MEMS WITH THIN-FILM AEROGEL

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ABSTRACT

This paper reports the first successful incorporation of aerogel thin films for MEMS fabrication. We developed the photolithography procedures and various surface micromachining methods for highly porous thin films. The development is verified with micro bridges, cantilevers, and membranes made of aerogel thin films, completing the entire procedures for two aerogel thin films, silica and alumina. Also discussed will be the new opportunities that aerogel MEMS bring about.

INTRODUCTION

Aerogels are highly porous nanostructured materials typically prepared by sol-gel processing. Due to their unique characteristics: lightweight, high relative surface area, low dielectric constant, and low thermal conductivity, aerogels have numerous applications [1] in various areas including aerospace [2]. The use of aerogel for MEMS, however, has never been reported until today. The goal of our research is to introduce aerogel as a new class of thin film material for MEMS fabrication. For example, when an aerogel thin film is used, the mass of a micromirror can be reduced by 70-90%, increasing the resonant frequency and shock resistance, while preserving the optical surface area. A related idea has been verified with honeycomb patterned micromirrors [3]. For another example, the exceptional insulating ability of aerogel films is highly desirable for micro thermal devices, which typically suffer from high thermal loss as a consequence of being small. In any case, it is essential to develop micromachining procedure of aerogel thin films compatible with other typical MEMS fabrication, both in the procedures and the use of equipment.

Dealing with porous thin films gives rise to some new issues. First, during the photolithography process, would photoresist (PR) be trapped in the nanopores of the aerogel thin film and cause serious problems? Second, when patterning aerogel thin films, would the etch rate increase significantly because of high surface-volume ratio? Third, would gas or low molecular weight liquid etchants penetrate through this porous thin film and attack the underlying layer? This paper describes an empirical study that investigates all of these questions. The entire procedures for two aerogel thin films, silica (SiO$_2$) and alumina (Al$_2$O$_3$), are reported: aerogel preparation, photolithography, etching, and structure releasing.

EXPERIMENTAL PROCEDURE

Aerogel preparation

Mesoporous silica and alumina aerogel thin films are deposited by spin coating. Silica coating solution is prepared with TEOS (tetraethylorthosilicate), H$_2$O, HCl and Ethanol. Alumina coating solution is prepared with ASB (Aluminun-Sec-Butoxide), H$_2$O, HNO$_3$ and anhydrous ethanol. The surfactant material, amphiphilic tri black copolymer (Pluronic F127 from BASF : (EO)$_{106}$(PO)$_{70}$(EO)$_{106}$), is dissolved in the prepared solutions and forms templates in the oxide matrix of mesoporous materials. The porosity of the material and the pore structure can be controlled by the concentration of copolymer. After spin coating, silica and alumina films are baked at 100°C for 12 hours. The furnace temperature is ramped slowly up to the heat treatment temperature of 400°C and 500°C for silica and alumina, respectively. The heat treatment is performed to burn out the copolymer; the remaining porous skeleton forms the aerogel thin film. The thickness of a single layer is 500 nm for silica aerogel and 300 nm for alumina aerogel, and multi-layer coating has been used successfully to increase film thickness. Some physical properties are listed in Table 1. TEM micrographs have confirmed that the pore size is less than 10 nm and that the pores are uniformly arrayed (Fig. 1). The surface roughness of aerogel thin film is less than 5 nm, suitable for optical application. Metal coating on the aerogel films has been proven successful in increasing the reflectivity [4].

Table 1. Physical properties of aerogel films

	Silica aerogel	Alumina aerogel
Pore Volume	1.16 cm³/g	0.80 cm³/g
Average Pore Diameter	72 Å	61.8 Å
Porosity	72 %	70.8 %
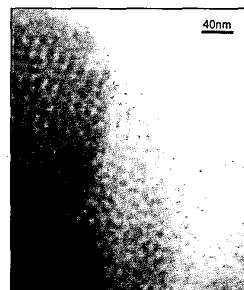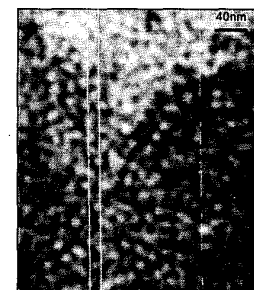
| (a) Silica aerogel | (b) Alumina aerogel |

