Vertically aligned carbon nanowires (CNWs)

: Top-down approach using photolithography and pyrolysis

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ABSTRACT

Top-down approach for vertically aligned CNWs has not reported yet although it is necessary for immediate integration of CNWs with micro and nano systems.

In this paper, vertically aligned pyrolyzed carbon nanowires (CNWs) is fabricated by photolithography with modification and pyrolysis as a top-down approach which the control of shape and position of CNWs is reliable. Sub-㎛ high aspect ratio (HAR) SU-8, negative tone photoresist, tip array fabricated using modified photolithography is transformed to carbon structure with shrinkage which reduces the dimension of CNWs to a few hundreds of nanometer or less. EDS analysis shows that fabricated CNWs is consist of pure carbon. The atomic structure of fabricated CNWs is amorphous as shown in XRD analysis.

We expect that the new fabrication method for vertically aligned CNWs as top down approach has a huge potential to expand an application to various micro and nano systems for their controllable positioning, array, and shape.

Keywords: carbon nanowires, vertically aligned, pyrolysis, SU-8 nano tip, high aspect ratio.

1 INTRODUCTION

A vertically aligned carbon nanowires (CNWs) offers a lot of opportunity to break through the ordinary micro and nano systems electrically, mechanically, and chemically [1]. The fabricating methods can be characterized as bottom up having the advantage of reducing scale of the structures rather than positioning at several specific points and well controlled shapes of the structures. Top-down approach for vertically aligned CNWs has not reported yet although it is necessary for immediate integration of CNWs with micro and nano systems.

In this paper, vertically aligned pyrolyzed carbon nanowires (CNWs) is fabricated by photolithography with modification and pyrolysis as a top-down approach which the control of shape and position of CNWs is reliable. Sub-㎛ high aspect ratio (HAR) SU-8, negative tone photoresist, tip array fabricated using modified photolithography is transformed to carbon structure with shrinkage which reduces the dimension of CNWs to a few hundreds of nanometer or less as shown in Figure 1.

Pyrolyzed carbon fabricated by photolithography and photoresist thermal decomposition at high temperatures in inert atmosphere has been widely studied [ref]. Pyrolyzed carbon derived from photoresist has many advantages such as batch fabrication, fine resolution, and reproducibility. For that reasons, various applications such as microbatteries, image sensors, and biochemical sensors utilizing pyrolyzed carbon have been reported [2-4].

One of popular precursor of pyrolysis is SU-8, a negative tone thick photoresist. Its high sensitivity and low absorbance for UV light offer strength of high aspect ratio micro structure fabrication [5-7]. However, the realization of high aspect ratio sharp SU-8 tip still remain as a considerable challenge for a dimensional limitation or a complexity of process as the structure is reduced to micro or nano scale. In our recent research, this limitation has been solved by modification of photolithography to apply high aspect ratio SU-8 nano tip as a middle step of a fabrication of pyrolyzed carbon nanowire array and shape estimation method has been provided using numerical analysis of Huygens Fresnel diffraction principle [8].

2 FABRICATION

The fabrication process of HAR SU-8 tip and pyrolysis for CNWs is shown in Figure 2. The fabrication process of HAR SU-8 tip on fused silica substrate is based on one step UV photolithography with exposed dose control [8]. Chromium thin film circular apertures on a fused silica substrate are fabricated by conventional photolithography and etching processes (a). These apertures define the pattern of SU-8 (SU-8 100, Microchem, Co.) as a surface mask of backside exposure process (b). In the UV exposure process, UV band pass filter (λ=365 ㎚, bandwidth=10 ㎚, 079-0550 band-pass filter, OptoSigma Co.) is used for i-line exposure into SU-8 media. An exposed dose control

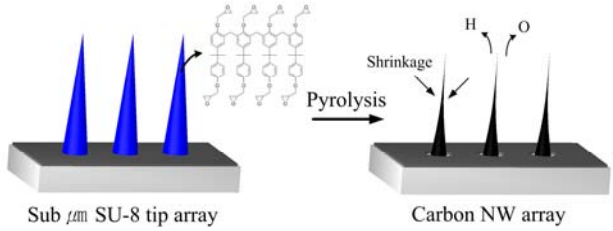

Figure 1 Schematic view of Top-down approach for CNWs fabrication.

