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Improved Adhesion Between C-MEMS and Substrate by Micromechanical Interlocking

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Abstract **—This paper describes a new method to improve adhesion between high-aspect-ratio carbon micro/nano-structure and silicon substrate by micromechanical interlocking over conventional Carbon Micro-Elecro-Mechanical System (C-MEMS) process. Anisotropic wet chemical etching using potassium hydroxide (KOH) solution and aqueous tetramethyl ammonium hydroxide (TMAH) is applied to form various aspect-ratio and spacing pits in silicon substrate with and without a thin film layer of silicon dioxide, respectively. Great improvement on adhesion is demonstrated that the photoresist structure is found to remain robustly attached to substrate during the process of prolonged SU-8 photoresist development and immersion in heated 40% potassium hydroxide at 80℃. Furthermore, Carbon MEMS after pyrolysis process is well bonded to silicon substrate without peeling off and high-aspect-ratio glassy-carbon MEMS remain upright.**

Keywords ***—micromechanical interlocking; Carbon MEMS; adhesion;***

I. INTRODUCTION

Conventional C-MEMS process combining photolithography and pyrolysis is an effective method for fabricating various types of high-aspect-ratio carbon micro/nanostructure [1]. C-MEMS process is providing a very interesting and promising material and micro/nano fabrication approach to Li-ion battery miniaturization, active DNA arrays and a wide variety of chemical and biological sensors, such as glucose sensor [2]. However, the interfacial delamination between carbon structure and substrate over conventional C-MEMS process may prevent it from wide applications. Carbon-substrate adhesion is a critical issue in reliable device integration. Many approaches have been developed to improve interfacial adhesion, such as the use of surface treatments on substrate or chemical adhesion promoters added into photoresist. But they are still limited in improvement of long-term reliability for the device made from C-MEMS process [3]. Micromechanical interlocking has also been proposed and used to improve adhesion between silicon and parylene films, furthermore, improved resistance to delamination during prolonged exposure to hydrofluoric acid buffer solution combined with sonic agitation qualitatively demonstrates the effectiveness of the method. Similarly, micromechanical interlocking also can be a promising method to improve adhesion between C-MEMS and silicon substrate through shaping pits in the surface of silicon substrate.

In this work, pits with various spacing and depth is defined by anisotropic wet etching using KOH and TMAH in silicon substrate, then SU-8 photoresist layer is spin-coated to fill the pits completely, finally photolithography and pyrolysis. Consequently, a structure of robust adhesion between C-MEMS and substrate can be achieved. Fig.1 shows the improved C-MEMS process flow. The effectiveness to improve adhesion by micromechanical interlocking during C-MEMS process is qualitatively assessed through prolonged development time of SU-8 photoresisit and immersion of SU-8 structure in heated KOH, also by whether high-aspect-ratio glassy-carbon MEMS retain upright after pyrolysis.

