

# ETCH STOP MATERIALS FOR RELEASE BY VAPOR HF ETCHING

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*The integrity of various materials upon exposure to vapor HF etching was investigated. Whereas silicon nitride did not withstand the vapor HF, it was found that  $Al_2O_3$  and  $AlF_3$  are fully inert, thus both are suitable etch stop materials. In addition, the reflectance of Al in the ultraviolet range was measured, and found to be unaltered by the etchant. The thickness of AlSiCu also remained the same. All materials are suitable for use in CMOS MEMS microsystems.*

**Key Words:** etch stop, vapor HF, sacrificial etching, release

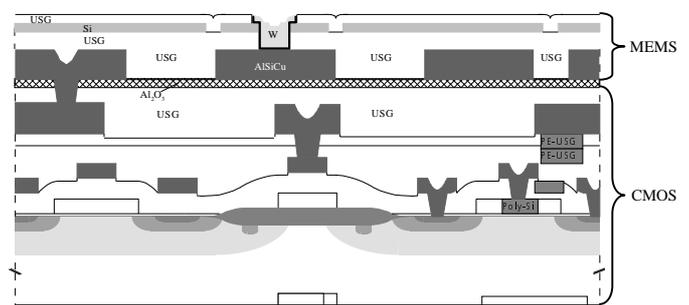
## I INTRODUCTION

Silicon dioxide is a commonly used inorganic sacrificial material in surface micromachining. An inorganic material such as silicon dioxide provides significant advantages over its organic counterparts in that it is thermally stable, can be machined to a high degree of accuracy, and is standard in a CMOS fabrication environment. Various methods for the release of MEMS devices exist, such as wet etching by hydrofluoric acid (HF) followed by supercritical drying, sublimation drying, or the use of self-assembled monolayers (SAM) that make the surface hydrophobic and thereby prevent stiction [1]. The use of wet etching techniques for the removal of sacrificial materials often excludes metals such as aluminum, which is a standard interconnect material in a CMOS circuit. The use of vapor phase HF etching allows the use of aluminum and at the same time provides stiction free release [2]. However, in addition to aluminum interconnects the intermetal dielectrics of a typical CMOS circuit consist of silicon dioxide. Integration of MEMS and CMOS therefore requires a passivation layer that chemically isolates the two during the vapor HF etching to prevent the CMOS from being damaged in the release process. It has previously been shown that  $Si_3N_4$  is partially etched by vapor HF, and forms residues with an increased thickness [3]. In an extensive study of the etch rates of various materials for MEMS, it was also found that silicon nitrides are etched to some extent, but that aluminum oxide in the form of sapphire is unaffected by vapor HF [4].

However, it was not verified whether non-crystalline aluminum oxide also withstands the vapor HF. Here, we present the results of an investigation of various materials that are of interest for use in CMOS MEMS microsystems devices. It was evaluated how well the various materials withstand a prolonged vapor HF sufficient to release a particular MEMS device.

## II CMOS MEMS MICROSYSTEMS

An example of a CMOS MEMS microsystem is shown in Figure 1. In this example, a MEMS device is post-processed onto a CMOS electronic circuit by surface micromachining techniques. The MEMS device contains metal conductors made of AlSiCu, a standard material used for metal interconnects in CMOS. In this case, a single level mechanical layer of monocrystalline silicon has been mounted on a sacrificial layer made of undoped silica glass (USG), which is deposited by plasma enhanced chemical vapor deposition of tetraethylortosilicate (PECVD TEOS). The silicon layer is formed by layer transfer from a silicon-on-insulator (SOI) wafer [5]. Electrical connections to the AlSiCu electrodes are provided by tungsten plugs.