Figure 1. TEM photographs of aerogels

Photolithography

Lithography on aerogel thin films poses new challenges since, among others, PR fills the nanopores of the aerogel thin films during PR coating. Because PR in the nanopores lacks the UV radiation during exposure, positive PR in aerogel layer remains

even after development and deposited as debris when the aerogel film is selectively removed. Fig. 2 shows the PR debris after BOE etching of silica aerogel thin film.
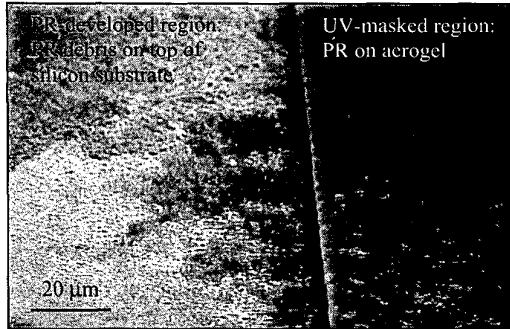

Figure 2. PR debris after aerogel removal

Two lithography processes were found successful for the silica aerogel thin film: removing the trapped PR before (Fig. 3(a)) or after (Fig. 3(b)) patterning the aerogel layer. Tested were 0.5-2.0 μm thick aerogel films. In Fig. 3(a), it was confirmed that the PR trapped in the aerogel film can be removed in O_2 plasma before the aerogel is patterned. In Fig. 3(b), it was confirmed that the trapped PR, deposited on the surface as debris when the aerogel film is etched away, can be removed by PR stripper. Although it has not been verified yet, a reasonable alternative is using negative PR to ensure that no resist remains trapped in the nanopores after development. Unlike the positive resist case, all unexposed resist will dissolve in the developer.
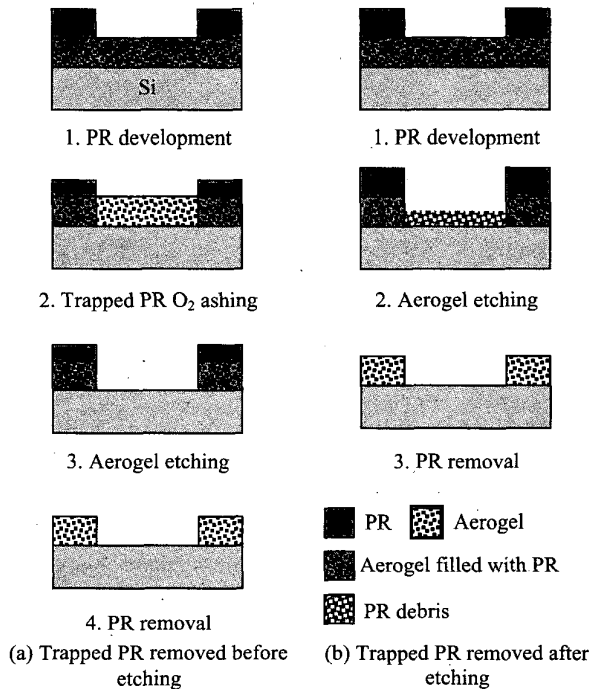

