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Aerogel & Porous Silicon MEMS for on-chip Vacuum Packaging

Extension to
“Aerogel MEMS for High Acceleration
and High Shock Applications”

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14. ABSTRACT The goal of this project is to develop a versatile on-chip vacuum-package technology for surface-micromachined devices. The resulting on-chip encapsulation technology is expected to provide a low-cost, high performance packaging solution for a wide variety of surfacemicromachined devices, especially micro- and nano-resonators. Our approach is to cover the MEMS structures with a nano-porous membrane before releasing. Release etching is performed by liquid- or gas-phase etching through the permeable membrane. Sealing is achieved by depositing a metal or dielectric film directly on the nano-porous shell in vacuum. Two types of nano-porous materials are developed: thin-film aerogels and porous polysilicon (PPS).					
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I. Project Goals: A Summary with Applications

The goal of this project is to develop a versatile on-chip vacuum-package technology for surface-micromachined devices. The resulting on-chip encapsulation technology is expected to provide a low-cost, high performance packaging solution for a wide variety of surface-micromachined devices, especially micro- and nano-resonators. Our approach is to cover the MEMS structures with a nano-porous membrane before releasing. Release etching is performed by liquid- or gas-phase etching through the permeable membrane. Sealing is achieved by depositing a metal or dielectric film directly on the nano-porous shell in vacuum. Two types of nano-porous materials are developed: thin-film aerogels and porous polysilicon (PPS).

Notable Accomplishments to Date:

- We have developed a wide range of aerogel and mesoporous thin films that are compatible with MEMS processing
- Aerogel microstructures, including suspended membrane, microbridge, and cantilever beams have been fabricated
- Mechanical properties of aerogel thin films have been successfully characterized
- Porous polysilicon was formed on dielectric layer by electrochemical etching for the first time
- Porous polysilicon windows was patterned by photography, allowing selective removal of sacrificial materials
- Fast releasing of sacrificial materials (e.g., PSG) through porous polysilicon was demonstrated for the first time

II. Detailed Progress Report

A. Aerogel Thin Film Materials Development

In this program, we have optimized mesoporous TiO_2 films and developed mesoporous thick (3-4 μm) ORMOSIL films. In order to optimize the mesoporosity of the TiO_2 films, we investigate the relationship between the amount of surfactant in coating solutions and the resulting porosity of mesoporous TiO_2 films. In the investigation of step coverage, mesoporous TiO_2 films exhibit a good step coverage when deposited on top of a sloped PSG layer with $\sim 2 \mu\text{m}$ height.

ORMOSIL (organically modified silicate) films are a "hybrid" material consisting of both organic and inorganic constituents that is designed to possess improved mechanical properties as compared to the inorganic (brittle) mesoporous oxides. The motivation for this work is that the organic modification to a silicate network will greatly increase the HF resistance and a mesoporous structure will allow for a sacrificial layer below to be etched by HF. Films with smooth surfaces and mechanical compliance for good step coverage have been prepared. A porosity of 15-20% has been achieved in first generation thin films. Work is underway to prepare mesostructured thick films.

A1. Development of Mesoporous TiO₂ Films

Fig. 1 shows how porosities of mesoporous TiO₂ films change with the amount of surfactant (Pluronic P65). The porosities of the mesoporous films vary from 30% to 45% with different amount of surfactant ranging from 0.05g to 0.5g. Increase of the porosity can lead to higher permeability while decreasing the porosities of the films results in higher mechanical strength. Finding out the optimized composition of the mesoporous TiO₂ films which possess not only proper permeability for HF to go through but also high resistance in HF solutions is one of our future works.

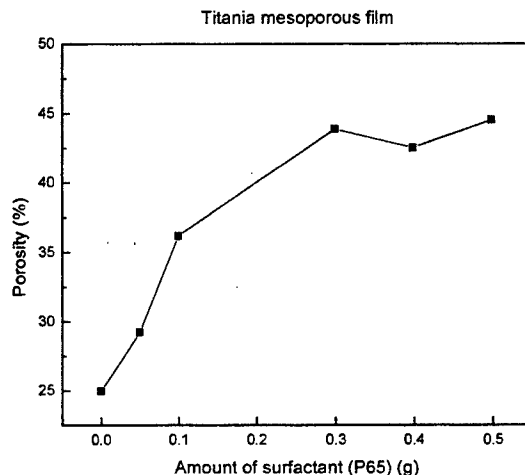


Fig. 1. The relationship between the porosity of mesoporous TiO₂ films and the amount of surfactant (P65) in the coating solutions

The HF resistance of the mesoporous TiO₂ films deposited on sapphire wafers were investigated before and the results demonstrated that TiO₂ films showed no apparent etching in buffered HF solutions for relatively long time (> 30 min). Based on these results, we continued to examine the etching resistance of the mesoporous TiO₂ films deposited on the Si wafers. However, all the films were completely delaminated when they were spin-coated directly on the silicon wafers. Titanium silicide, which is easily attacked by HF, may form between the mesoporous TiO₂ layer and Si wafer and cause the mesoporous TiO₂ layer to delaminate from the Si substrate. As an alternative approach, we introduced a low stress silicon nitride layer as a protecting layer. Although systematic investigation of the etching tests of the mesoporous TiO₂ films deposited on the silicon nitride wafers has not been done yet, it has been done and proved that these films can withstand in 7% BOE solution for 20 min without causing delamination. Furthermore, we have studied the step coverage of the mesoporous TiO₂ films on the sloped ~ 2 μm step of a PSG sacrificial layer (Fig. 2). The SEM picture indicates that the mesoporous TiO₂ film can perfectly attach to the PSG underlayer without any cracking after 600 °C heating. The next step is to develop membranes from these mesoporous films with a HF solution etching process.

