ROBUST PARYLENE-TO-SILICON MECHANICAL ANCHORING

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ABSTRACT

This paper describes a new technique for strongly anchoring parylene (poly-para-xylylene) layers on a silicon substrate. Parylene has gained interest for MEMS applications due to its excellent properties. More specifically, because of its flexibility (Yong's modulus =4GPa), its chemical barrier properties, its conformal deposition and its biocompatibility, parylene is of great interest for microfluidics and BioMEMS. One of the issues with parylene processing is adhesion and delamination problems, occurring during fabrication or during device operation. Here, we report a new technique for anchoring parylene films on silicon using DRIE-etched trenches and anchors. We demonstrate a new way to completely protect the adhesion of parylene even when exposed to aggressive chemicals.

INTRODUCTION

Parylene is conformaly deposited by means of CVD at room temperature. Pinhole-free films with thicknesses varying from 0.1μm to 20μm can be obtained. Parylene is compatible with conventional low-temperature microfabrication techniques (evaporation, sputtering, lithography, wet etching...) and can be patterned by oxygen plasma with an etching rate comparable to that of photoresist. Several parylene-based devices have been demonstrated [1,2]. Parylene has low gas permeability and is inert to most chemicals. In fact, to our knowledge there is no wet-etchants for parylene at room temperature. Good adhesion of freshly deposited parylene films can be achieved by A-174 adhesion promotion prior to deposition However, several chemicals used in microfabrication such as HF, BHF, piranha solution and acetone have been found to attack parylene/silicon interfaces or parylene/silicon dioxide interface. This causes problems during surface cleaning or photoresist stripping when dealing with substrates having patterned parylene structures. In this work we propose to use a combination of anisotropic and isotropic silicon etching using DRIE to create anchors to be filled with parylene, taking advantage of the conformal nature of its deposition. We will first demonstrate the effectiveness of this technique by testing the adhesion of patterned parylene structures with different anchors shapes and depth or with no anchors at all. We will then show that such anchors can be used to encapsulate "islands" of different materials (aluminum, silicon dioxide and photoresist) into parylene while providing good adhesion with the silicon substrate in order to ensure lasting protection even when exposed to BHF, acetone or other aggressive solutions and environments over extended periods of time. Figure (1) illustrates the concept.

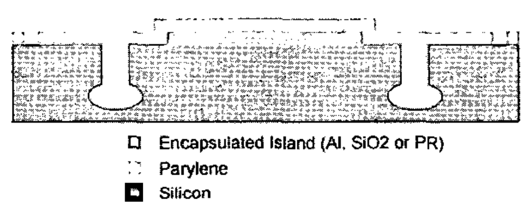


Figure 1: Island and Anchor concpt

FABRICATION

Parylene Anchoring

Figure (2) shows the process flow used to create the anchors. First, we use 20 to 60 loops of conventional Bosch process in a Plasmatherm DRIE to anisotropically etch 10μm to 30μm anchors and trenches into the silicon substrate. This etching ends with the anisotropic etching of the passivation layer, leaving a fresh silicon surface in the bottom of the trenches and passivation on the walls. We then add an SF6 etch step to create "mushroom-like" profiles. The SF6 step was chosen to be as long as 4 to 6 etch steps of our Bosch process (i.e. 24 to 36 seconds). Different anchors and trenches patterns were

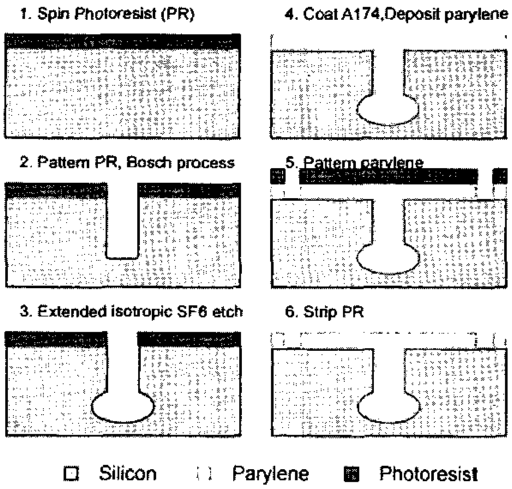


Figure 2: Process Flow

produced: circular anchors, cross-shaped anchors, continuous trenches, discontinuous trenches... After photoresist stripping and oxygen plasma ashing, an A-174 silane [5] adhesion promotion step is perfomed. The wafers are submerged in H_2O:IPA:A-174 100:100:1 for 30 minutes, then air-dried, rinsed in IPA for 15 seconds and air-dried again. It was found that baking the wafers at 60°C for 15 minutes significantly improves parylene adhesion. A 2.5μm is deposited on the wafers using a PDS2010 Labcoater from Specialty Coating Systems. The parylene layer is then patterned to isolate different regions of the dies, each region having a different anchoring pattern Parylene etching is performed using photoresist as a mask in a plasma etching system (200mT O_2, 400W). The etch rate is about 0.2μm/min. After dicing, the samples are exposed to 49% HF, BHF and acetone for various periods of time, with or without ultrasonic agitation. Finally, the samples are subjected to peeling test using adhesive tape.