1. PR development   1. PR development

2. Trapped PR O_2 ashing   2. Aerogel etching

3. Aerogel etching   3. PR removal

■ PR   ▦ Aerogel

▦ Aerogel filled with PR

▦ PR debris

4. PR removal

(a) Trapped PR removed before etching   (b) Trapped PR removed after etching

Figure 3. Patterning of silica aerogel

The lithography process for alumina aerogel thin films is quite different, as shown in Fig. 4, since alumina is slightly soluble in some developers (e.g., AZ 400K, consists of potassium borates and water). The alumina aerogel, comprised of a network with solid phase dimensions of only 5-10 nm, is dissolved in developers. As a result, alumina aerogel thin film can be patterned during the PR development step. The subsequent step of etching the thin film is not necessary. However, a developer that does not attack alumina, (e.g., AZ developer, consists of phosphoric acid, trisodium salt, sodium silicate, and water) will pattern only the PR but not alumina aerogel. This is used, for instance, when a lift-off process is required to pattern metal on top of the alumina aerogel thin film. For non-aerogel-etching developers, the lithography process flow is identical to that of silica aerogel; PR trapped in alumina aerogel nanopores can be removed either before or after aerogel patterning. The choice of developer properties allows more flexibility in the lithography process.
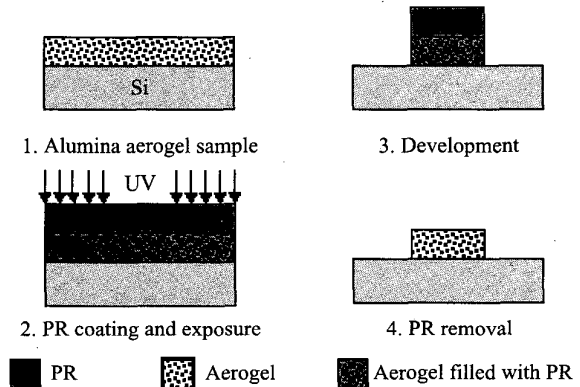

1. Alumina aerogel sample   3. Development

2. PR coating and exposure   4. PR removal

■ PR   ▦ Aerogel   ▦ Aerogel filled with PR

Figure 4. Patterning of alumina aerogel

Etching

Table 2 lists the etch time of various etchants for 1 μm-thick silica and alumina aerogels thin films. Since the aerogels have high surface area and thin network of solid, these porous films etch much faster than non-porous ones. Because high etch selectivity and rate are desirable for surface micromachining processes, using aerogel as a sacrificial layer facilitates the release of large area structures. For example, BOE etch rate of silica aerogel is 35 times faster than that of PSG, which is typically used as a sacrificial layer material in the MUMPs process. In other words, the etch selectivity would increase 35 times by using silica aerogel instead of PSG as sacrificial layer material.

Using alumina aerogel as a sacrificial layer and a high etch selectivity etchant, freestanding silica aerogel structures becomes possible. In this case, both the sacrificial and structural layers are made of aerogels. An "aerogel surface micromachining", involving only aerogels in processing, can be fulfilled by this idea.

To use aerogel as a structure material with a non-porous sacrificial layer, higher etch selectivity is necessary. In the following section, we will demonstrate how to obtain freestanding alumina aerogel structures by employing silicon as a sacrificial layer. As indicated in Table 2, XeF$_2$ dry etching is the best

candidate, because XeF_2 etching has high etch selectivity between silicon and alumina aerogel and is isotropic and thus undercuts the silicon, which helps to release the structure. However, RIE of silicon has also proven to be a successful etching method to release alumina aerogel structures, even though RIE etching is not as isotropic, i.e., does not have as much undercut. The details will be discussed in the next section. .

Table 2. *Etch time for 1 μm silica and alumina aerogel films in various etchants*

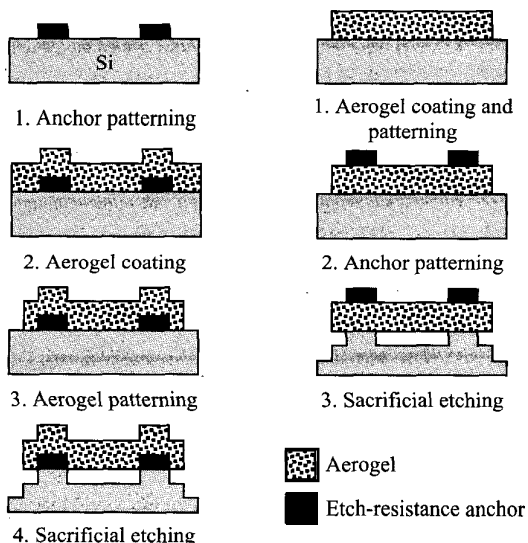| Etchants	1 μm silica aerogel	1 μm alumina aerogel
49% HF	< 1 sec	< 5 sec
BOE[a]	5 sec	1 min
Piranha[b]	Non-etched	Etched
30 wt% KOH	< 5 sec	< 5 sec
AZ 400K[c]	> 10 min	~1 min
XeF_2	< 5 pulses	No apparent etching (> 120 pulses)
RIE[d]	~2 min	No apparent etching (> 30 min)
DRIE	~3 min	> 10 min ·