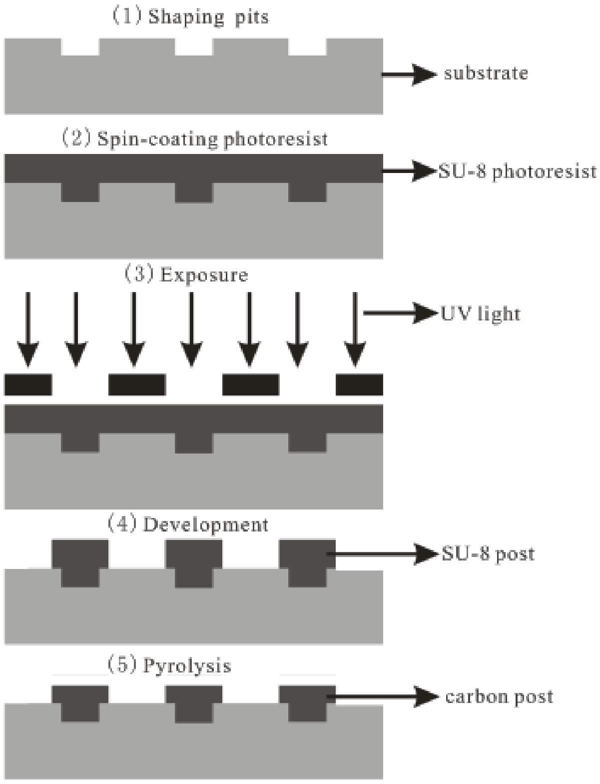

Figure 1. Schematic of the improved C-MEMS process flow

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II. MICROMECHANICAL INTERLOCKING MODELING AND DESIGN

Adhesion may be defined as a force which applied to the interface between two materials to join them together and resist separation. Up to the present, the mechanisms of adhesion are still not fully understood and there exists various points of view and keeps a sharp disagreement about the mechanisms of adhesion in academia field. In general, the seven main mechanisms of adhesion which have been proposed are to be found in the literature, namely: adsorption, electrostatic, diffusion, chemical bonding, weak boundary layer effects, mechanical interlocking and polar theory. Rarely do such mechanisms act in isolation, but rather there are overlaps [4,5].

Micromechanical interlocking is constructed by penetrating a material into some configurations of the irregular pits in the surface of the substrate, its simulating schematic diagram is as follows in Fig.2 and Fig.3. Under this condition, the major source of interfacial adhesion is arised from micromechanical interlocking of the material into the irregularities of substrate surface. At the same time, the adsorption caused by interfacial van der Waals bonds between molecules of two different materials or chemical bonds also can play an indirect important role to the adhesion.

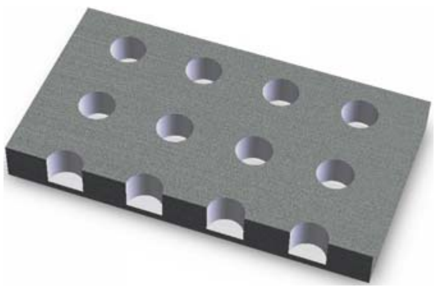


Figure 2. The simulating graph of the pits in the substrate

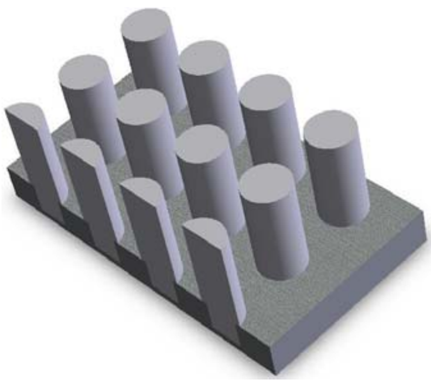


Figure 3. The simulating graph of adhesive post bonded to the substrate by micromechanical interlocking

The irregularity of substrate surface fall into three models as depicted in Fig.4. Compared to smooth substrate surface, the pits in substrate produce mechanical constraint to adhesive materials, which stop the materials from peeling off substrate and make two different materials join stronger. Apparently, the "ink-pot" type pits in Fig.4 partially act to close the delamination of the interface, furthermore, the higher values of θ_1, the more pronounced effect. But, along with θ_1 increasing, the adhesive material is more difficult to fill the pits completely. Therefore, we must optimize geometric structure of the pits.

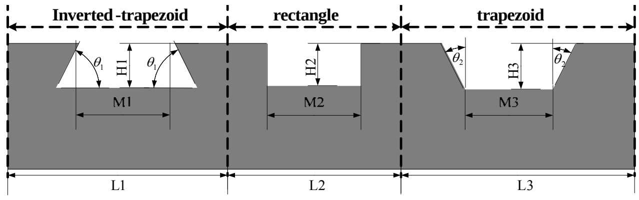


Figure 4. Cross-section schematic of the irregularity of the substrate surface

The van der Waals force can be expressed as follows:

$$F_{\mathrm{v}} = \frac{SA}{6\pi d^3} \tag{1}$$

Where A is Hamaker constant, d is the distance of two contact surfaces and S is the contact area. From (1), we know that the adhesion increases with the contact area between dissimilar materials under similar conditions.

