Microfabrication and Mechanical Characterization of Suspended Carbon Microstructures

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ABSTRACT

Carbon structures, in micro- or nano-scale, have spread around with widespread interest due to their potential applications in medical devices, sensor applications and microelectronics. This work highlights the successful fabrication of suspended carbon-micro and nano electromechanical systems (C-MEMS/NEMS) by UV/EB lithography and pyrolysis method. The mechanical property of these suspended structures is also investigated with nanoindentation method. Our starting material is a negative photoresist, SU-8. We solve charging problem by forming a thin metal layer before electron beam (EB) writing using various methods, such as EB evaporation, sputtering system, and thermal evaporation. By partly depositing a thin layer of a metal to prevent the repelling of negative charged electrons, we have successfully formed various suspended carbon structures, such as bridges and networks. Young's modulus for a range of load values is calculated and detailed.

Keywords: carbon-microelectromechanical systems, suspended structure, pyrolysis, photoresist, nanoindentation

1 INTRODUCTION

Carbon in nature presents itself in many forms such as diamond, graphite, buckeyballs, nanotubes, nanofibers, glassy carbon, and diamond-like carbon (DLC). Complex three-dimensional (3D) structures have been achieved and a wide range of electronic properties from wide bandgap semiconductor (diamond) to metallic (graphite) are possible because of the fact that the coordination chemistry of carbon eases the continuous mixture of single and double - bonded carbon atoms in a structure [1]. These materials are used in a variety of applications, based on their different crystalline structures and morphologies which enable very different physical, chemical, mechanical, thermal and electrical applications. It is a well-known fact that Carbon has a wide electrochemical stability window which makes it interesting for sensor applications and the fact that suspended micro/nano structures are free of Van-der-waal's interactions with the substrate makes them much more interesting for integration in mechanical, electrical, and electrochemical measurements [2]. Microfabrication of carbon structures using current processing technology includes focused ion beam and reactive ion etching. Recently a novel technology to microfabricate carbon micro structures based on UV lithography and pyrolysis process was reported [3]. However the idea of fabricating various complicated suspended three-dimensional carbon structures and maintaining the mechanical integrity is still a big challenge. Here suspended C-MEMS structures were fabricated by EB lithography and pyrolysis by accurately controlling the processing parameters and their mechanical stability is analyzed. In addition, nanoindentation is utilized to analyze the mechanical properties of suspended C-MEMS structures.

2 FABRICATION

Suspended C-MEMS structures can be fabricated in three main steps: (1) UV lithography, (2) EB lithography, and (3) pyrolysis process.

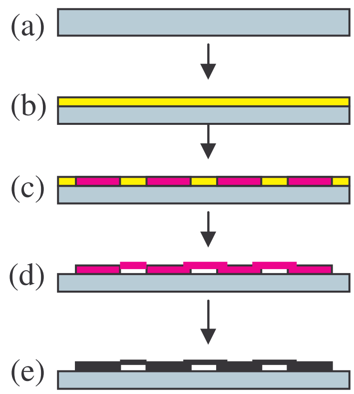

Figure 1. (a) Si substrate (100); (b) SU-8 film of 100 µm thickness was spin-coated; (c) patterning the photoresist by UV exposure; (d) patterning suspended structures by EB writer, and then developing; (e) finally pyrolyzing the photoresist patterns to convert them to carbon structures.

The substrate we used was Si (100) surface, which was spin-coated with SU-8 negative chemically amplified resist. The SU-8 resist was then patterned to get SU-8 posts which have a diameter of 50 µm and have a spacing of 50 µm between them. The patterned photoresist posts (non-developed) were then transferred to E-beam lithography system to write the desired pattern on these posts. Eventually the unexposed photoresist was washed away in developing process to leave us the suspended SU-8 structures. The suspended carbon structures were obtained by pyrolysing the SU-8 structures in a diffusion furnace at about 900^0C for 1 hr in a forming gas environment. The process flow of the fabrication steps is shown in Figure 1. Mechanical testing of the structures is then performed using NanoIndenter XP®.

3 RESULTS & DISCUSSION

Suspended thin SU-8 bridges of about 30 µm in width, 50 µm in length, and 10 µm in thickness were patterned between SU-8 post arrays. After pyrolysis, suspended carbon microstructures with about 15 µm in width, 50 µm in length, and 3-5 µm in thickness are shown in Figure 2. It can be seen that after the high temperature pyrolysis process, the carbon posts and suspended bridges shrink isometrically, retaining a major part of their structure and kept similar shapes as the SU-8 microstructures. For some of the structures, because of the unbalanced drag force, the posts were found to bend towards each other. A major problem here is charging up during EB writing on nonconductive SU-8 surface because of the accumulation of negative charged electrons. The repelling effect of the accumulated charge with the incoming electron results in further difficulty to focus and align the electron beam on surface.