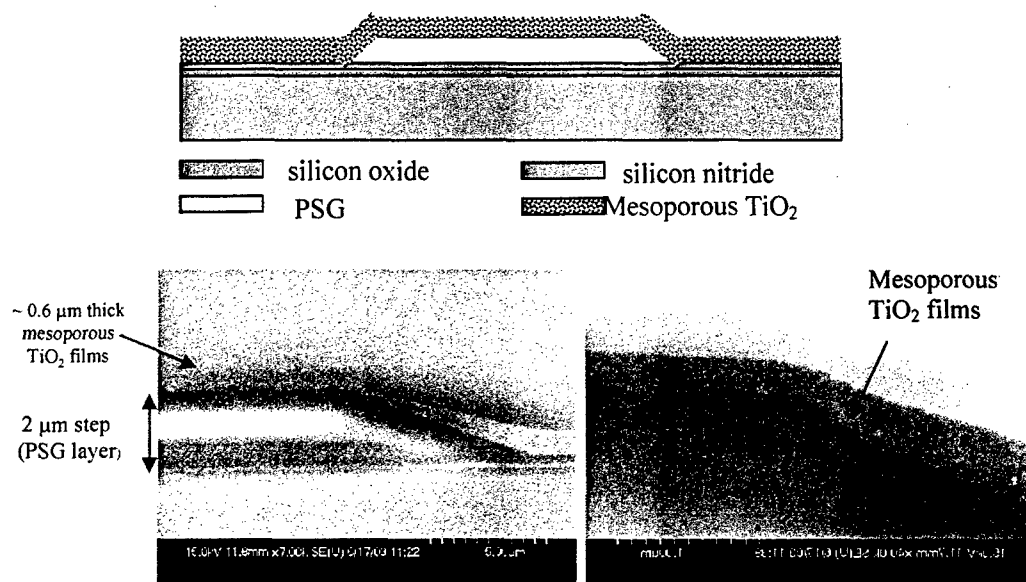


Fig. 2. SEM cross-section images of a mesoporous TiO₂ film on a sloped PSG layer

The crystallinity of mesoporous TiO₂ powders was also investigated. Rutile phase TiO₂ is desired since it is a thermally stable phase of TiO₂ and it probably has higher HF resistance than anatase phase. As reported previously, we have successfully synthesized pure anatase phase in both powder and film cases, and at this time, we synthesized rutile phase TiO₂ by simply decreasing the amount of surfactant (P65) from 0.5g to 0.1g. Fig. 3 displays the X-ray diffraction patterns of mesoporous TiO₂ powders calcined at different temperatures. An anatase phase was well developed when powders were heated to 400°C. With increasing calcination temperature, the anatase phase began to transform to a rutile phase, and at 600°C, only pure rutile phase was observed. Since pure rutile phase TiO₂ have been well developed in powders, our future approach will be synthesizing rutile phase mesoporous films.

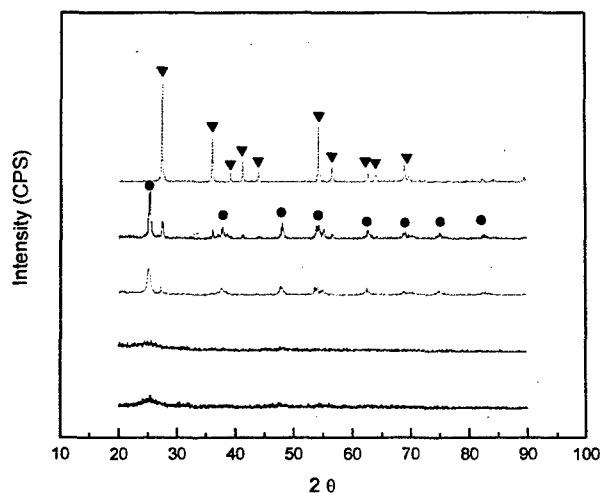


Fig. 3. X-ray diffraction patterns of mesoporous TiO₂ powders calcined at different temperatures

