Fabrication of Silicon Microtips with Integrated **Electrodes**

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ABSTRACT

This paper describes a simple method to fabricate silicon microtips with integrated self-aligned polarization electrodes for development of MEMS and electron field emission devices. The method is based on the wet bulk micromachining of the silicon substrate in KOH solutions and utilizes low stress PECVD SiOxNy films obtained at low temperatures (320°C) as structural material for both mechanical support and electrical insulation of the electrodes. For the electrodes sputtered Cr films were utilized. The microtip formation process was studied by optical and electronic microscopy to analyze tip geometry and the characteristic etch rate of the different stages during the tips formation. The fabricated devices were matrixes with different numbers of microtips, each with a typical height of 52 µm and with diameter at the apex below 1 µm. In the best case, the distance between the apex of the tips and the metallic electrode was lower than 5 µm. The results also show that the low stress SiOxNy film is essential to attain the necessary flatness required by the process.

Index Terms: Microtips, MEMS, FED.

1. INTRODUCTION

The possibility of producing controlled mechanical movement or performing free movement in response to an external agent is the fundamental fact that supports the impressive evolution experimented by Micro Electro-Mechanical Systems (MEMS) [1,2,3] and by the enormous commercial importance that MEMS technology has reached in recent years in the sensors and actuators market [4,5]. However, besides the production and detection of movement, MEMS technology is strongly related to the capacity to fabricate microstructures with 3D geometry. Among these microstructures, silicon microtips are one of the more interesting due to their particular geometry and because they allow the development of low voltage electron field emitting devices. In this way, Si microtips exhibit applications in different fields, such as atomic force microscopy (AFM), electron beam lithography, microwave power amplifiers and in the so called Vacuum Microelectronics, specially for field emission displays (FED's) [6,7,8].

Different approaches have been reported for the fabrication of Si microtips but the simplest techniques are based in bulk micromaching of the Si substrate. For example, silicon tips with excellent geome-

try and size in the range of a few microns and smaller have been fabricated through wet anisotropic etching in alkaline solutions, mixed or not with dry isotropic corrosion steps, and followed by thermal oxidation sharpening [9,10]. Other processes utilize porous silicon as a sacrificial layer in isotropic wet etching, also in alkaline solutions and with similar results [11]. All these processes are simple but have the inconvenience that the polarization electrode must be positioned externally, through manually operated micromanipulators with resolution in the micron range.

To overcome this problem, and taking advantage of the simplicity of silicon micromachinig in alkaline solutions, in this paper a method of fabricating silicon microtips with self-aligned integrated electrodes on (100) oriented Si wafers is proposed.

2. MATERIALS AND PROCESS

The proposed method is based on the underetch observed when the Si substrate is anisotropically etched in KOH solutions through square holes in a mask oriented 450 in relation to the [110] direction of Si (100) wafers. In other words, the microtips are formed in the Si region under the masking material.

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This way, if the masking material has suitable structural and mechanical properties, a self-sustained membrane can be produced. This membrane can be utilized to mechanically support integrated metallic electrodes deposited on it. It must be observed that the resulting microtips will be self-aligned to the electrodes. However, to obtain appropriated self-alignments, it is essential to utilize a low stress material that preserves its mechanical stability and flatness after the Si corrosion in KOH. For example, thermal silicon dioxide (SiO₂), a common masking material for Si etching, is not an appropriate choice due to its low resistance to KOH solution and relatively high internal stress, which leads to non-flat self-sustained films.

In recent years, we have optimized the deposition conditions to obtain silicon oxynitride films (SiO_xN_y) with properties especially suitable for MEMS development. These films can be easily removed in HF, have a high resistance to KOH solution and exhibit very low internal mechanical stress [12,13]. Furthermore, in previous projects [14,15] we have utilized backside Si bulk micromachining techniques to fabricate self-sustained grids and membranes (flat and corrugated) of SiO_xN_y with large areas, up to ~1cm². We therefore utilized thick low stress silicon oxynitride films (SiO_xN_y) obtained by Plasma Enhanced Chemical Vapor Deposition (PECVD) at low temperature (~320°C).

