicon Nanopore array by inverted pyramid technique

New skills acquired during summer research:
I have learnt the basics of generating microdroplets using alternating current electrospray. I have also acquired various Nano-level fabrication techniques. I have undergone gowning training for the clean room. I have learnt to use machines like Evaporator, Plasma Enhanced Vapor Chemical Deposition (PEVCD), Reactive Ion Etching (RIE), Autostep and Stepper for mask making.

Abstract:
We present the method to produce microdroplets encapsulated by styrene shell by AC Electrospray method. We used the noble idea of Ohmic heating generated due to applied electric field at the tip of capillary to polymerize the styrene thus forming the shell. We have also fabricated Silicon nanopore array by inverted pyramid technique to achieve multi-target quantification of droplets.

Practical application/end use of research:
The encapsulation of liquid droplet can protect the droplet from outside deteriorating effects of environment such as oxidation and moisture particularly in pharmaceutical industry. If composition of polymer and parameters of AC spray is selected appropriately then we can make more stable droplets of serum containing DNA for PCR and then using these droplets for analysis. The benefits of nanopore array is that we can try AC spray out of the entire array but with different primers in each nanopore, thus resulting in massively large multi-target quantification.

References:
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II. Pan, Z., Wang, C., Li, M., & Chang, H. C. (2016). Universal Scaling of Robust Thermal Hot Spot and Ionic Current Enhancement by Focused Ohmic Heating in a Conic Nanopore. Physical review letters, 117(13), 134301
III. Ao, Z., Yang, Z., Wang, J., Zhang, G., & Ngai, T. (2009). Emulsion-Templated Liquid Core− Polymer Shell Microcapsule Formation. Langmuir, 25(5), 2572-2574
IV. Kuo, P. L., Turro, N. J., Tseng, C. M., El-Aasser, M. S., & Vanderhoff, J. W. (1987). Photoinitiated polymerization of styrene in microemulsions. Macromolecules, 20(6), 1216-1221
V. Deng, T., Chen, J., Wu, C. N., & Liu, Z. W. (2013). Fabrication of inverted-pyramid silicon nanopore arrays with three-step wet etching. ECS journal of solid state science and technology, 2(11), P419-P422
VI. Park, S. R., Peng, H., & Ling, X. S. (2007). Fabrication of nanopores in silicon chips using feedback chemical etching. Small, 3(1), 116-119.
VII. Bajwa, I. (2016). KOH etching of (100) Si wafer, No 2.

Project summary:
The AC spray chip setup is shown in figure 1. The styrene solution's flow is controlled by syringe pump and water solution's flow is controlled by air pump. The 10g of styrene solution's composition includes 5g of styrene, 2.088g of DVB and 2.852 g of Span-80. The water solution has 10mM KCl dissolved in it.

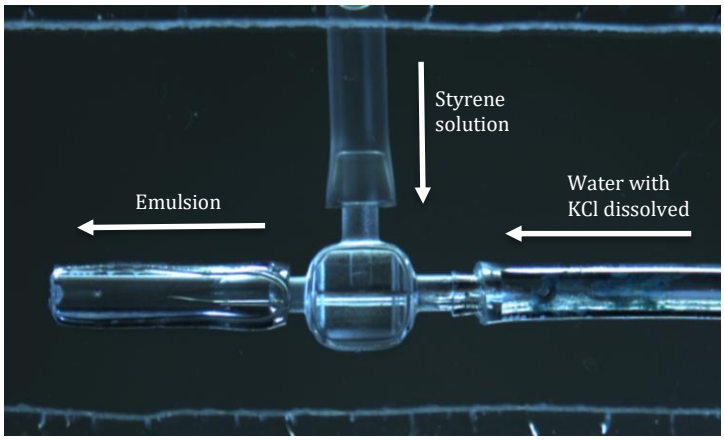

Figure 1: Typical chip setup for AC electrospray

The electric field generated by wave generator is applied to water solution. There is Ohmic heating at the tip of capillary due to applied electric field. As water is forced through the capillary, it forms droplet at tip and styrene around the droplet get polymerized because of heat thus forming shell around droplet.