A2. Development of ORMOSIL Film Material

We have developed thick (2-4 μm) films that allow for good step (2 μm) coverage. Previously we worked with a methacrylate-modified silica system. Although this methacrylate-based ORMOSIL system produced 5 μm thick films with thermal stability up to 250°C on flat surfaces, porosity was very difficult to achieve. Therefore, we pursued other precursors. All films during this reporting period have been prepared by mixing tetramethoxy silane ($\text{Si}(\text{OCH}_3)_4$; TMOS) and organic trimethoxy silane ($\text{R-Si}(\text{OCH}_3)_3$) or a bridged silsesquioxane ($(\text{OCH}_3)_3\text{Si}-\text{R}-\text{Si}-(\text{OCH}_3)_3$), R denoting an organic group. In the bridged silsesquioxane, the organic modifier is covalently bonded to two Si-O_x branches rather than dangling as in the case of the methacrylate system. We have had the most success with the bridged silsesquioxane 1,2 bis trimethoxysilyl ethane ($(\text{OCH}_3)_3\text{Si}-\text{CH}_2\text{CH}_2-\text{Si}-(\text{OCH}_3)_3$; BTME). The improved network connectivity has helped to create a mesostructure and improve mechanical properties of the film. Sols for these films are synthesized with excess solvent to allow for a stable sol, the sol is aged for 2-3 days with subsequent solvent evaporation at 60°C. If solvent evaporation is slow and controlled the viscosity can be changed to fine-tune the resulting film thickness.

The major goal for step coverage is to prepare films with proper mechanical flexibility to cover a step without cracking or delaminating. The mechanical compliance is highly dependent on the type and amount of organic modification to the silicate matrix. Step heights of 2, 3, 4 and 5 μm patterned on a Si wafer were studied with 1:3 and 1:6 TMOS:BTME films. Initially, film attachment to the Si substrate was problematic. Post HF patterning of Si, film attachment was unfavorable (Fig. 4). After testing a few wet chemistry methods to terminate the Si surface with silanol (Si-OH) groups we concluded a fresh Piranha treatment is sufficient (Fig. 5). The best step coverage was obtained with a 3.5 μm step containing 3wt% (starting sol) of the anionic surfactant cetyltrimethylammonium bromide (CTAB, not removed) and dried at 120°C overnight (Fig. 6).

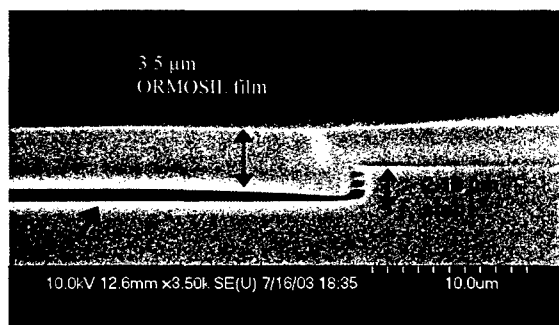


Fig. 4 SEM cross-section image of ORMOSIL film on a 2.18 μm step on post-HF Si substrate. Delamination throughout surface (arrow).

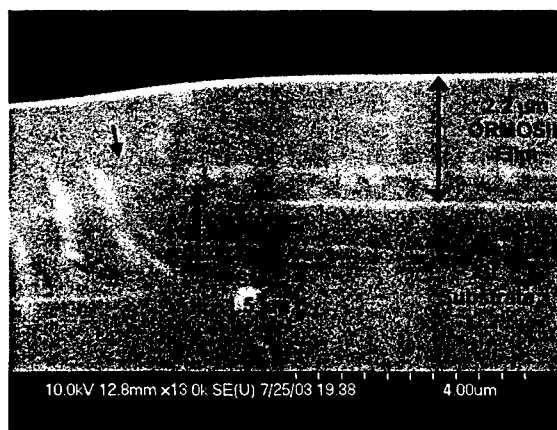


Fig. 5 SEM cross-section image of ORMOSIL on 1.5 μm step after Piranha treatment, good attachment. Defects were introduced during dicing for SEM sample preparation (arrow).

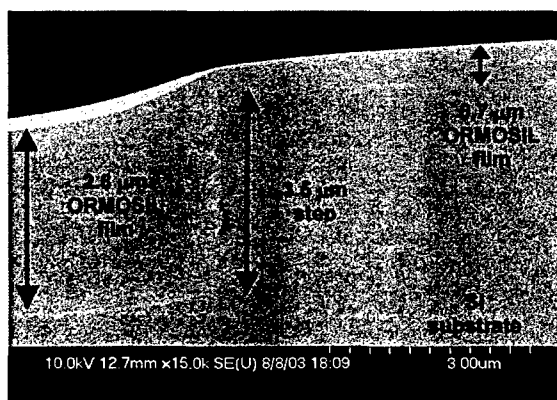


Fig. 6 SEM cross-section image of ORMOSIL film containing 3wt% (weight of initial sol) CTAB (not removed) covering a 3.5 μm step after substrate treatment with Piranha, dried at 120°C overnight.