Besides these properties, SiO_xN_y films also exhibit good dielectric properties. This is of particular importance since to observe electron field emission conductivity in the polarized microtips, it is necessary to remove any other conduction mechanism or leak currents. This makes it essential to guarantee the electric insulation between the Si substrate (where the tips are fabricated) and the integrated suspended electrodes. The method proposed here depends critically on the superior chemical resistance, mechanical strength and electrical properties of our PECVD SiO_xN_y.

The studied devices were matrices with different numbers of microtips, which were fabricated following the step sequence described in Fig.1. The process starts with the deposition of a 1.8 µm thick low stress SiO_xN_y film obtained by PECVD on (100) oriented Si wafer (Fig. 1a). After that, a 3500 Å film of chromium (Cr) obtained by magnetron sputtering is deposited and the electrode geometry is defined by conventional photolithography (Fig. 1b and c). The next step is a photolithographic process to open square holes in the SiO_xN_y film, rotated 45° in relation to the [110] direction (Fig.1d). The final step is the wet etching of the exposed Si wafer regions in a KOH solution (28.7%, 80°C) to form the Si microtips under the SiO_xN_y which, after the corrosion, becomes a self-sustained film (Fig.1e).



Figure1. Step sequence to produce the Si microtips : (a) Deposition of 1.8 μ m thick PECVD SiOxNy film and (b) of a 3500 Å film of chromium. (c) Patterning of the electrode geometry. (d) Square holes (inclined 45°) are opened in the SiOxNy film. (e) Anisotropic wet etching of Si in KOH solution to form the microtips.

It is important to observe that the deposition and patterning of the Cr film is made before the Si corrosion to form the tips. This guarantees the selfalignment between the tips and the electrode and simplifies the microtips fabrication process.

As mentioned before, the deposition conditions to obtain SiOxNy films with the necessary high resistance to KOH corrosion and low internal stress were optimized in previous projects and are described in more detail in reference [16]. The films are deposited from appropriate gaseous mixtures of silane (SiH₄) and nitrous oxide (N₂O) in a conventional capacitively coupled r.f. PECVD reactor. The deposition temperature was 320°C and the other parameters are shown in Table I.

Table I: Deposition parameters of the SiO_xN_y films deposited by PECVD

Material	SiH ₄ Flow	N ₂ O Flow
	(sccm)	(sccm)
SiO _x N _y	15	37.5
Pressure	r.f. Power	Temperature
(mTorr)	(W)	(°C)
34	200	320

The structures were studied by optical and electronic microscopy to analyze the tip formation process, the etch rate in the different stages of the tips formation and the final geometric characteristics of the microtips.

3. RESULTS AND DISCUSSION

The corrosion process to form the final microtips involves three different stages which, due to the different exposed crystallographic planes, are characterized by different tip shapes and etch rates. As we can see in Fig.2, the first stage begins when an underetch is induced in the region among four neighboring square holes in the SiOxNy masking material. In this stage, the produced cavities have vertical walls that move laterally, under the SiOxNy film. These walls correspond to (100) planes and, since they are vertical, collapse abruptly when the neighboring cavities meet each other. The result is the formation of a truncated pyramid with a relatively simple geometry and faces that correspond to (111) planes.

The second and third stages (see Fig.3 and 4) are related to the corrosion of the truncated pyramid in the center of Fig.2c and are much more complex due to the appearance and subsequent disappearance of crystallographic planes with high Miller indexes. The second stage is characterized by a rapid corrosion of the edges where the {111} sides of the truncated pyramid intersect (see Fig.3). The characteristic geometry of the resulting structure is normally attributed to the appearance of {311} planes that start to appear at the upper vertices of the truncated pyramids. Note that the second stage ends when the {111} planes meet and the top of the structure forming a regular octagon (see Fig.3c).



