Final Project Report

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I. Introduction

The objective of this short-term project is to fabricate miniature corrugated parylene diaphragms for earphones that are currently being developed by Adaptive Technologies under an Air Force-sponsored SBIR grant. The prototype diaphragms will be provided to ATI for integration into speakers.

II. Original Fabrication Process

Four masks were designed and made from Georgia Tech MiRC mask shop. A box of 25 prime grade 4" wafers was purchased from Nova Electronic Materials, Inc. The fabrication processes were originally determined and summarized in figure 1. It includes the following steps:

1) Grow 0.5um of thermal silicon dioxide, then spin thin S1813 photo resist to define mask layer for DRIE silicon etch process. Etch silicon dioxide in BOE.
2) Carry out Bosch Si etching to required channel depth (50-100um). Then grow 1.0um of wet thermal silicon dioxide, remove silicon dioxide in BOE, re-grow wet thermal silicon dioxide, remove again in BOE. Inspect and measure radius of curvature in SEM (additional oxidation steps may be required).
3) Coat with Al of 1µm thick as sacrificial layer for releasing of device later on. Then coat with parylene of desired thickness (2.5-5µm) as diaphragm material.
4) Spin coat planarization agent followed by spin SPR220 photo resist to pattern releasing grooves.
5) ICP etch parylene to define the releasing grooves.
6) Spin coat planarization agent followed by spin 15µm SU-8 resist to define center disks.
7) Spin coat planarization agent followed by spin 150µm SU-8 photo resist to define support rings.
8) Release completed membrane with Al etchant.
1. Resist pattering and RIE etch oxide as corrugation mask

2. Etch corrugations by DRIE and smooth wall and corner roughness by oxidation

3. Coat a sacrificial metal layer followed by coating Parylene

4. Define releasing groove mask with planarization and resist patterning
III. Preliminary Results

1. Si Corrugations (fabrication steps 1-2):

The fabrication was successful according to SEM inspection. The dimensions of the corrugation were as expected and the roughness of the walls as well as the geometry of the corners can be clearly seen in the SEM pictures.
2. Thick Metal Layer as Sacrificial Layer (fabrication step 3):

The deposition of Al metal of >1μm is challenging because the stress level builds up as the thickness increase. This is especially true with DC sputterer at high sputtering rate. As a result, the deposited film peeled off after taking the wafer out of the sputterer. We then
switched to electron beam evaporation with moderate evaporating rate and the result was
good as shown in the following picture. The metal thickness was 0.5μm in this case.

![Figure 3: A layer of Al was confromally deposited on Si mold](image)

3. Parylene Coating and Patterning (fabrication steps 4-5):

Parylene layer was deposited with a parylene coating system (model 2010 from Specialty
Coating System). The adhesion between the parylene layer and Al layer was good. However, the coating was not very conformal as indicated by the SEM inspection as well as the result of released diaphragms at a later stage.

![Figure 4: Defects in ill-conditioned parylene coating process](image)
Figure 5: A released diaphragm in which pieces of parylene segmentations were observed

We then prepared another wafer with corrugations and adjust the depositing parameters on the parylene coating system. The result turned out to be very conformal as expected.

To pattern the releasing grooves in the parylene layer with many corrugations on the wafer, we first planarized the wafer using a planarization agent from Futurrex Inc. Then a layer of SPR220 was spun on to define the releasing grooves. The etching of parylene was done in a RIE tool. The result was successful as indicted in microscope picture below, in which the circular shape of parylene pattern with corrugations is defined by the releasing groove.
4. Fabrication of Center Disk and Supporting Ring (fabrication step 6-7):

4.1 Experiment on compatibility between Parylene and thin SU-8 layer as center disks
   1) Grow 2µm thick parylene on wafer
   2) Spin coat 10µm SU-8 on top of the parylene
   3) Soft bake the resist at 95°C for 5min with slow ramp-up of the temperature from 25°C and slowly cool the wafer down to 25°C
   4) UV expose the resist at 160mJ/cm²
   5) Post exposure bake the resist at 95°C for 2min with slow ramp-up of the temperature from 25°C and slowly cool the wafer down to 25°C
   6) Develop the resist to define the center disk patterns
   7) Inspect the stress level of thin SU-8/parylene combination as well as the adhesion between them

As a result, the stress is manifested by micro cracks in the SU-8 pattern before the parylene film is released from the wafer. Further more, as we released the parylene later on (see section 4), the SU-8 center disk appeared in a bowl shape, which is another indication of big internal stress, though the adhesion between the SU8 disk and parylene seems to be strong enough to hold both layer together.