A3.Development of Mesoporous Structure in ORMOSILs

Film composition and the nature of the inorganic-organic network is very important to allow for mesoporosity to be developed. Using a trimethoxy silane, where the organic group is terminal, decreases connectivity and compactness of the network. The more organic component the more difficult it is for the network to fully envelop around the surfactant mesostructure during the condensation and gelling stages. In contrast, a bridged precursor should not decrease connectivity and should not decrease compactness as much. Our efforts have focused on using a bis trimethoxysilyl ethane (BTME) based sol and the anionic surfactant cetyltrimethylammonium bromide (CTAB) to develop mesoporous materials. We have studied the following compositions:

TMOS	BTME	Methanol (anhydrous)	Water (0.04 HCl)	CTAB Wt % of initial sol
1	1	20	15	2 and 3
1	3	25	33	6, 12, and 18
1	6	52	59	

Table 1 Compositions (molar ratios) studied for the development of mesoporosity in ORMOSIL materials

The initial amount of solvent was kept constant and equal to total precursor volume. The relative amount of water was kept constant at 1.5 times that of theoretical hydrolysis for their respective TMOS:BTME ratio. The films were formed by spin coating, dried at 120°C overnight and heat treated at 275°C for 45 minutes. Films of 1:3 TMOS:BTME with 18 wt % CTAB were calculated to have a ~20% porosity, 1:6 TMOS:BTME with 6 and 12 wt % CTAB have 20% and 16% porosity, respectively. Porosity was calculated by relating the index of refraction to the skeletal density from ellipsometry measurements. Our films were compared to dense silica and assumed to be a non-absorbing, non-scattering, homogenous film.

We have begun thermogravimetric (TGA) studies of CTAB. This analysis provided the burn out profile of the surfactant to be within 175-300°C when the temperature is increased at 1°C/minute. However, if the temperature is stepped by 10°C and held for 20 minutes, most (90 %) of the surfactant is burned by 225°C. This finding has helped to better understand how to optimize the surfactant removal from the ORMOSIL matrix. We are currently performing TGA and FTIR measurements on 1:3 and 1:6 TMOS:BTME samples to investigate whether the chemical nature of the matrix, specifically the Si-C bonds, is intact after heat treatments. We are also currently working on BET surface area measurements of powder samples, and on increasing the thermo-mechanical stability of the films in order to be able to perform the heat treatments on thick samples.

B. Development of Porous Polycrystalline Silicon (PPS) Membrane

B1. Vacuum sealing test of PPS cavity

Previously we proposed porous polycrystalline silicon (PPS) as potentially more practical permeable shell for sealed cavity applications. PPS has the following several advantages over aerogel: (1) Electrochemical etching can produce relatively thick thus stronger PPS shell layer; (2) PPS has perfect step coverage; (3) it is fully compatible with surface micromachined devices.

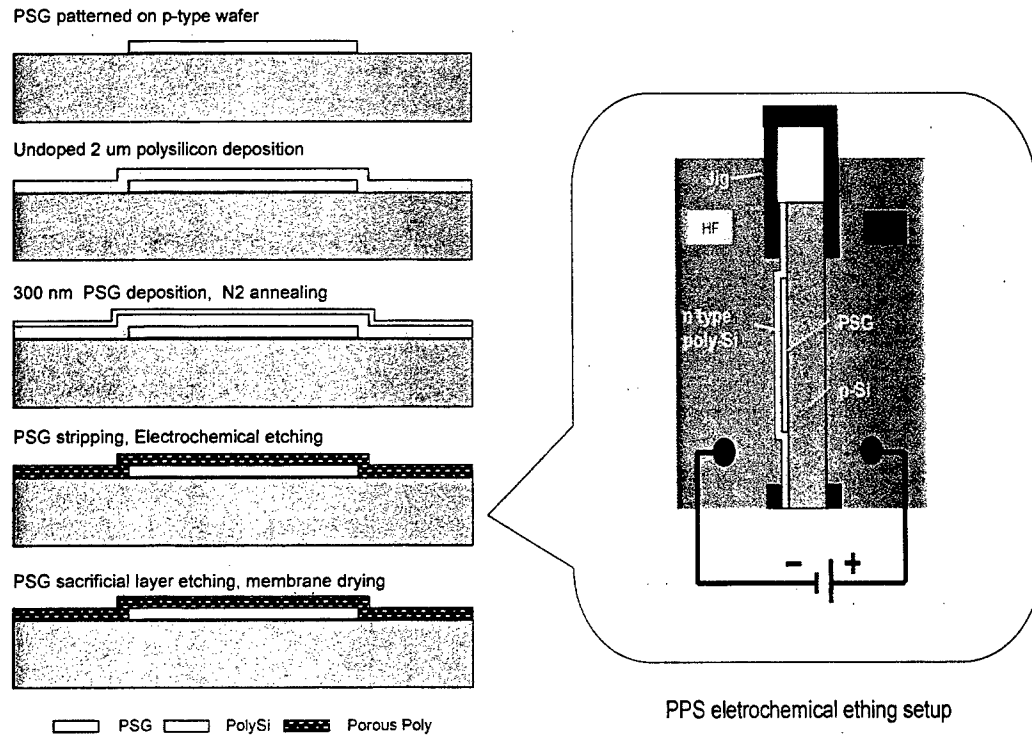


Fig. 7 Process flow of PPS membrane

The process flow to form PPS membrane is shown in Fig. 7. We reported before cracks and wrinkles are observed at most of the PPS membranes due to high stress generated in electrochemical etching and the stress can be released by annealing at 700 °C in N₂ for 5 mins. Fig. 8 is a stress free PPS membrane. The PPS membrane survived without any breakage in high vacuum (1×10^{-6} torr) and high temperature (750 °C), which are desired vacuum sealing conditions.

