ACHIEVING HIGH-ASPECT-RATIO CNT MICRONOZZLES FOR ELECTROSPRAY IONIZATION

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We report on the application of the Carbon Nanotube Templated Microfabrication (CNT-M) technique to the fabrication of microscale nozzles for use in electrospray ionization sources for mass spectrometry. Some of the electrospray nozzle structures are a factor of two longer than those made by deep reactive ion etching (DRIE) of silicon. They have been fabricated on-chip and could be incorporated with other microfluidic components to allow the production of a compact mass spectrometer capable of in-situ measurements of organic molecules for astrobiology applications.

CNT-M is a method for the creation of high-aspect-ratio microstructures by filling patterned arrays of vertically aligned carbon nanotube forests (VACNTs) with materials deposited by chemical vapor deposition [1]. The VACNTs serve as a 3-dimensional framework that is infiltrated using chemical vapor deposition with a wide variety of materials including carbon, silicon and silicon oxide, nitride and carbide.

Electrospray ionization (ESI) is a widespread and versatile ionization technique often used for analyzing large macromolecules. The usefulness of ESI stems from its ability to nondestructively ionize large molecules. Electrospray ionization works by flowing analyte-containing fluid through an emitter with a narrow aperture. A high electric potential is applied to the emitter and the fluid becomes charged. As the charged fluid leaves the emitter electrostatic repulsion disperses the solution into a fine aerosol. The aerosol droplets then undergo cycles of evaporation and division as they pass the Rayleigh limit. The end result of these cycles is charged molecules which can then be analyzed with a mass spectrometer. Electrospray ionization has the capability to operate at very low flow rates, which was the advantage of allowing long integration times to increase the signal to noise ratio of the detector and more efficiently using limited quantities of the analyte. The electrospray method also allows much of the solvent to evaporate which provides much higher concentrations of analyte in the detector and increases sensitivity.

Although previous nozzles have been fabricated by DRIE of silicon, higher aspect ratios are desired than can be achieved with by DRIE. The desired pore diameter is 10µm and the desired height is over 500µm.

CNT-M is well suited to fabricating pore structures. In previous work we demonstrated fabrication of 3 µm pores that were over 600 µm long [1]. However, the nozzle also requires producing this pore in an isolated post. Fabrication of this post has highlighted a challenge in fabricating isolated features by CNT-M. This challenge is to overcome what we have termed the "proximity" effect of iron catalyst in CNT growth. We have observed that the amount of iron catalyst present around a particular geometric feature has a pronounced effect on the straightness and maximum height of the CNT forest in the feature. On one early test sample, variations in CNT height of 100 microns were observed across a single die, and the maximum growth height was slightly below 200 microns. This variation corresponded well to the relative isolation of the features. To combat this effect a lithographic mask was designed with sacrificial walls surrounding each nozzle in order to provide additional catalyst and encourage taller and more uniform CNT growth. After implementing these changes, the maximum nozzle height increased by a factor of four. We have grown these test nozzles (see Fig. 1) with a high degree of straightness up to 500 microns, which provides the required external aspect ratio of 10:1.

References

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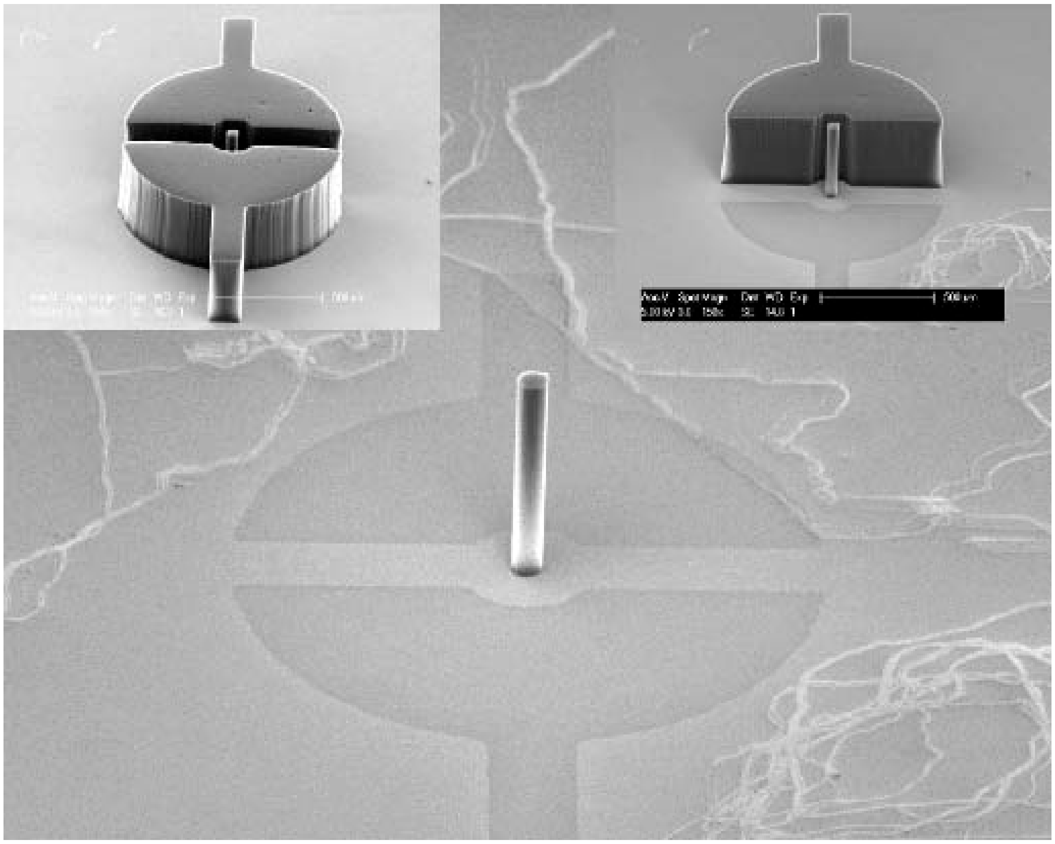

Figure 1. *Center: nozzle test structure; upper left: carbon nanotube forest with upper and lower sacrifical components, upper right: after lower sacrificial component is removed. Nozzle is about 400 microns high. Sacrificial components are about 1mm across. White lines on the substrate are tweezer marks.*