[a] 40% Ammonium : 49% HF = 1 : 6
[b] Sulfuric Acid : Hydrogen Peroxide = 5 : 1
[c] DI water.: AZ 400K = 5 : 1
[d] Pressure ratio of reactant gases: ($CF_4 : O_2 = 5 : 1$), Power: (200 W)

Structure releasing

1. Anchor patterning

2. Aerogel coating

3. Aerogel patterning

4. Sacrificial etching

(a) Anchor underneath aerogel

1. Aerogel coating and patterning

2. Anchor patterning

3. Sacrificial etching

▓ Aerogel

■ Etch-resistance anchor

(b) Anchor on top of aerogel

Figure 5. *Anchoring schemes for sacrificial process*

With the ability to perform lithography in hand, the processes to micromachine various aerogel microstructures have been developed. The most notable aspect is that etchants can penetrate through the aerogel layer and attack the layer underneath. New concepts of sacrificial etching are necessary to obtain anchored structures. The anchors can be defined either by placing blocks of etch-resistance thin film underneath (Fig. 5(a)) or on top of (Fig. 5(b)) the aerogel layer. Although the silicon substrate is depicted as the sacrificial layer in Fig. 5, a discrete layer can be used as the sacrificial layer as well, if it is important to control the gap accurately. Based on these anchoring schemes, bridges, cantilevers, and membranes have been successfully constructed.

RESULTS AND DISCUSSION

Aerogel micro bridges and cantilevers

As shown in Fig. 6, an array of micro bridges made of 0.8 μm-thick alumina aerogel is released by XeF_2 dry etching of the silicon substrate. Aluminum (Al) is chosen as the anchor material because of its etch-resistance in XeF_2 and its adhesion to the alumina aerogel. With this process we have successfully released cantilevers up to 1100 μm-long with a gap ~5 μm, as shown in Fig. 7. Despite the slight bow at the tip, relatively flat cantilevers of this length and thickness indicate a very low stress gradient in the film; this is surprising for a spin-coated material. The mechanical properties of the aerogel thin films are currently under investigation.
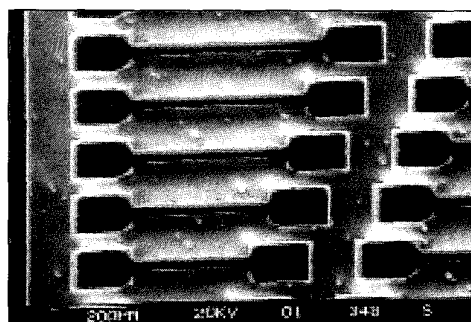

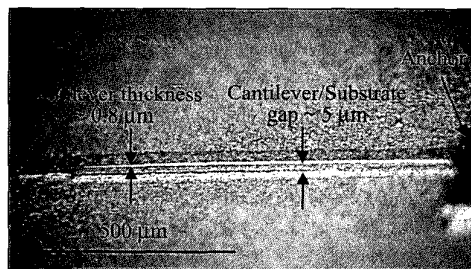
Figure 6. *Alumina aerogel micro bridges*

**Figure 7.** *Alumina aerogel cantilever*

Because of the etchant permeability of aerogel, the nanopores in the aerogel films act as natural etching holes. Removing the sacrificial layer underneath a large aerogel plate is not an issue, unlike the usual non-porous materials where the sacrificial layer must be removed by undercut etching and is therefore limited by diffusion. Now, even RIE can release aerogel structures, since undercut from the edge of the structure is not required. Moreover,

extremely small gaps between released structure and substrate can be achieved by this method. Theoretically, the gap can be as small as half of the aerogel solid network wall width (nm's).
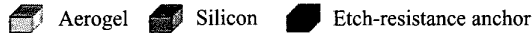
Aerogel membrane
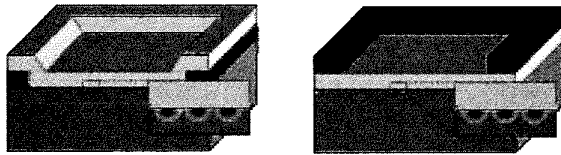
1. Etchant (e.g. XeF$_2$) penetrates through aerogel and starts to attack sacrificial material (e.g. Si).
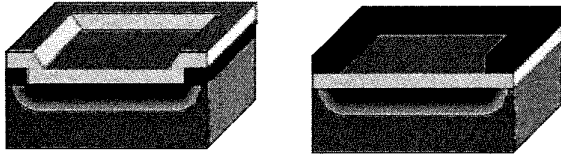
2. Aerogel membrane obtaining