Figure 2. First stage of tip formation: (a) square holes in the masking material before the Si etching in KOH. (b) middle of the first stage, before the (100) walls collapse. (c) Formation of the isolated truncated pyramid marking the end of the first stage.



Figure 3. Second stage of tip formation. (a) Start, (b) evolution and (c) end, when a regular octagon on top of the structure is formed.



Figure 4. Third stage of tip formation when the sharpening of the structure occurs until the complete formation of the microtip. (a) and (b) show the evolution and (c) the end of the third stage.

The third stage (shown in Fig.4) is characterized by the sharpening of the structure until the complete formation of the microtip, which ends when the apex of the tip unglues completely from the oxynitride film.

Top view images (from an optical microscope) of the microtips in the first and second stages and a 3D image of the tips after the third stage are shown in Fig.5. The etch rates for each stage are indicated below the images. The indicated etching rates were obtained from the lateral displacement of the planes under the SiO_xN_v film.

The fabricated structures were matrices with different number of microtips made from square holes with sides of 100, 200, 300 and 400 μ m spaced by a constant distance of 50 μ m. After the Si corrosion, this spacing leads to a 50 μ m wide oxynitride lines that stay self-sustained and form the SiOxNy grid that supports the Cr electrode. The width of these oxynitride lines also defines the final microtip size. Therefore, in this paper, all the microtips are approximately 52 μ m high. In Fig.6 we show the SiO_xN_y grid over different matri-



Figure 5. Top view of the microtips in the (a) first and (b) second stages of formation. In (c) a 3D image of the tips after the third stage (note that the SiOxNy grid was removed to better visualize the tips geometry).

ces of microtips as viewed from a scanning electron microscope (SEM). In Fig.6b, the tips are formed from square holes of 400x400 μ m². As can be seen, the grid exhibits excellent structural integrity and flatness, even for a self-sustained length larger that 1 millimeter. In Fig.7 we show a close-up of the final aspect of the tips under the self-sustained SiO_xN_y film, and we can observe that the apex diameter is lower than 1 μ m and the distance to the SiO_xN_y film is lower than 5 μ m.

The most critical point of the process is to determine the moment to interrupt the corrosion, when the tip is already formed and it detaches itself from the SiOxNy grid. In fact, at this point the top of the tips becomes exposed and the corrosion continues at a very high speed, making the tips collapse and quickly disappear. On the other hand, it is well known that it is difficult to attain a fine control of Si wet





Figure 6. (a) Different matrices of microtips as viewed from a scanning electron microscope (SEM). (b) Detail of a microtip matrix formed from square holes of 400x400 μ m². Note the excellent structural integrity and flatness of the self-sustained SiOxNy grid.



Figure 7. Close-up of a microtip as observed from a SEM. (a) The typical height of the tips is ~52 μ m and the diameter at the vertex is bellow 1 μ m. (b) The distance between the apex of the tips and the SiOxNy is lower than 5 μ m.

etching in KOH. An alternative is to utilize a slower corrosion method to finalize the third stage. Following this approach we utilize, with satisfactory results, dry etching in SF6 plasma for the final sharpening of the tips.

The previous discussion was focused on the fabrication process. Thus, to better observe the tips geometry and the self sustained SiOxNy grid, the previous images show the tips without the Cr electrodes. However, since our purpose is to apply the microtips in electron field emission devices, it is essential to evaluate the integration of the metallic electrode. As indicated in Fig.1, the self-alignment between the tips and the metallic electrodes is attained by depositing and patterning the Cr film before the Si corrosion to form the tips. The result can be observed in Fig.8, where we show the final aspect of a matrix of 72 microtips with the self-aligned integrated electrodes as they appears in an optical microscope.