4.2 Experiment on compatibility between Parylene and thick SU-8 layer as support rings
   8) Spin coat 150µm SU-8 on top of a 4µm parylene covered wafer
9) Soft bake the resist at 95°C for 35min with slow ramp-up of the temperature from 25°C and slowly cool the wafer down to 25°C
10) UV expose the resist at 300mJ/cm²
11) Post exposure bake the resist at 95°C for 15min with slow ramp-up of the temperature from 25°C and slowly cool the wafer down to 25°C
12) Develop the resist to define the supporting rings
13) Inspect the stress level of thick SU-8/parylene combination as well as the adhesion between them

As a result, the stress is first manifested by micro cracks in the SU-8 pattern before the parylene film is released from the wafer. Secondly, as we released the parylene later on (see section 4), some SU-8 supporting rings curved and thus distorted the parylene from being flat. More dramatically, some SU-8 supporting rings were detached from the parylene membranes, suggesting the large internal stress of SU-8 deteriorated the adhesion between SU8 and parylene.

5. Releasing of Parylene by Metal Sacrificial Layer (fabrication step 8):

When the fabrication on a wafer was complete up to step 7, we immersed the wafer in Al etchant to release the diaphragms. Due to the significant stress level of SU-8 center disk and supporting rings, we avoided using ultrasonic means to increase the Al etch rate because it would detach the SU-8 structures from parylene diaphragms. However, we did try to use the magnetic stir bar to agitate the etchant while setting the wafer upside down inside the etchant solution. Therefore, it should take less time to release the diaphragm than it does from a static etchant solution. However, it still took more than 48 hours to completely release the diaphragms. In addition, the released diaphragms were curved in SU-8 center area and the SU-8 supporting rings were detached from most devices, as described before.

6. Conclusion and Lessons on the Preliminary Results:

The fabrication processes based on figure 1 was completely and carefully carried out. The preliminary results suggest two challenging issues in the originally determined processes.

First, the intrinsic stress of released SU-8 films as structure material is prohibitively high, particularly when the area of released SU-8 film is large and/or if the releasing agent slowly attack/react with SU-8, which introduces another level of stress to the SU-8 film. As a result, the released SU-8 film deforms dramatically and thus distorts the parylene film attached to it. Therefore, we conclude that SU-8 films as released from a substrate are not suitable as the structure material for this project.

Secondly, the releasing of diaphragm from the substrate based on metal sacrificial layer is very time consuming. This is particular true for this project because the diaphragms to be released have relatively large area. Therefore, the commonly practiced metal sacrificial layer in MEMS/IC fabrication seems to be impractical in this project.
The fabrication issues were brought to a meeting between both parties. The outcome of this meeting was the following major changes on the fabrication process. First, replace the Si mold by resist mold for corrugated membrane fabrication. Along this change, metal sacrificial layer is replaced by resist sacrificial layer as indicated in figure 2. This modification should reduce the releasing time significantly, which should yield higher outcome of the fabrication experiment, despite the fact that the mold will be one-time use compared to the multiple use of Si mold. Second, the fabrication of SU-8 supporting is eliminated because it is determined that, due to the relatively larger size of the diaphragm, commercially available O rings can satisfy the function of SU-8 support rings. Third, fabricate the center disk using metal layer instead of SU-8.

As a summary, the modified fabrication process is depicted as follows:

1. Spin-coat negative resist as sacrificial layer followed by flood UV exposure

2. Multilayer coating thick positive resist to define corrugations

3. Coat Parylene

4. Define releasing groove mask with planarization and resist patterning
5  Etch releasing groove

6  Define Metal center disk with planarization and lift-off

7  Release the diaphragm along with center disk

Figure 7: The modified fabrication steps for corrugated diaphragm

The fabrication process above includes the following details:
1) Spin 10μm NR-5 negative resist and expose it to UV to crosslink the resist.
2) Spin a layer of SPR220 on top of NR-5, cure the resist, then spin a second layer of SPR220 until it reach a thickness of 50μm before UV exposure to define the corrugations.
3) Coat the wafer with parylene of desired thickness (2.5-5μm) as diaphragm material.
4) Spin coat planarization agent followed by spin SPR220 photo resist to pattern releasing grooves.
5) ICP etch parylene to define the releasing grooves.
6) Spin coat planarization agent followed by spin SPR220 photo resist to pattern center disks. Use electron beam evaporation to deposit 1μm Al or Cr and then lift-off resist to define the metal center disks.
7) Release completed membrane with Acetone or resist remover along with ultrasonic agitation.
V. Final Results