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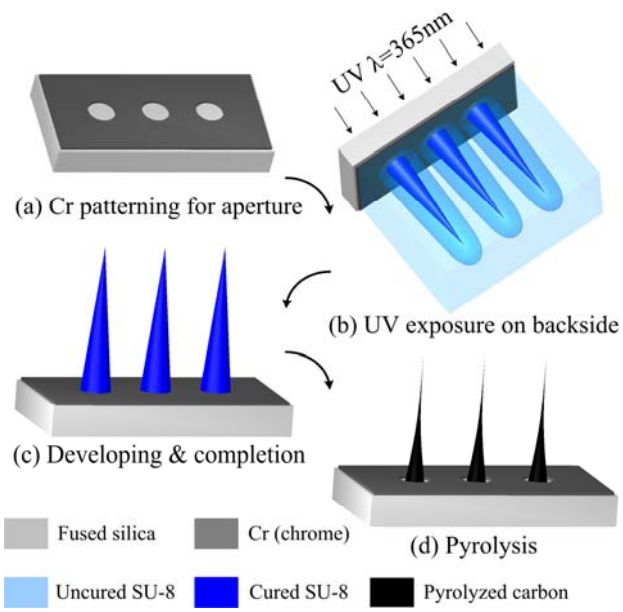

Figure 2 Fabrication process based on photolithography and pyrolysis.

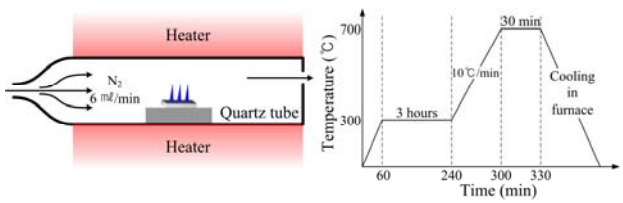

Figure 3 Pyrolysis process and temperature condition.

can produce tip shape of SU-8 whose radius is decreased. After development process, SU-8 tip array is completed (c).

The fabricated SU-8 tip is carbonized by pyrolysis process and vertically aligned CNWs following the shape of the SU-8 tip is fabricated (d). Figure 3 shows the schematic view of quartz tube furnace for pyrolysis and its temperature profile. Nitrogen gas flow of 6 ㎖/min provides an inert environment and prevent an oxidization of SU-8 tip. The SU-8 tip is dehydrated and other volatile chemicals are evaporated, in 300℃ for 3 hours and carbonized when temperature is increased to 700℃. The increasing ratio of temperature is 10℃/min. The temperature is maintained at 700℃ for 30min and cooled to ambient temperature naturally in the furnace with nitrogen atmosphere.

3 RESULTS

Figure 4 shows SEM images of the fabricated HAR SU-8 tips with φ1.0 and 1.4 ㎛ diameter of aperture on fused silica wafer for various exposed dose in photolithography. When the exposed doses are varied from 100 to 200 mJ/㎠, the fabricated tip is sharpened to sub-㎛ width by