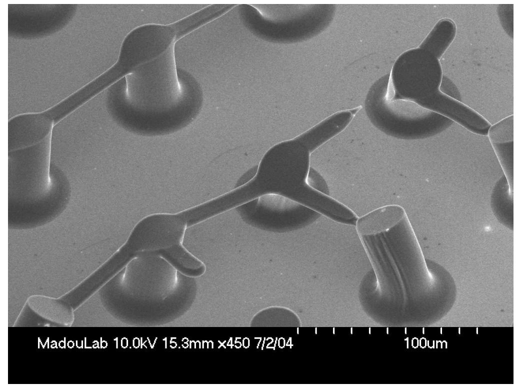

Figure 2: A typical SEM picture of carbon posts with suspended micro structures (after pyrolysis).

In order to prevent the charging of the SU-8, thin layers of gold (10 nm) were deposited by both sputtering method and e-beam evaporation method. After that EB writing was performed to pattern suspended structures. Unfortunately after removing Au layer by wet etching, it was observed that a thin SU-8 layer was formed which could not be removed by developer. It was also concluded that during both sputtering and EB evaporation processes, the SU-8 surface was attacked and exposed by the high energy ions and/or X-rays resulting in the surface cross linking and inability to pattern suspended structures.

In order to prevent both charge-up and complete cross-linking of the surface, we partially mask the area of the SU-8 posts with a dummy wafer and then evaporate Au (10 nm) on the unmasked part of the sample. Next, we conduct the routine aligning on metallized part and EB writing on unmetallized parts to obtain the desired suspended pattern. The EB written area was successfully developed. The SU-8 posts are then pyrolysed in a forming gas environment at 900^0C for one hour to convert it to carbon [4]. Figure 3 shows the perfectly horizontal suspended carbon microstructures with no bending of the carbon posts. It can be observed that the suspended carbon microstructures were about 10 µm in diameter and about 50 µm in length. In this case, since there is equal pulling force on either side of the post, the stress acting on the post is balanced. In Figure 4, it was observed that there was bowing of some of the carbon posts due to an unbalanced pulling force. In this case, there was inward bending of the carbon posts due to the radial nature of the drag force components. It can also be observed from Figure 5 that the suspended structures were patterned in a straight line. The bending of the posts occurred only at the edge posts because of unidirectional drag forces on these posts.

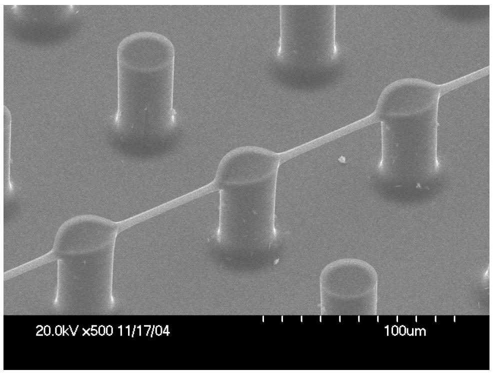

Figure 3. Typical SEM picture horizontal carbon microstructures suspended between carbon posts formed after developing and pyrolysis.

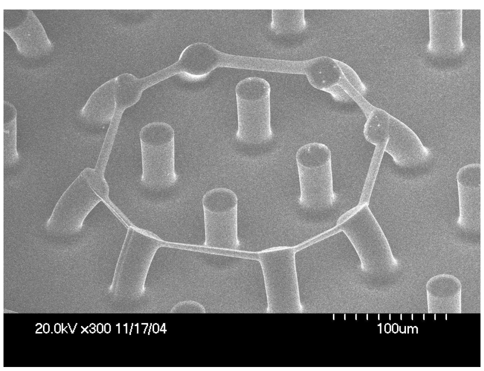

Figure 4. Typical SEM picture showing the Ring type C-MEMS structures fabricated by EBL and pyrolysis of SU-8 photoresist.

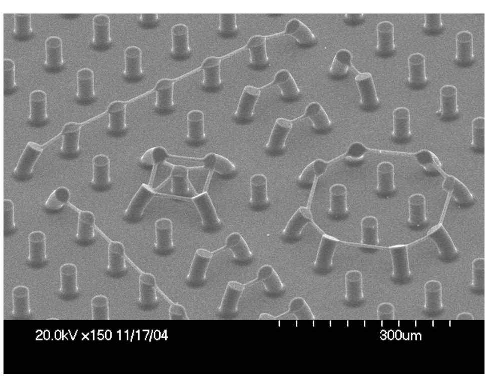

Figure 5. A SEM image of microstructures with various designs.