1. Positive Resist Mold on Negative Resist Sacrificial Layer (fabrication steps 1-2):

A tone conversion of the corrugation mask was first completed to prepare the resist mold fabrication. The fabrication was successful according to SEM inspection. The dimensions of the corrugation were measured by non-destructive method using the Wyco Optical Profilometer. Both the height and the width of the mold show in Fig. 8 are about 50µm.

Figure 8: Corrugations formed in multilayered SPR resist
2. Parylene Coating and Patterning (fabrication steps 3-5):

Parylene layer was deposited with a parylene coating system (model 2010 from Specialty Coating System). The adhesion between the parylene layer and resist mold was good and the coating was conformal.

As before, we use planarization agent from Futurrex Inc. to pattern the releasing grooves in the parylene layer with corrugations on the wafer. However, since the corrugations stick out of the substrate plane in the current design, much thicker planarization agent than the original design is needed, which results in some difficulties in the removal of the planarization agent after the top layer resist is patterned for the releasing grooves. As a result, the etching of parylene to define the releasing grooves has only limited success within the time constraint imposed by the project.

3. Fabrication of Metal Center Disk (fabrication step 6):

We tried to deposit a layer of 1μm thick Al on top of a 4μm parylene layer using electron beam evaporation. However, the stress of the Al layer cracks the parylene layer dramatically, as shown in the microscope picture.

![Microscope Picture](image)

This result is totally different than our previous experiment where the Al layer was deposited before parylene coating. Then the stress levels of different Al thicknesses were tested and it turned out even 0.3μm of Al cracks the parylene. Of course, the stress of metal layers is tool specific. However, we are limited to electron beam evaporation tool due to the mask available and time...
constraint. Therefore, the idea of using thick metal layer as the center disk was discarded within this project.

4. Releasing of Parylene by Resist Sacrificial Layer (fabrication step 7):

When the fabrication on a wafer was complete up to step 6, we immersed the wafer in Acetone or negative resist remover to release the diaphragms. It took overnight to completely release the diaphragms from the substrate. However, resist residues may still attach to some membranes. Thus, ultrasonic agitation was needed to completely remove the resist and produce pure parylene diaphragms with corrugations. As a result, a picture was taken after the completion of all fabrication steps and shown below (Fig. 8 left). For comparison purpose, a diaphragm before releasing was also shown in Fig. 8 (right).

It is observed that the released diaphragms still have some curvature, particularly around the edge. And the curvature varies from wafer to wafer. This result suggests that the stress level of parylene is not negligible and it depends on the coating conditions of the tool. However, we didn’t have enough time to optimize the coating parameters.

5. Summary on the Final Results:

The fabrication processes based on figure 2 was carefully implemented. However, due to the time constraint imposed by the project, steps 4-6 were not thoroughly investigated to produce the best results. Only limited success was achieved. As a result, we could only deliver about 10 devices according to the modified fabrication processes.

In order to provide enough diaphragms to carry out the successive steps of this project, namely, assembling the diaphragms to speakers and testing the performance, we made two extra wafers up to step 3 of the modified fabrication processes. We then diced one of the wafers into squares and released the diaphragms in Acetone and ultrasonic bath. A total of 80 pure parylene diaphragms (i.e., without metal center disks) were collected and delivered. It is hoped that the speaker company can assemble the square diaphragms by means such as stamping them into circular shape if necessary. Another wafer is delivered as it is
fabricated up to step 3. We suggest that the wafer be CNC machined by a third party to define the releasing groove and carried out with the releasing step (step 7). That should produce a great number of devices out of this wafer. It is also hoped that they can test the performance of the corrugated diaphragms even if the metal center disks are missing.