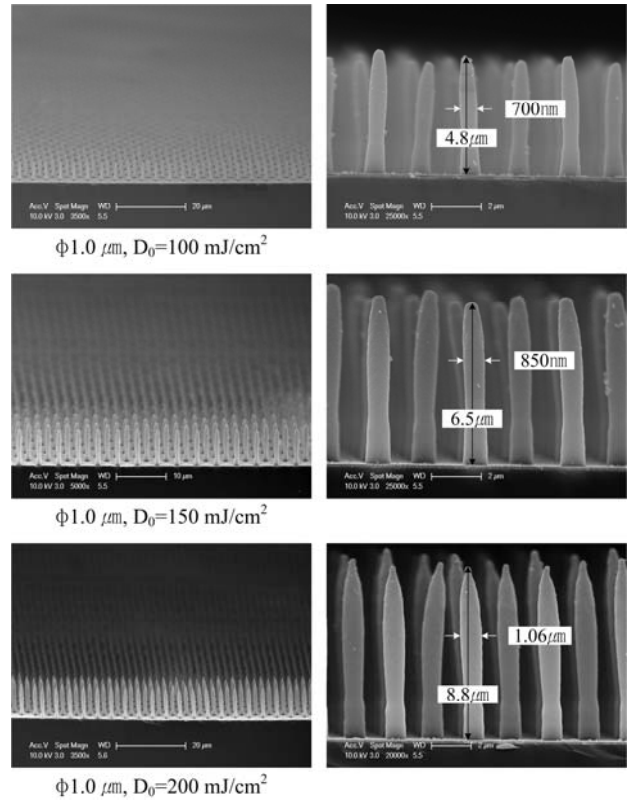

Figure 4 Fabricate results of HAR SU-8 tip array for various aperture diameter and exposed dose.

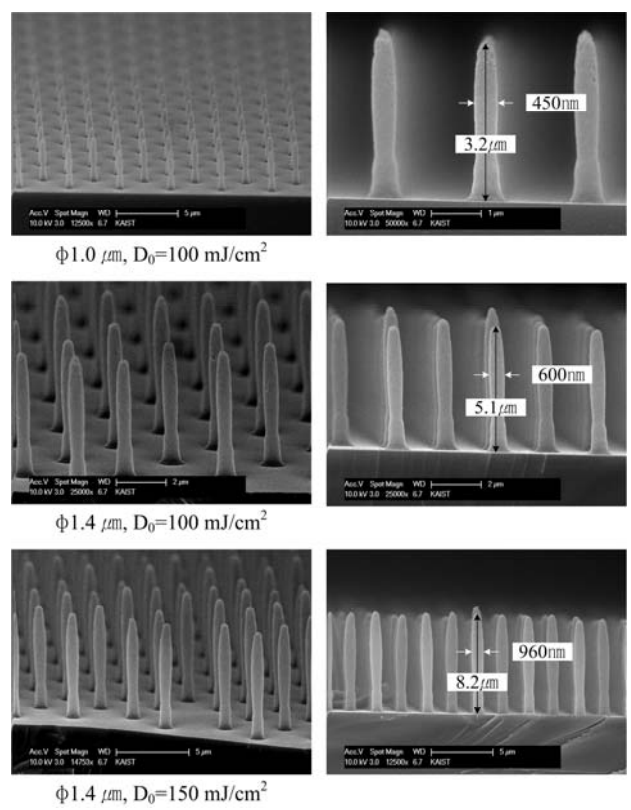

Figure 4 Fabricate results of Carbon nanowires.

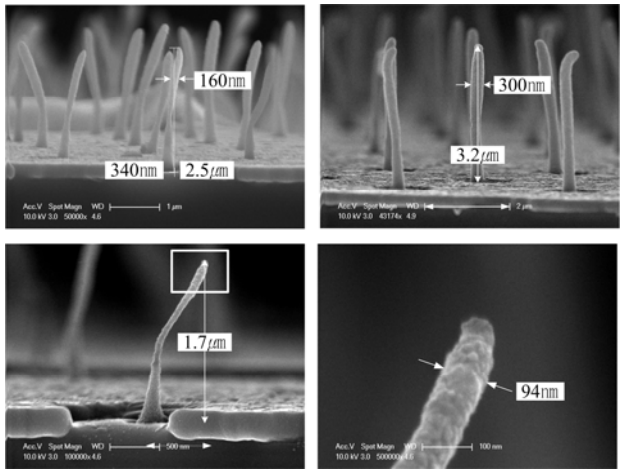

Figure 6 Occasional results of CNWs.