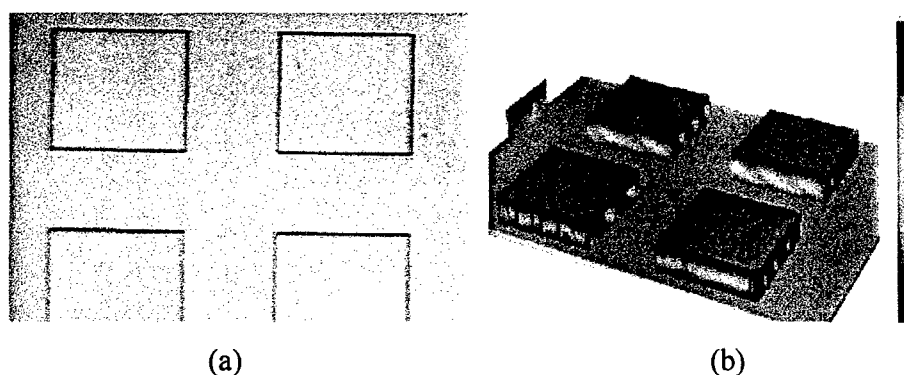


Fig. 8. Released PPS membrane. Membrane size is 600 μm . (a) is the microscope top view. (b) is the Wyko 3D image.

In the vacuum sealing experiment, 1.2 μm LPCVD low temperature oxide (LTO) was deposited to seal the PPS cavity. The deposition temperature was 450 $^{\circ}\text{C}$ and pressure was 240 mtorr. If the cavity is successfully vacuum sealed and there is no stress in the film, the cavity will bend down due to the pressure difference (Fig. 9).

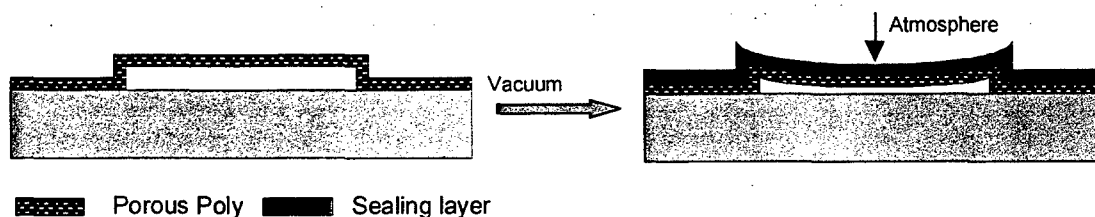
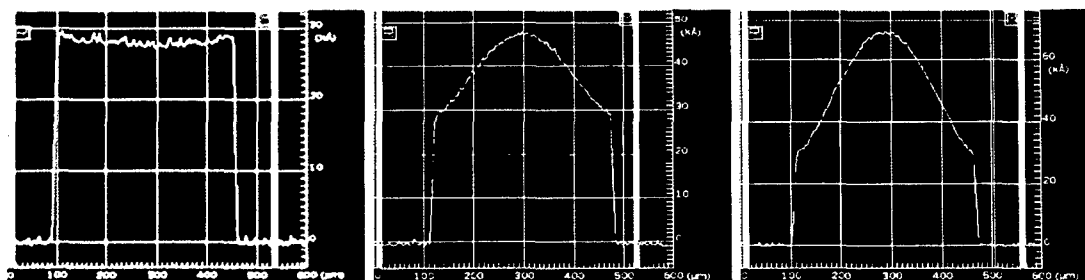


Fig. 9. Released PPS membrane. Membrane size is 600 μm . (a) is microscope top view. (b) is Wyko 3D image.

After LTO deposition, the cavity was found to bulge up gradually. The scanning profile of the cavity measured by Dektak changed with time as shown in Fig. 10. For comparison, another sample was stored in vacuum right after LTO deposition to ensure no air leakage (Fig. 11). There was no bulging up observed in this sample when it was stored in vacuum. But the gradual bulging up was observed after this sample was taken out of the vacuum environment. This confirmed that the PPS cavity was vacuum sealed right after LTO deposition. But air leakage caused the membrane to bulge up. Since there is high stress in LTO film, the membrane will bulge up instead of staying flat when the pressure inside and outside are the same. Suspecting air leakage is probably caused by pin-holes in LTO, we will deposit denser films such as low stress nitride to seal the cavity.



(a) (b) (c)

Fig. 10. Dektak scanning profiles of PPS cavity after LTO sealing. The sample is stored in ambient. (a): right after deposition; (b): 4 hours after deposition; (c): 22 hours after deposition.