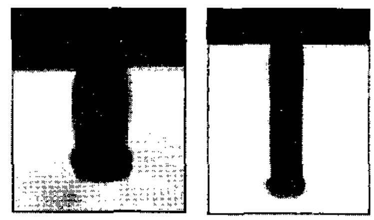
Protected IsLands

The process-flow used to fabricate the encapsulated islands of silicon dioxide and aluminum And photoresist is based on the process described above. The island material: 2.2μm thermal oxide or 2000Å aluminum is grown/deposited and patterned. Then the surrounding trenches are patterned by DRIE using the described above. After photoresist stripping an oxygen plasma ashing is performed to ensure a clean surface for the parylene deposition. For the photoresist islands, the process starts with the growth of a 5000Å layer of thermal oxide. This layer is patterned with the trenches pattern. A 3μm photoresist layer is spun, baked and the islands are patterned. Then the trenches are etched by DRIE using the thin oxide layer as a mask. The A-174 adhesion promoter is applied on all substrate as described before. Finally, a 3μm parylene is deposited and patterned. Again, the samples are then exposed to BHF, HF or acetone with or without ultrasonic agitation.

RESULTS

Parylene Anchoring

Figure (3) shows examples of obtained cross-sections, 10μm and 30μm deep. Longer SF6 etching durations were found to widen the whole trench. This suggests that the passivation was completely etched during the isotropic silicon etching.



(a) 10μm-deep trench (b) 30μm-deep trench

Figure 3: Obtained Cross-Sections (optical microscope)

Figure (4) and (5) show samples after 1 hour in BHF. As expected, delamination occurs but is reduced by the presence of anchors arrays. On the other hand, the regions isolated by trenches are totally free of delamination. Comparable results are obtained after longer exposure to BHF (up to 24 hours). Figure (6) shows a sample after 2 minutes spent in 49% HF. As can be seen, the regions surrounded by continuous a continuous trench as opposed to the portion of parylene that had a regular

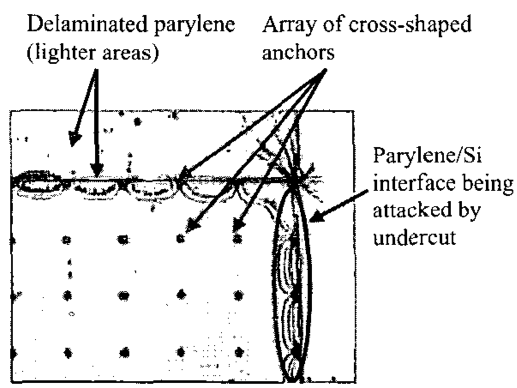


Figure 4: Surface after 1 hour in BHF

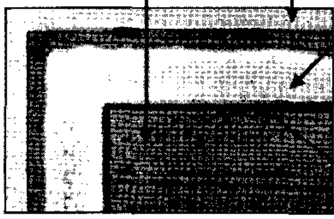


Figure 5: Surface after 1 hour in BHF

parylene/silicon interface. However, this figure also shows that HF penetrates the parylene from the top and attack the interface (clear spots). No difference was observable between the samples having 10µm-deep trenches and those having 30µm-deep trenches. Annealing of the parylene films at 180°C was found to slightly improve resistance to HF.

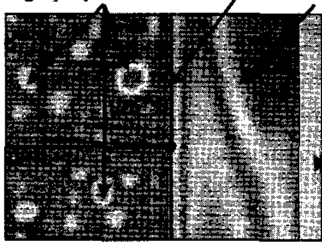


Figure 6: Surface after 2min in 49% HF

Figure (7) shows a sample after annealing, 2 minutes in 49% HF and peeling test. It can be observed that only the parylene that was surrounded by a continuous trench remained on the substrate.

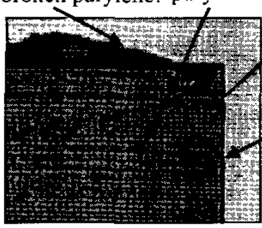


Figure 7: After 2 minutes in 49% HF and peeling test.

Region having arrays of anchors were found to have little effect on the peeling test outcome. On the other hand, adhesion is significantly improved in regions having trenches. Figure (8) shows a portion of parylene film peeled away from the substrate.