Among all three models in Fig.4, the contact area is much larger than smooth substrate, which results in better adhesion compared to without using micromechanical interlocking. Fig.4 shows geometric parameters of three types pits, we assume that the following condition holds:

$$M1 = M2 = M3 \tag{2}$$

$$L1 = L2 = L3 \tag{3}$$

$$H1 = H2 = H3 \tag{4}$$

but also the area of vertical sidewall is M, the project area of substrate is N, namely the surface area of smooth substrate. Under these conditions, we can obtain contact surface area:

$$S_1 = N + 2M / \sin\theta_1 + 2M \cdot \cot\theta_1 \tag{5}$$

$$S_2 = N + 2M \tag{6}$$

$$S_3 = N + 2M / \cos\theta_2 - 2M \cdot \tan\theta_2 \tag{7}$$

where the inclination angle $\theta_i \in [0,90]$, i=1, 2 with 1 and 2 for inverted-trapezoid and trapezoid, respectively.

$$\begin{aligned} S_1 &= N + 2M/\sin\theta_1 + 2M \cdot \cot\theta_1 = N + 2M\frac{1+\cos\theta_1}{\sin\theta_1} \\ &= N + 2M\sqrt{\frac{(1+\cos\theta_1)^2}{\sin^2\theta_1}} = N + 2M\sqrt{\frac{(1+\cos\theta_1)^2}{1-\cos^2\theta_1}} \\ &= N + 2M\sqrt{\frac{2}{(1-\cos\theta_1)} - 1} \geq N + 2M = S_2 \end{aligned} \tag{8}$$

$$\begin{aligned} S_3 &= N + 2M/\cos\theta_2 - 2M \cdot \tan\theta_2 = N + 2M\frac{1-\sin\theta_2}{\cos\theta_2} \\ &= N + 2M\sqrt{\frac{(1-\sin\theta_2)^2}{\cos^2\theta_2}} = N + 2M\sqrt{\frac{(1-\sin\theta_2)^2}{1-\sin^2\theta_2}} \\ &= N + 2M\sqrt{\frac{2}{(1+\sin\theta_2)} - 1} \leq N + 2M = S_2 \end{aligned} \tag{9}$$

$$S_3 = N + 2M\sqrt{\frac{2}{(1+\sin\theta_2)} - 1} \geq N \qquad (10)$$

By aforesaid calculations, we can obtain the following result:

$$S1 \geq S2 \geq S3 \geq N \qquad (11)$$

And further we also can get by the combination of (1) and (11):

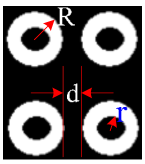
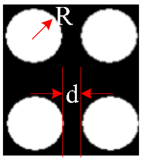
$$F_1 \geq F_2 \geq F_3 \geq F_0 \qquad (12)$$

Where F_i represents van der Waals force, i=0,1,2 and 3 for smooth, inverted-trapezoid, rectangle and trapezoid, respectively.

If we assume that chemical bonds could be formed between adhesive material and substrate, the large contact area is beneficial to form more chemical bonds , which make the interfacial joint strength larger when the project area is equal.

All analysis above shows that the inverted-trapezoid in substrate is the most favourable structure to the improvement of the adhesion, rectangle take second place, trapezoid third place, smooth the poorest. It also exists optimum geometric parameters for each structure of three, because all kinds of structure is easy to produce stress concentration which could result in fracture in the internal of adhesive material. Especially, the inverted-trapezoid can most greatly improve the adhesion, but it is also the easiest to produce significantly larger stress concentration than other. As a result, the attainment of adhesive failure stress is prior to the delamination between adhesive material and substrate. To achieve the maximal degree improved adhesion, we must carefully select and design parameters, such as H, L, M ,θ and density of the pit in substrate.