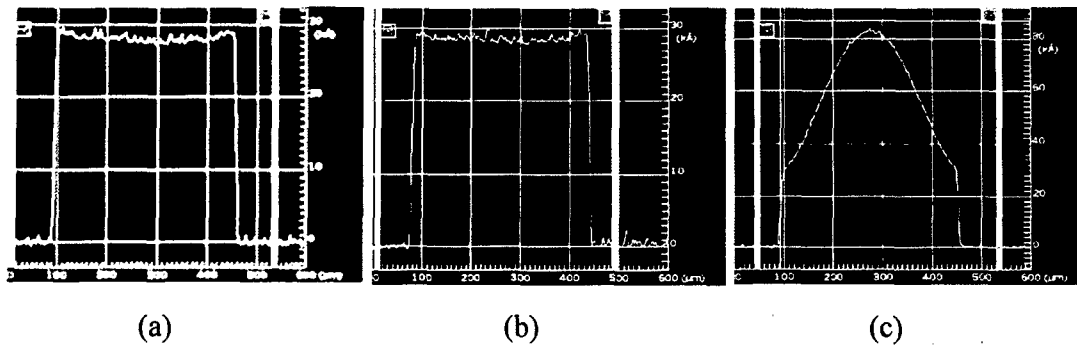


Fig. 11. Dektak scanning profiles of PPS cavity after LTO sealing. The sample is stored in vacuum. (a): right after deposition; (b): 22 hours after deposition; (c): the sample is taken out of the vacuum chamber.

B2. Porous polycrystalline silicon (PPS) formation on silicon nitride

Most surface micromachining device is fabricated on silicon nitride. To be compatible with typical surface micromachining process, PPS should be able to form on silicon nitride. In order to create electrical contact between polysilicon and silicon substrate for electrochemical etching, openings through nitride layer are made. This becomes an issue during electrochemical etching. The large current density at the electrical contact area leads to electro-polishing of silicon and electrical contact between polysilicon and silicon is lost (Fig. 12). Thus, the polysilicon layer can not be turned porous all the way and the sacrificial PSG layer underneath can not be etched away.

To solve this problem, photoresist is used to define the electrochemical starting area in polysilicon as shown in Fig. 13. The photoresist can prevent HF from entering the contact area and solves the problem of electro-polishing. Another important finding is that large current should be applied at the beginning of the electrochemical etching since polysilicon of large area is exposed, then small current should be applied to prevent electro-polishing in the polysilicon protected by photoresist. SEM pictures in Fig. 13 show the electrochemical etching result. In the left SEM picture, the gap between PSG and polysilicon indicates PSG underneath is starting to be etched after the top polysilicon is porous. The cross section of the PPS membrane is shown in right SEM picture after the underlying PSG was removed by HF, which diffuses through the porous polysilicon.

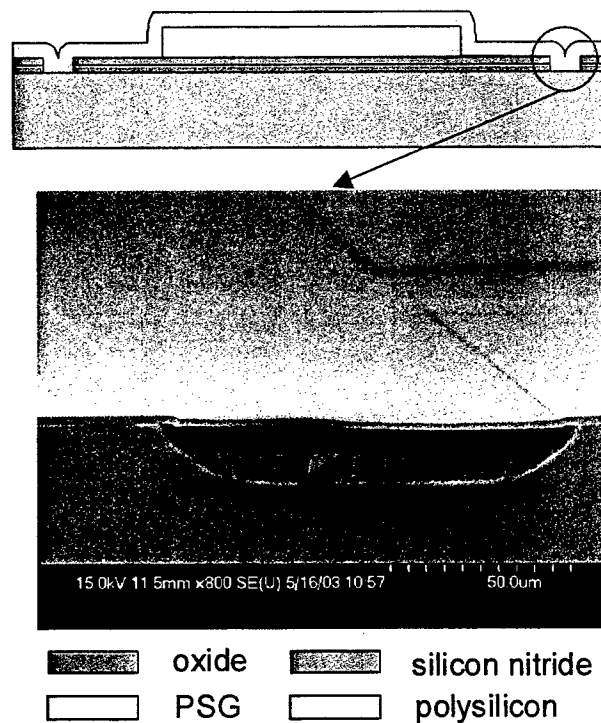


Fig. 12. Electro-polishing of silicon in the contact area during electrochemical etching.