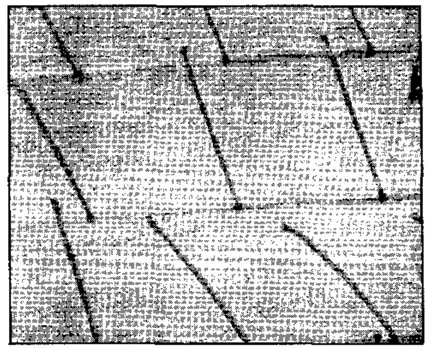


Figure 8: Parylene film torn off the substrate.

Clearly the aspect ratio of the parylene "legs" is much higher than that of figure (3.b). It is believed that the parylene entered plastic regime before being peeled off.

Protected Islands

Figure (9) shows a sample with Al islands after 1 hour exposure to BHF with ultrasonic agitation. The Al island on the right is surrounded by a double circular trench and covered with parylene. The left island is not surrounded by any trench. It can be clearly seen that the unprotected island is attacked by the BHF from the side as the center is still unaffected. On the other hand, the island surrounded by a continuous trench is unaffected. After 24 hours in BHF, all the aluminum but that surrounded by continuous trenches has disappeared. The islands surrounded by trenches are unaffected.

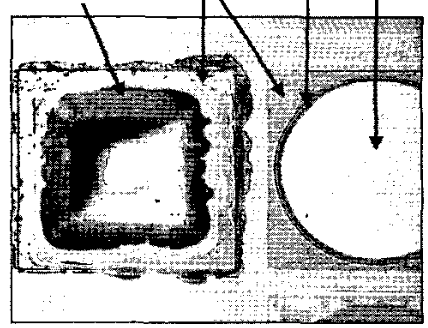


Figure 9: Island without/with surrounding trench after 1 hour in BHF with ultrasonic agitation

Figure (10) shows a sample after exposure to HF for 10 minutes. As can be seen, even though the parylene film is not delaminated, the small aluminum islands were etched away. This

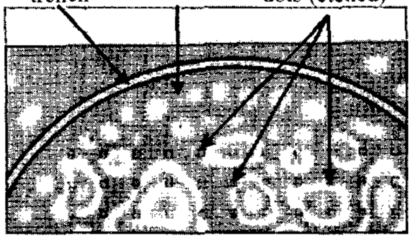


Figure 10: Sample after 10 minutes in 49% HF. The array of aluminum dots has been etched away

observation confirms that the HF can indeed penetrate the parylene. Figure (11) shows a sample with silicon dioxide islands surrounded by continuous trenches. Interestingly, despite previous observations, the surrounded oxide islands survive while the islands not surrounded are etched away. Measuring the oxide thickness on a cross-section shows that the oxide thickness is unchanged. Surface profiling can explain that phenomenon.

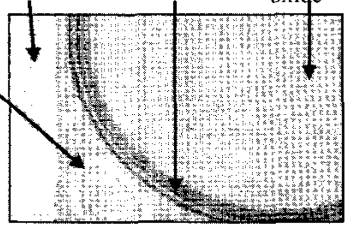


Figure 11: Covered oxide island after 10 minutes in 49%HF

Figure (12) shows the surface profile. The left part was an oxide island with no surrounding trench. As can be seen the oxide was completely etched away and the parylene film is stuck on the substrate. The right part shows an island after exposure to HF. The oxide is not etched away but the parylene film is

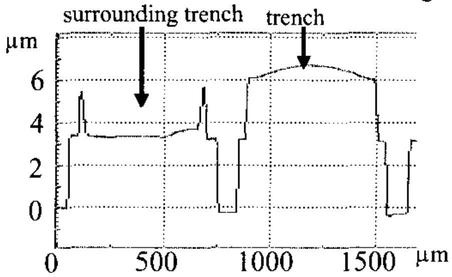


Figure 12: Surface profiling after 10 minutes in HF

bulging over the island. This suggests that the HF did in fact penetrate the parylene film and started to etch the oxide, producing gaseous byproducts that lifted the parylene layer, thus preventing further etching. This can be confirmed by heating up the sample, which leads to further bulging (over 20µm maximum deflection). This also suggests that the parylene film is indeed pin-hole free.

CONCLUSIONS

We demonstrated the efficiency of DRIE-etched trenches to improve dramatically the adhesion between parylene films and silicon substrates. Combined with an A-174 pre-deposition surface treatment, continuous trenches were shown to eliminate parylene delamination even when the samples are exposed to BHF for 24 hour or 49% HF for 2 minutes. We think this new technique can be of great value for parylene devices, virtually eliminating delamination problems during processing (e.g. when using acetone or BHF treatment) or during device operation (e.g. when high pressures are generated in channels or cavities). It has also been shown that HF can penetrate parylene films and attack the underlying parylene/silicon interface.

ACKNOWLEDGMENTS

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