TABLE1 RIE AND ANISOTROPIC WET CHEMICAL ETCHING MASK FEATURE DIMENSIONS AND SPACINGS

Mask features		
Dimensions(r:μm) Spacings(d:μm)	R=30,40,50,60,70 r=20 d=20,30,40,50,60	R=30,40,50,60 d=20,30,40,50,60

III. EXPERIMENTAL

Polished 2# silicon wafers with and without a thin film layer(about 300nm) of silicon dioxide are cleaned for 15 mins in a solution of H_2SO_4 and H_2O_2 mixed in volume ratio 2:1 at 150℃, respectively. Then baking substrate for 15 mins in a 200℃ convection oven. After that positive photoresist BP218 is spin-coated by spinning at approximately 500rpm for 15s and 3500rpm for 40s and then baking photoresist for 2mins in a 100℃ hotplate. Exposure to define openings to the underlying silicon dioxide is carried out in a Karl Suss MA6 lithography tool for about 50s. Mask pattern transfered to underlying silicon dioxide is achieved by wet etching in a 20% hydrofluoric acid buffer solution and RIE , respectively. The silicon dioxide film layer is aimed to achieve micromechanical interlocking with overhanging profile similar to inverted-trapezoid. Subsequently, silicon is wet chemical etched by KOH and TMAH solution to form various aspect-ratio and spacing pits regarding patterned silicon dioxide as mask or photoresist, respectively. The shapes and dimensions of the mask used during the experiments is depicted in table 1. The various depth of the pits is achieved by changing etching time, solution concentration and temperature. Finally, photoresist is removed and samples are thoroughly rinsed in deionized water and dried in the oven. The primary process is to follow the schematic presented in Fig.5. As Fig.6 shows an example of substrate with many pits fabricated by the above process.

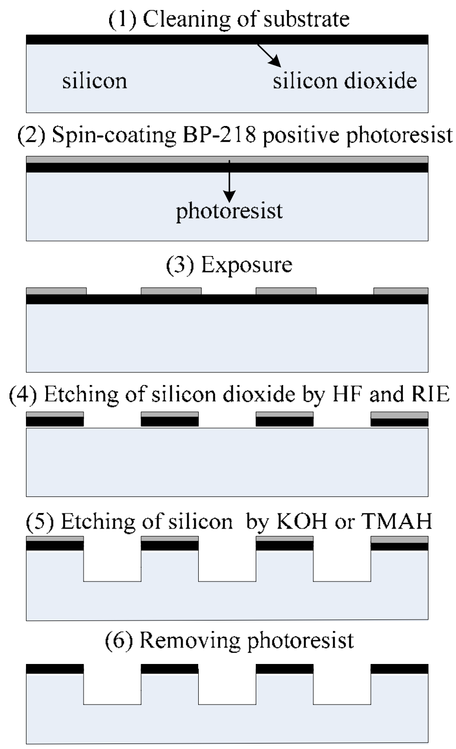


Figure 5. Schematic of the process flow etching pits in substrate

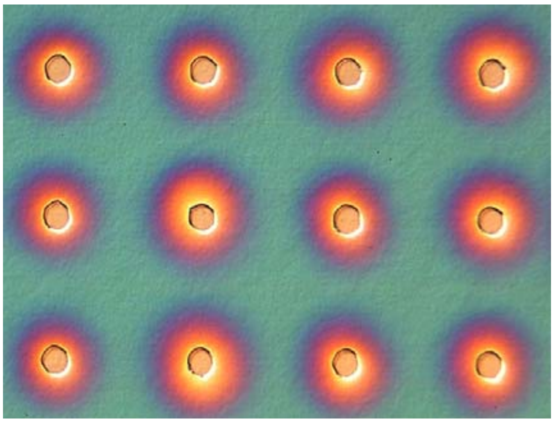


Figure 6. The pits in substrate to form micromechanical interlcking

SU-8 is then spun using two-stage process to encourage uniform spreading and pits filling. Prior to spinning, applied

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SU-8 is required to spread over most of substrate surface by simultaneously tilting and rotating , a settling period of 10 mins is required to allow for etched pits to be filled completely. Also, it is need to stand for several hours in order to relax stress sufficiently before development. In the end, C-MEMS structure derived from SU-8 photoresist can be obtained by photolithography and a three-step pyrolysis process in an open ended quartz-tube furnace.