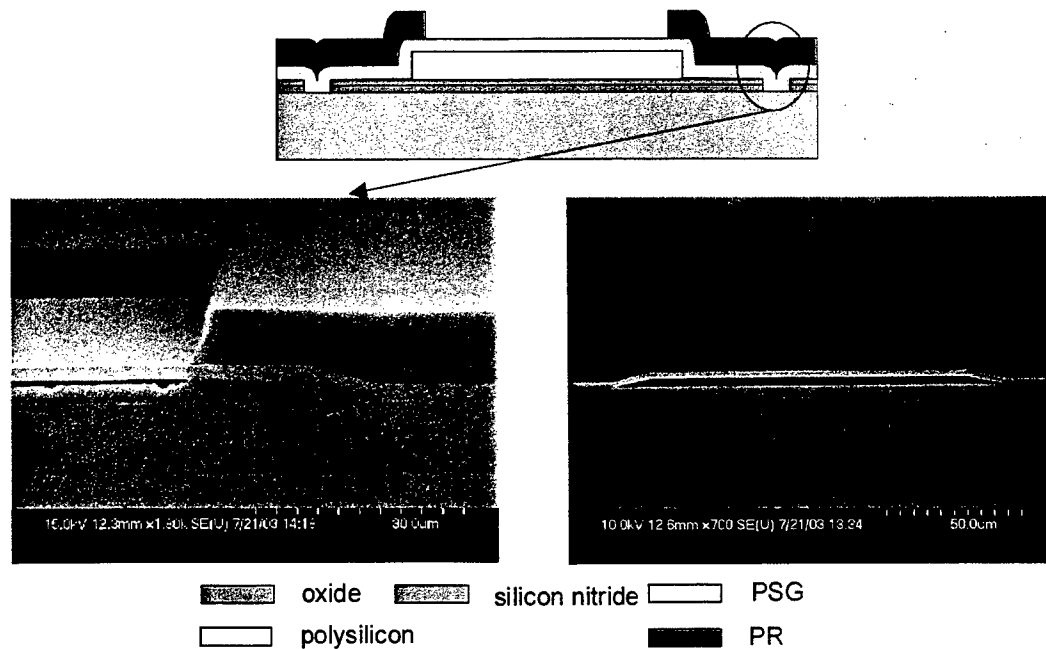


Fig. 13. PPS cavity successfully formed on silicon nitride.

A conclusive test of the vacuum sealing is demonstrated by a “dual-cavity” design shown in Fig. 14. Two cavities are joined by a connecting pipe and then sealed by the depositing a low-stress silicon nitride film deposited by plasma-enhanced chemical vapor deposition. Since the

silicon nitride is deposited at low pressure, the surface profile of the cavity showed a concave shape, as shown by the profile in the left-hand side of Fig. 14. There are two possible causes of the concave shape: (1) the cavity is indeed sealed by the silicon nitride, as we hope to achieve; or (2) the surface bulkled due to the residue stress of the PPS or silicon nitride film. To conclusively determine the mechanisms, we broke the cavity on the right-hand side intentionally using a probe tip. If the concave shape resulted from vacuum sealing, the the cavity should restore to flat-top shape since the vacuum is broken now. The surface profile scan on the right-hand side of Fig. 14 showed that the cavity top became much flatter than the before, and the height difference is about $1.6\text{ }\mu\text{m}$ compared with before breaking vacuum. This test has conclusively demonstrated successful vacuum sealing of the PPS cavity by silicon nitride.

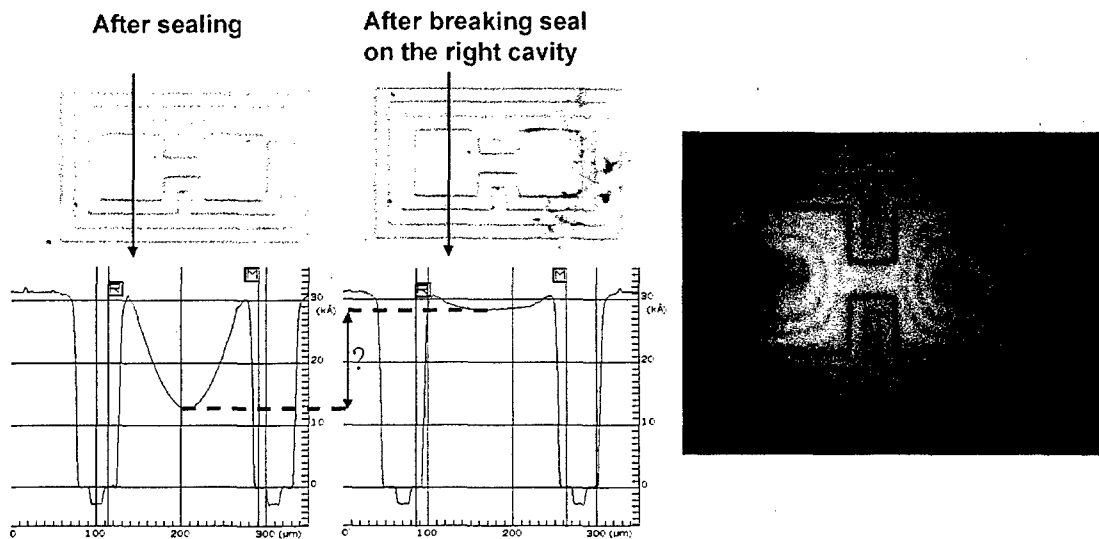


Fig. 14. PPS cavity successfully formed on silicon nitride.