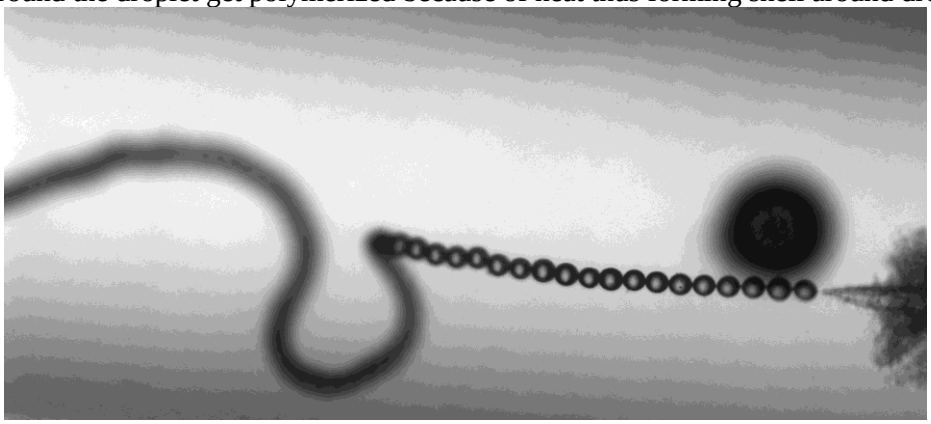

Figure 2: The formation of thread of mono-disperse droplets with styrene shell

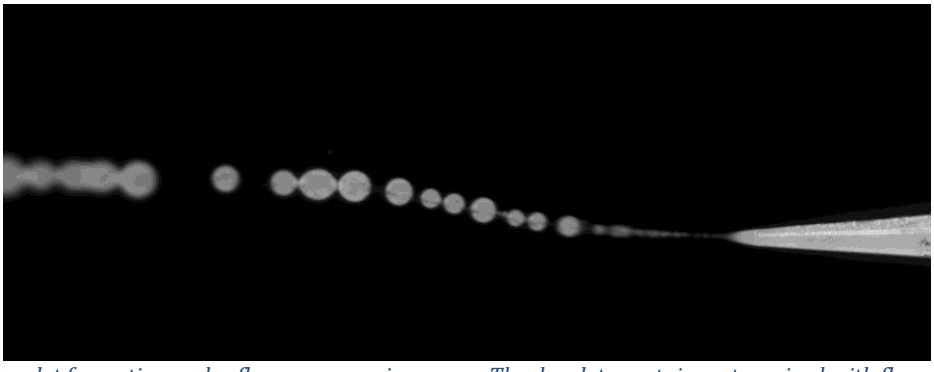

Figure 3: Droplet formation under fluorescence microscopy. The droplets contain water mixed with fluorescence die.

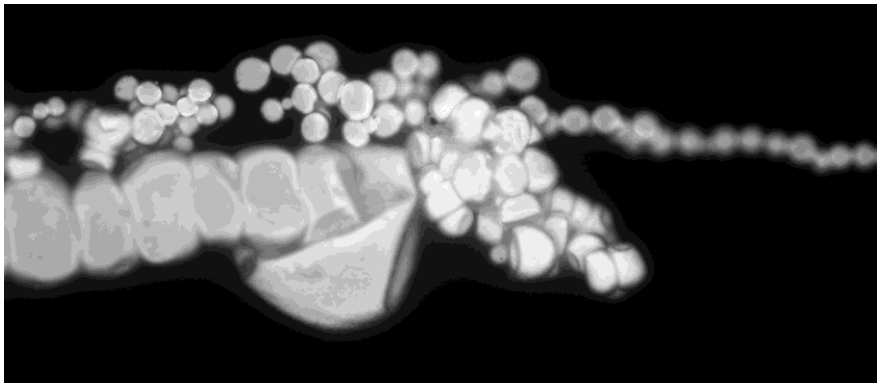

Figure 4: The droplets with distorted shape shows that there is shell covering them and it has squeezed because of liquid extraction from it

The second part of the project includes the fabrication of Silicon Nanopore Array. Silicon wafer used were 500µm thick, double-sided polished, <100> oriented wafers with diameters of 100 mm. The mask designing was done by software called L-edit. The two masks were designed and used. One with three smaller windows of size 4µm X 4µm, 5µm X 5µm and 6µm X 6µm and other with three 1200µm X 1200µm size window. These masks of size 5" are fabricated by machine called Stepper.
The process of fabricating nanopore is described as follows

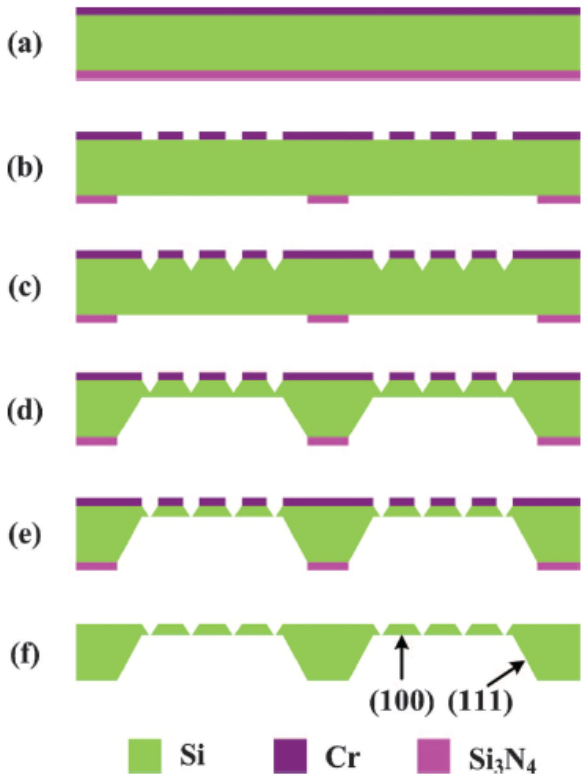

Figure 5: Steps for fabricating inverted Si nanopore [5,6]

(a) 700nm thick Si_3N_4 is deposited first using PECVD technique on Si and then 300nm thick Cr layer is deposited using evaporator machine.
(b) The photoresist is deposited on both side using spin and bake technique. Mask with smaller window is used to transfer the pattern to Cr layer using Autostepper machine and mask with larger window is used to transfer the large window pattern to Si_3N_4 layer.
(c) Grooves with slanted edges were made using KOH wet etching method at 50°C on Cr layer first.
(d) KOH wet etching at 70°C on Si_3N_4 side is done to thin the substrate at faster rate.
(e) KOH wet etching is now performed at 30°C to precisely control the opening of the pores.
(f) Layers of Cr and Si_3N_4 are removed.