IV. RESULTS AND DISCUSSIONS

Qualitative adhesion assessment is carried out by immersing samples of SU-8 photoresist structure in heated 40% potassium hydroxide solution at 80℃. The structure of SU-8 photoresist almost peel off smooth substrate after about half and an hour, however, the pitted substrate still remain significantly bonded to SU-8 structure after 5-10 hours. The results agree with the previous theoretical result that the adhesion of the substrate could be improved on pitted substrate as compared to smooth substrate. It is also found that the pits etched by TMAH could endure longer time than KOH without delamination between SU-8 structure and substrate under similar conditions. In [6], it is reported that the surface roughness of the substrate is significantly different after etching by KOH and TMAH solution, but also KOH solution could produce smaller surface roughness. We can conjecture that the adhesion strength is significantly influenced by surface topography. Further, adhesion strength improves as surface roughness increases. The result could be interpreted that the rough surface is composed of a large number of negligibly small pits, which produce micromechanical interlocking effect to improve adhesion. It also indicates that micromechanical interlocking is helpful to improve adhesion between adhesive and substrate. By pyrolysis, C-MEMS derived from based-micromechanical-interlocking SU-8 structure retain fully straight. Fig.7 shows carbon microstructure prepared by conventional C-MEMS process. Fig.8 shows carbon microstructure prepared by combining conventional C-MEMS process and micromechanical interlocking. The difference of the process between Fig.7 and Fig.8 only lies in whether or not use micromechanical interlocking. It is well to know from Fig.7 and Fig.8 that micromechanical interlocking is helpfull to keep carbon post straight and not peel off substrate.

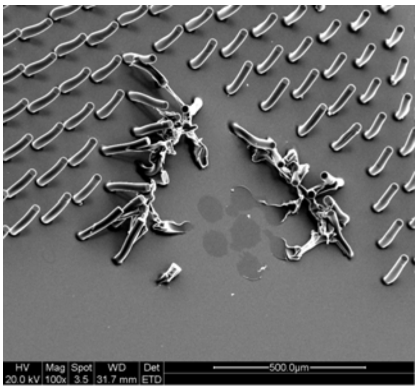

Figure 7. Collapse of high-aspect-ratio carbon microstructure prepared by C-MEMS without micromechanical interlocking

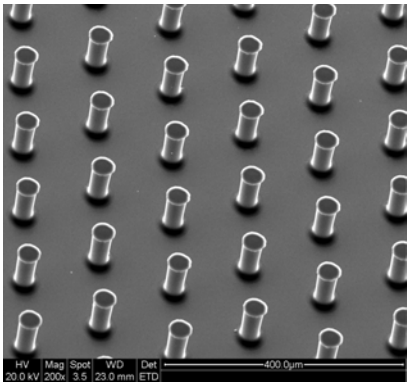

Figure 8. Straight high-aspect-ratio carbon microstructure prepared by C-MEMS with micromechanical interlocking

V. CONCLUSIONS

Micromechanical interlocking is a promising technology which can greatly improve interfacial adhesion of two different materials. In this work micromechanical interlocking and C-MEMS is successfully combined to fabricate high-aspect-ratio carbon post array. Collapse appearing during conventional C-MEMS process is effectively solved. The preliminary qualitative results provide a basic foundation for further studies in the enhancement of the adhesion by optimizing the dimensional parameters of the pis in the substrate.

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