⬜ Aerogel ⬛ Silicon ⬛ Etch-resistance anchor

(a) Anchor underneath aerogel (b) Anchor on top of aerogel
Figure 8. Aerogel membrane fabrication

(a)

Etching anchor (Al) Alumina aerogel membrane

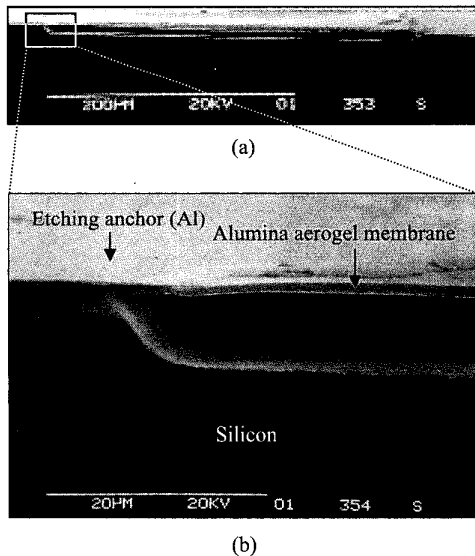Silicon

20μM 20KV 01 354 S

(b)
Figure 9. Alumina aerogel membrane

Fig. 8 shows the fabrication process of aerogel membranes. Tested membranes sized from 100 μm × 100 μm to 1000 μm × 1000 μm were all successfully released. Etching through nanopores and subsequent vacuum sealing have been reported for up to 190 nm-thick polysilicon [5]. With the tremendous pore volume in aerogel, much thicker films can be used for the same purpose. Since the structural strength is proportional to the third power of the film thickness, thick shells of aerogel eliminate the

need for reinforcement, a limitation of the thinner polysilicon structures.

An aerogel membrane is also demonstrated, as shown in Fig. 9. The ability to make cavities with such ease is especially attractive, as it is related to a wide range of applications, such as on-chip vacuum sealing for high-Q devices and liquid/gas filter.

CONCLUSION

This study investigated the MEMS process for new MEMS materials, aerogel thin films. Proposed lithography procedures resolved the issue of PR being trapped in the aerogel nanopores. The etch rates of aerogel thin films for various etchants were compared. Etchant permeability of aerogel thin films has been proven. Releasing large area aerogel structures with extremely narrow sacrificial layer is feasible. Fabrication of aerogel micro bridges, cantilevers, and membranes have been demonstrated.

ACKNOWLEDGEMENTS

REFERENCES

1. L. W. Hrubesh, "Aerogel Applications", *J. Non-Crystalline Solids*, Vol. 225, 1998, pp. 335-342.

2. http://aerogel.msfc.nasa.gov/

3. P. R. Patterson, G.-D. J. Su, H.Toshiyoshi, and M.C. Wu, "A MEMS 2-D Scanner with Bonded Single-Crystalline Honeycomb Micromirror", *Late News Dig., Solid-State Sensor and Actuator Workshop*, Hilton Head Island, SC, June 2000, pp. 17-18.

4. J.-A. Paik, B. Dunn, N. Kitazawa, S.-K. Fan, C.-J. Kim, M. C. Wu, "Preparation of Mesoporous Oxides for MEMS Structure," *2000 MRS (Materials Research Society) Fall Meeting Proceedings, EE: Materials Science of Micromechanical Systems (MEMS) Devices III*, Boston, Nov. 2000.

5. Lebouitz, K.S.; Mazaheri, A.; Howe, R.T.; Pisano, A.P., "Vacuum encapsulation of resonant devices using permeable polysilicon," *MEMS99*, pp. 470-475.