diffraction of exposed UV. The width of the SU-8 tips on 1 ㎛ diameter circular aperture are 700 nm, 850 nm, and 1.06 ㎛ and their aspect ratios are 6.9, 7.6, and 8.3 when the exposed doses are 100, 150, and 200mJ/㎠, respectively. It is found that the aspect ratio of the tip is increased more than 10 on a larger circular aperture. This tendency is also shown in micro scale SU-8 tip whose aspect ratio is almost 30 when the diameter of circular aperture varied from 3 to 10 ㎛ [2]. Figure 5 shows SEM images of the fabricated vertically aligned CNWs pyrolyzed from SU-8 tip array shown in previous figures. It is found that vertically aligned CNWs on fused silica wafer are well fabricated. When the SU-8 tip is pyrolyzed to CNW, a decomposition of hydrogen and oxygen induces shrinkage of dimension of the CNW. The SU-8 tip on 1 ㎛ diameter circular aperture whose height and width are 4.8 ㎛ and 700 nm is carbonized to CNW whose height and width are 3.2 ㎛ and 450 nm. The widths are 600 and 960 nm and aspect ratios are 8.5 and 8.6 when the exposed doses of SU-8 tips, the precursor of CNW, are 100 and 150 mJ/㎠ and its diameter of circular aperture is 1.4 ㎛.

The fabricated CNWs occasionally shows narrow and high aspect ratio features which width is lower than 100 nm and aspect ratio is more than 10 in Figure 6.

4 ANLYSIS

EDS analysis (EDAX, Ametek Inc.) shows the chemical composition of fabricated CNWs. It is found that fabricated CNW consists of pure carbon following the significant peak of carbon in Figure 7. The peaks of Si and O represent a fused silica wafer and the peak of Pt represents Pt coating on CNWs surface for SEM analysis. In XRD analysis using thin film X-ray diffractometer (D/MAX-RC(12kW), Rigaku Co.) a significant peak of intensity compared with reference of graphite was not found in Figure 8. It does

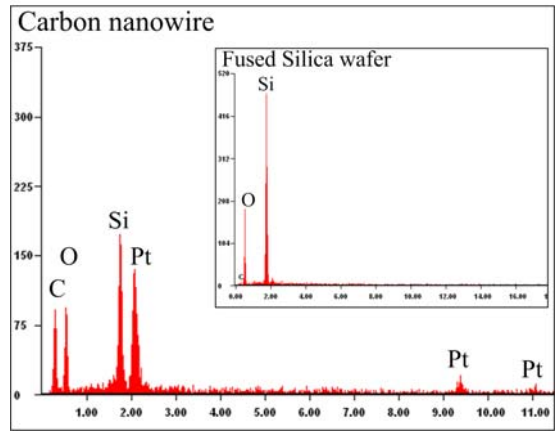

Figure 7 EDS analysis of fabricated CNW compared with fused silica wafer.

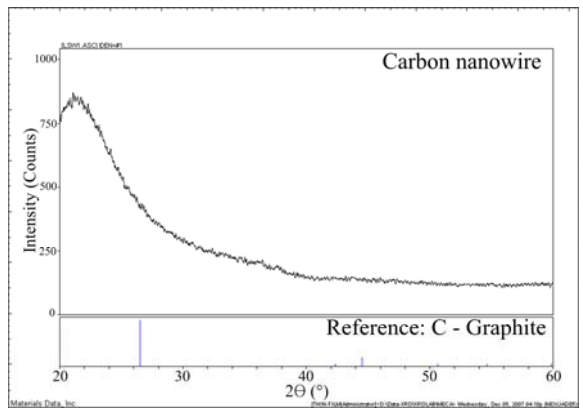

Figure 8 XRD anlaysis shows that the atomic structure of fabricated CNW is amorphous.

means that the atomic structure of fabricated CNWs is amorphous.

5 CONCLUSION

In this paper, we found that a pyrolysis process and modified photolithography can be used as top-down method of vertically aligned carbon nanowires (CNWs). Width and aspect ratio of the fabricated CNWs are several hundred nanometer width and around 8. In occasional results, it is found that width of fabricated CNWs are reduced to below 100 nm and aspect ratio is almost 20. These results mean that under 100 nm width CNWs can be fabricated by the proposed process with optimal pyrolysis condition. Hence, optimization of pyrolysis condition is needed to reduce dimension of CNWs as a future works. Furthermore, the mechanical and electric properties of CNWs can be controlled by the conditions.

We expect that the new fabrication method for vertically aligned CNWs as top down approach has a huge potential to expand an application to various micro and nano systems for their controllable positioning, array, and shape.

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