**Figure 1: Schematic of a CMOS-MEMS microsystem with a buried passivation layer that allows release by vapor HF etching of the undoped silica glass (USG) sacrificial layer of the MEMS structure.**

The mechanical materials shown in this example can be replaced by a wide variety of materials,

including ceramics and metal alloys, depending on the application. Common to them all would be the use of silicon dioxide as the sacrificial material. One particular advantage is that silicon dioxide allows the use of well established processes for chemical mechanical polishing (CMP). It is therefore possible to obtain a high planarity of the CMOS substrate as well as very low surface roughness. The silicon dioxide layer then provides an ideal base for the further surface micromachining. This is important in photonic applications, especially those that involve the use of ultraviolet light, where an exceptional planarity and surface quality is required. The device shown in Figure 1 is a cross-section of a micromirror. An array of thousands of such mirrors form a spatial light modulator (SLM) that can be used to control the phase of the reflected light, with application in such areas as adaptive optics (AO) and microlithography [6]. Deflection of the mirrors is obtained by applying a voltage between the mirror plate and one of the two electrodes on both sides of the mirror. It is important that the mirror is planar and that the mirror to electrode gap is accurately defined.

A key element of the structure shown in Figure 1 is the buried passivation layer in between the MEMS and the CMOS part of the device. This layer protects the CMOS circuit during the release process. It has been the main objective of this investigation to find a suitable material which is compatible with both the CMOS and the MEMS processes, and that would be entirely inert to the vapor HF. The etch stop material would be applied on top of the wafer containing the CMOS electronics. Vias would have to be formed to provide electrical connections to the CMOS. Formation of the MEMS structure would then follow by using surface micromachining techniques.

### III EXPERIMENT

The etching system used is commercially available, and consists of a heated sample holder that forms the lid of a closed container containing a small quantity of a standard 49% HF solution [7]. The system differs from some of the more complex systems that are currently available [3,8], in that it runs at atmospheric pressure and does not use any

additional flow of nitrogen or methanol. Instead, the system relies on the evaporation at room temperature of HF from the reservoir, which creates a saturated water/HF atmosphere. It is believed that the etching of the oxide is initiated by water adsorbed on the sample surface, which leads to the formation of silanol groups. The hydrofluoric acid then attacks the silanol groups to form volatile silicon tetrafluoride, which is then desorbed from the surface, and the etching of the oxide continues [9]. Water is both a catalyst and a byproduct of the reaction. The oxide etch rate can therefore be effectively controlled simply by adjusting the sample temperature. A higher substrate temperature leads to less adsorbed water, and thus a reduced etch rate.

In a first experiment, MEMS structures similar to the one shown in Figure 1 were built directly on silicon wafers. These devices were then etched with vapor HF to get an idea of the etch parameters needed to achieve a complete release. These samples also contained a layer of  $\text{Si}_3\text{N}_4$  (PECVD) between the electrodes, and it was a secondary objective of the experiment to check the suitability of this material as an etch stop layer.

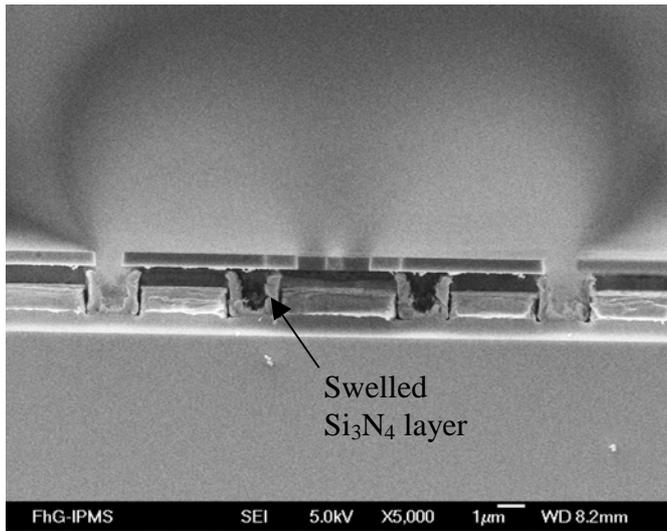
Materials test samples with the various thin films were prepared by coating 6" wafers using a various deposition techniques such as sputtering, evaporation, and PECVD. The wafers were then cleaved to form samples of size 20mm x 20mm. The dielectric materials intended for use as etch stop were coated directly onto bare silicon as well as on wafers covered with a layer of silicon dioxide. The oxide was included to better reveal the presence of pin holes in the films. It was anticipated that poor material density of the various thin film materials would lead to attack of the underlying oxide, which would be visible by inspection in an optical microscope. The thin films on silicon were included to allow more accurate determination of the optical constants needed for thickness measurements.