As we can see in Fig.8, the structural integrity and flatness of the self-sustained SiO_xN_y grid allows the production of Cr electrodes with lengths in the order of a few millimeters. In fact, in Fig.8a the square holes in the SiO_xN_y films have dimensions of 200x200 µm² which lead to self-sustained Cr lines with a maximum length of up to 2.8 mm without evidences of structural damages. Furthermore, in Fig.8b we can see the selfalignment of the electrodes and a small circular hole with a 20 µm diameter exactly over the tip apex.

The circles observed in Fig.8b correspond to a small region of the SiO_xN_y grid that must be removed before the deposition of Cr film. For simplicity, these holes were not depicted in the step sequence of Fig.1 but they represent an essential feature to allow the electron flow between the tips and the Cr electrode (note that in finished devices, the tip must be detached from the electrode). The typical aspect of this feature is shown in Fig.9, where we can see the Si



Figure 8. Final aspect of the Si microtips with the self-aligned integrated electrodes. (a) Matrix of 72 tips formed from square holes of 200x200 μ m². (b) Detail of the same structure. Images from an optical microscope.

Figure 9. (a) Typical aspect of Cr electrode over the tips in the third stage of the tip formation. (b) Close-up of the circular region where the SiOxNy must be removed to permit the electron collection in field emission devices. Images from a scanning electron microscope.

tip, the SiOxNy self-sustained grid and the Cr line in the third stage of the tip formation.

A critical factor in structures with integrated electrodes is the continuity of the Cr film. In fact, due to the SiOxNy thickness, the Cr film must cover a ~ 2 µm step in the circle region, which is not an easy task given the size of the step. The absence of continuity leads to the removal of a Cr disk from the top of the tips. A consequence of this is the loss of the electrode near the tip apex. This also results in the undesired corrosion of the top of the Si tips through the circular hole in the Cr electrode, altering the tips geometry. This corrosion is showed in Fig.10 and represents an unexpected result that should be better studied or avoided.





Figure10. (a) Removed Cr disks from the electrode lines on the top of the tips, leading to the loss of the electrode near the tip apex. (b) Undesired corrosion of top of the Si tips through the circular hole in the Cr electrode. Images from a scanning electron microscope (SEM).

4. CONCLUSIONS

In this paper we proposed a simple method to fabricate silicon microtips with self-aligned integrated electrodes. The method is based on the anisotropic etching of (100) Si wafer in KOH solutions and takes advantage of the under-etch observed when the etching is carried out through square holes in the mask oriented 450 respect to the [110] direction. As metallic electrodes, we use Cr films because this material is resistant to KOH corrosion. This allows the deposition the Cr film before the Si corrosion to form the tips, guaranteeing the self-alignment and making the overall process even simpler.

On the other hand, the method requires a low mechanical stress material, with high resistance to KOH corrosion and that preserves its mechanical stability and flatness after the Si tips formation. So, the results described here depends critically on the superior chemical resistance, mechanical strength and electrical properties of our PECVD SiOxNy.

Matrices were fabricated with different number of Si microtips with a height of 52 μ m and a diameter below 1 μ m at the apex. The results demonstrated that the structural integrity and flatness of the SiOxNy grid allow the production of self-sustained Cr electrodes at up to ~5 μ m away from the tip apex in lines with length of a few millimeters

Besides this and despite the promising results, some experimental and technological details must be better studied. For example, it is necessary to clarify the mechanism of the tips' formation and its relation with the tips' final geometry. Also it is necessary to study the minimum attainable controlled distance between the Cr electrode and the tip apex. It is also necessary to improve the process in order to guarantee the continuity of the Cr film in the region near the tip apex. As previously described, if this requirement is not satisfied, undesired corrosion of top of the Si tips will occur, altering the tip geometry.

At the moment we are working in the electrical characterization of the structures in order to identify the conduction mechanism and the occurrence of the electron field emission phenomena.

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