B3. Fabrication of micro sealed cavity with resonators inside

After addressing the major issues of using PPS as permeable shell for sealed cavity application, we have started a fabrication process of micro sealed cavity with resonators inside. The resonators inside will indicate the vacuum level of the sealed cavity. MUMPS is chosen to fabricate those resonators because MUMPS service is widely used for typical surface micromachining application (Fig. 15(a)). A $4\text{--}6\text{ }\mu\text{m}$ PSG layer will be deposited and patterned on MUMPS chip to create enough deflection space after the cavity is vacuum sealed (Fig. 15(b)). Followed is $2\text{ }\mu\text{m}$ undoped polysilicon deposition. To get symmetric doping in the polysilicon, 300 nm PSG was then deposited on top and annealed at $950\text{ }^{\circ}\text{C}$ in N_2 (Fig. 15(c)). After the top PSG layer was stripped off, some part of the polysilicon can be selectively turned porous by electrochemical etching in $1:1\text{ HF:ethanol}$ (Fig. 15(d)). Then, the underlying PSG was removed by HF, which diffuses through the porous polysilicon (Fig. 15(e)). After the structure is dried in supercritical CO_2 dryer, the sealing material will be deposited. The sealing method can either be LPCVD deposition which has conformal coating or metal evaporation which has very high

vacuum. The evaporated metal only needs to cover the top surface of the cavity, which is partly turned porous by selectively electrochemical etching (Fig. 15(f)). Finally, the contact pads are opened for electrical contact (Fig. 15(g)). Figure 16 shows the SEM image of a released microbridge covered by a porous polysilicon (PPS) shell. The microbridge has been released through the PPS.

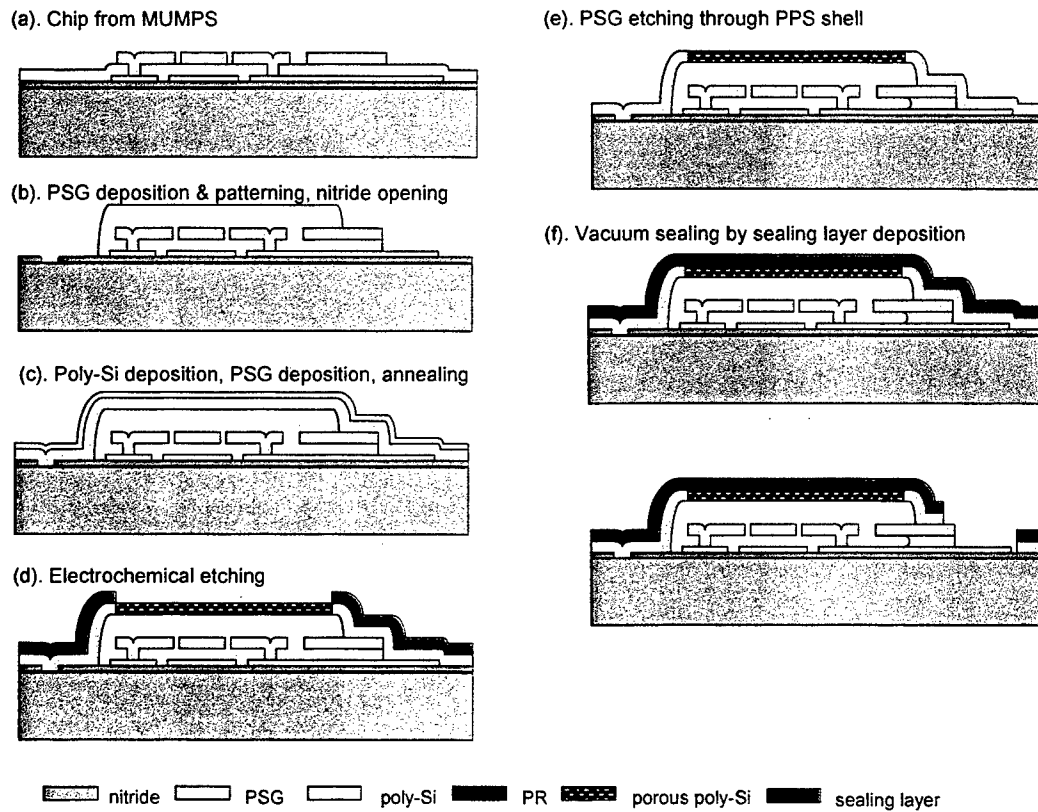


Fig. 15. Micro sealed cavity process flow. PPS is used as permeable shell.

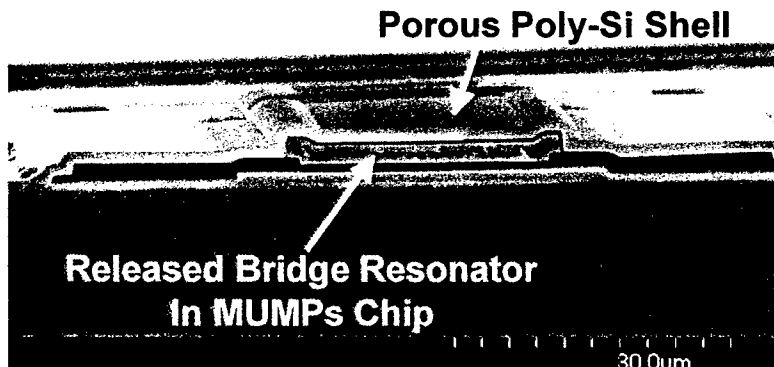


Fig. 16. SEM of released microbridge covered by a porous polysilicon (PPS) shell. The microbridge has been released through the PPS.

III. Publication and Presentation

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