The etched samples were first inspected in an optical microscope for signs of attack by the vapor HF. Those samples that did not show any significant degradation of the surface quality were subject to further measurements. For the thickness

measurements of the dielectric films Variable Angle Spectroscopic Ellipsometry (VASE) was used. x-ray reflectometry was available for the thickness measurements of the metal films, providing the thickness below a maximum limit of about 200nm.

#### IV RESULTS

It was found that the MEMS structures were fully released after etching for 60 minutes with the samples kept at a temperature of 60°C. The silicon dioxide was completely removed, but as can be seen in Figure 2, the Si<sub>3</sub>N<sub>4</sub> between the electrodes swelled significantly. In fact, it swelled enough to touch the underside of the MEMS structures, which would, of course, inhibit the free movement of the structures. This demonstrates very clearly the unsuitability of PECVD Si<sub>3</sub>N<sub>4</sub> as an etchstop layer for release using vapor HF etching.



**Figure 2: The result of vapor HF etching of a device with an etch stop layer of Si<sub>3</sub>N<sub>4</sub>, showing incompatibility with the release process**

The results of the etching of the materials test samples are summarized in Table 1. The same etching time of 60 minutes at 60°C was used to etch all of the samples. It turned out that the various materials were either completely inert or strongly affected by the HF vapor. Two dielectric materials stood out as particularly resistant and did not show any apparent attack by the vapor HF. These were the thin films of Al<sub>2</sub>O<sub>3</sub> and AlF<sub>3</sub>. The Al<sub>2</sub>O<sub>3</sub> was deposited using two different methods, sputtering and evaporation. In both cases the thin film thickness remained unchanged after etching,

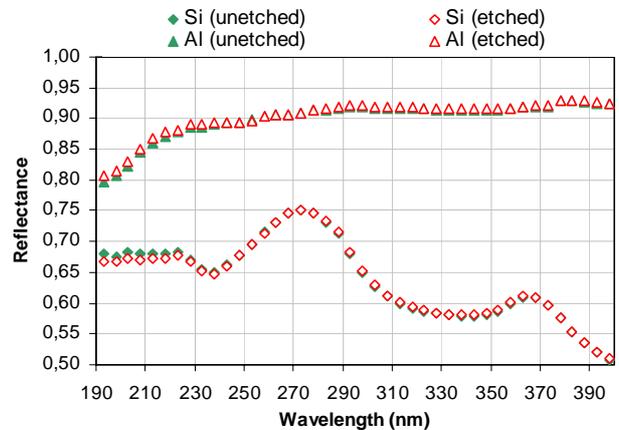
as determined by ellipsometry. Other dielectric materials, such as Ta<sub>2</sub>O<sub>5</sub>, MgF<sub>2</sub>, LaF<sub>2</sub> and also Si<sub>3</sub>N<sub>4</sub> did not withstand the etch, but were more or less completely removed.

**Table 1. Result of material integrity study of various ceramics and fluorides upon exposure to 60 minutes of vapor HF etching at 60°C**