Mechanical characterization of the suspended structures was performed using the nanoindentation tests. Different from regular indentation measurement on thin films, the indentation tip is used to deflect the suspended beams. Physically, a pyramid-tip indenter is driven perpendicularly into the suspended beam. The beam is deflected under a fixed load, as shown in Figure 6. The overall displacement, d, includes the displacement of the beam d_b and the displacement of the tip inside the beam d_t.

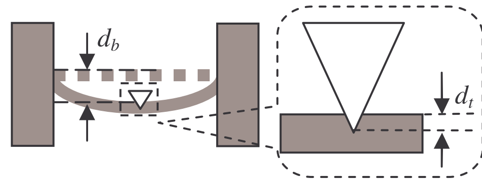

Figure 6. The deflection of the suspended beam. The overall displacement $d = d_b + d_t$.

Young's modulus E of the suspended beam can be estimated as [5]:

$$E = \frac{Pl^3}{192 \cdot Id_b} \tag{1}$$

where P is the load, l is the beam length, I is the area moment of the inertia about the centroidal axis of the beam cross section. The suspended beams used in the nanoindentation tests have a length of 50 µm and a diameter of 5 µm. The area moment of the inertia I can be calculated as:

$$I = \frac{\pi r^4}{4} = 491(\mu m^4) \tag{2}$$

Figure 7 shows a load-displacement curve of indenter on suspended beam with a maximum load as 3 mN. The maximum displacement d is recorded as 839 nm while the final displacement d_t is 253 nm. The actual beam deflection d_b is 586 nm. From Equation (1), the Young's modulus of the suspended beam is calculated as 6.79 GPa. A series of nanoindentation tests with different load forces were performed on the suspended beams. The calculated Young's moduli are shown in Table 1. The Young's moduli are not consistent in the whole force range (2-20 mN). The main reason is that the beam may not be completely elastic. After unloading, the suspended beam may not return to its original position. There exists a permanent deflection of the beam. Therefore, the tip displacement d_t is overestimated by adding the permanent deflection to the actual tip displacement. In other words, the actual beam displacement d_b is underestimated by the value of the permanent deflection. Consequently, the Young's modulus E is overestimated based on Equation (1). In general, the values calculated at lower loads are more accurate. Other reasons affected our analyses include bended posts under indentation and penetration of the 3-sided pyramid tip into the beam. To estimate the working range, higher forces were applied on the suspended beam. The beams yielded when the applied load is larger than 100 mN. The suspended beams broke under higher load greater than 500 mN.

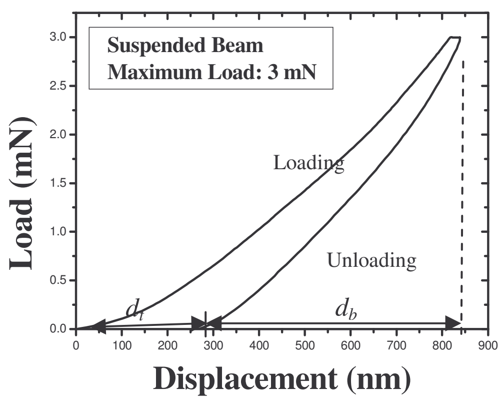

Figure 7. Nanoindentation load-displacement curve on suspended beam with a maximum load as 3 mN.

Table 1. Calculated Young's Moduli of the suspended beams under different loads.

Load, P (mN)	Beam Deflection, d_b (nm)	Young's Modulus, E (GPa)
2	411	6.45
3	586	6.79
5	807	8.22
10	1322	10.03
20	2390	11.10
100	n/a	Beam Yield
500	n/a	Yield with beam broken

4 CONCLUSIONS

In conclusion, suspended C-MEMS structures were formed by UV/EB lithography and pyrolysis method, and characterized for their mechanical properties with nanoindentation. The problem of charging was solved by forming a thin metal layer before EB writing using various methods, such as EB evaporation, sputtering system. By partly depositing a thin layer of a metal to prevent the repelling of negative charged electrons, we have successfully fabricated various complicated suspended carbon structures. Mechanical characterization was performed using the NanoIndenter, and it was found that the calculated Young's moduli are about 6.45 GPa when the load is about 2 mN.

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