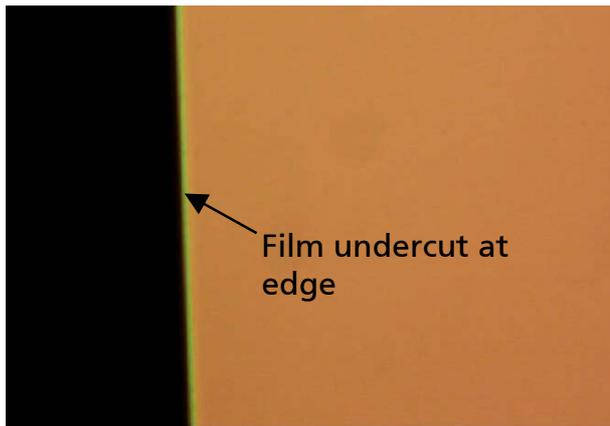
Material	Visual insp.	Comment
Al <sub>2</sub> O <sub>3</sub> (s)	ok	t=56nm (unchanged)
Al <sub>2</sub> O <sub>3</sub> (e)	ok	t=150nm (unchanged)
Ta <sub>2</sub> O <sub>5</sub> (s)	fully etched	(unsuited)
MgF <sub>2</sub> (e)	milky	(unsuited)
LaF <sub>2</sub> (e)	light milky	(unsuited)
AlF <sub>3</sub> (e)	ok	t=267nm (unchanged)
Al (s)	ok	R <sub>248</sub> =89% (unchanged)
AlSiCu (s)	ok	t=66±2nm (unchanged)
TaSiN (s)	roughened	(unsuited)
Si <sub>3</sub> N <sub>4</sub> (p)	fully etched	(unsuited)
Si	ok	R <sub>248</sub> =66% (unchanged)
TiN (s)	slightly roughened	(use possible)
Ti <sub>3</sub> Al (s)	ok	(use possible)

s=sputtered, e=evaporated, p=PECVD

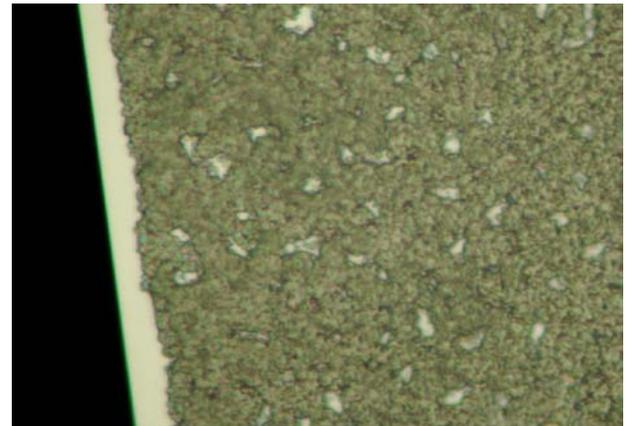
Of the metals tested, both Al and AlSiCu stands out as particularly resistant to the etchant. The thickness of AlSiCu was determined by x-ray reflectometry, and found not to change within the measurement accuracy. Aluminum is particularly interesting for use as a reflective layer in photonic devices that operate in the visible and ultraviolet range of the spectrum. The reflectance in the range 193nm to 400nm was found to be unchanged as a result of the etching, as shown in Figure 3.



**Figure 3: Reflectance of aluminum and silicon before and after vapor HF etching**



(a)



(b)

**Figure 4: Microscope pictures (1000x) of the surface of  $\text{Al}_2\text{O}_3$  (a) and TaSiN (b) after 60 minutes of vapor HF etching at  $60^\circ\text{C}$ . The  $\text{Al}_2\text{O}_3$  was unaffected by the etching, and the underlying silicon dioxide was well protected. The surface roughness of TaSiN increased significantly, indicating poor resistance to the etchant.**

Also shown in Figure 3 is the reflectance of silicon before and after etching, which confirms that the vapor HF does not significantly degrade the optical properties of silicon.

Finally, the metals TaSiN, TiN, and TiAl were tested. Of these materials, only TaSiN was found to be significantly influenced by the etchant. As shown in Figure 4, surface became very rough, in fact too rough for the thickness to be determined by x-ray reflectometry. Both TiN and TiAl appeared withstand the etchant well. The surface roughness of TiN increased slightly, but the material would still function as a hillock suppression thin film on top of AlSiCu interconnects.

## V CONCLUSION

Various materials for use in CMOS MEMS microsystems were tested for compatibility with a vapor HF release process. It was confirmed that  $\text{Si}_3\text{N}_4$  is attacked by the vapor HF etchant, and is thus unsuited as an etchstop material for the process used. It was also found that both  $\text{Al}_2\text{O}_3$  and  $\text{AlF}_3$  are unaffected by vapor HF etching, and thus should be suitable etch stop materials for protection of CMOS electronics in a CMOS MEMS microsystem. Furthermore, it was found that the reflectance of Al is unchanged by the etchant, and that the thickness of AlSiCu used for interconnects remains unchanged.

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