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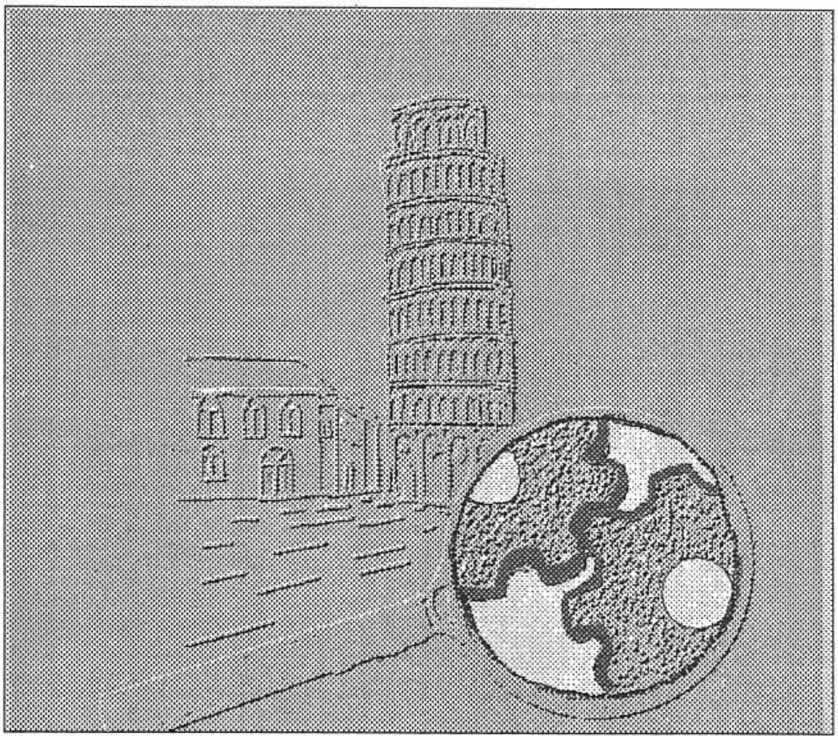

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WORKSHOP DIGEST

THE BLACK SILICON METHOD
A UNIVERSAL METHOD FOR DETERMINING THE PARAMETER SETTING OF A FLUORINE BASED REACTIVE ION ETCHER IN DEEP SILICON TRENCH ETCHING WITH PROFILE CONTROL

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Very deep trenches (up to 200 μm) with high aspect ratios (up to 10) in silicon are etched using a fluorine-based plasma (SF6/O2/CHF3). Isotropic, positively and negatively (i.e. reverse) tapered as well as fully vertical walls with smooth surfaces are achieved by controlling the plasma chemistry. A convenient way to find the processing conditions needed for a vertical wall is described: the Black Silicon Method. This new procedure is checked for three different Reactive Ion Etchers (RIE); two parallel plate reactors and a hexode. The influence of the r.f. power, pressure, and gas mixture on the profile will be shown. Scanning Electron Microscope (SEM) photos are included to demonstrate the Black Silicon Method, the influence of the gases on the profile, and the use of this method in fabricating Micro Electro Mechanical Systems (MEMS).

1. INTRODUCTION

Silicon profile control is an important topic in microtechnology. Especially vertical walls are needed to get high feature densities. Most commonly, wet chemical etchants are used to create anisotropic profiles, because it is cheap and easy to use. However, the etched profile is controlled by the crystal orientation, so there is minor freedom in etching different tapered profiles. Dry plasma etching is becoming a standard tool in microtechnology. Although the basic investments are much higher in dry etching, it is possible to etch controllable profiles without using the crystal orientation. Plasma etching can be divided into three main groups; the physical ion beam etching (IBE), the synergetic reactive ion etching (RIE), and the chemical plasma etching (PE). Generally, IBE shows only positively tapered profiles, low etch rates, and low selectivity, whereas PE gives rise to isotropic profiles, high etch rates, and high selectivity. In RIE it is possible to provide the plasma with a chemical etchant for the etching of the substrate, a passivator for blocking the etching at the sidewalls of a trench, and an ion source for the local removal of the passivation layer at the bottom of the etching trenches. When these processes are controlled in the correct manner, it is possible to create all kinds of trenches with excellent profile control, high etch rates and selectivity. To increase the etch rate, standard RIE is modified to create a higher density plasma [a-f], but these etchers are expensive and therefore less attractive.

Normally, halogen-based plasmas are used for the chemical etching of silicon [a-n], because of their high etch rates. Except for the fluorine-based plasmas, these gasses are particularly hazardous (e.g. chlorine, bromine, and chlorinated compounds) and special precautions are recommended. The passivation layer can be grown; *1* from polymer precursors which are let in to the plasma [h-j], *2* by resputtering mask material [k], *3* by inserting gases which act as an oxidant forming siliconoxyfluorides [a,l-n], *4* or by freezing the normally volatile reaction products of the silicon with the radicals [c,d]. The deposition of a polymer film has the disadvantage that this film is thermally less stable than a growing inorganic siliconoxyfluoride film and the freezing of reaction products uses the expensive (cryogenic) coolers The resputtering of mask material is not acceptable because areas which should stay clean are also contaminated. Because the passivating film is very thin the incoming ions don't have to be highly energetic, so the selectivity can be very high and the substrate damage is low. Also, because of the low energy of the ions, trenching and faceting are not found and it is very easy to change the direction of the impinging ions thus changing the etched profile. A major problem during etching silicon vertically is the forming of "grass" on the silicon surface, because of all kinds of micro masks deposited or grown on the silicon [n] (see fig. 1).

In this study, the low-toxic and easy to handle SF6/O2/CHF3 gases are chosen for the reactive ion etching of silicon [n]. A short description of the etch mechanism of silicon in such a plasma is given and a new method to find the fully vertical profile regime is introduced which uses the forming of grass: the Black Silicon Method. When this regime is found, CHF3 is added to prevent the forming of grass. It is shown that this method is a practical tool for finding the parameter setting of an arbitrary RIE. The influence of the r.f. power, pressure, and gas composition on, and its relation to, the profile is given in a diagram. With the help of this diagram it is easy to direct the chemistry for the desired profile. The aim of this work is the fabrication of high aspect ratio (depth/width) features for the use in Micro Electro Mechanical Systems (MEMS). As an example, a MEM comb driven xy-stage is given.

2. THE SYNERGETIC MECHANISM OF SF6/O2/CHF3 PLASMAS

In an SF6/O2/CHF3 plasma, each gas has a known specific function and influence, so the etched profile is easily controlled just by changing the flow rate of one of these gases [n]. In such a plasma SF6 produces the F* radicals for the chemical etching of the silicon forming the volatile SiF4. O2 creates the O* radicals to passivate the silicon surface with SiOxFy, and CHF3 is the source for the CFx+ ions which etches the SiOxFy layer in one direction forming the volatile COxFy (see diagram 1). Of course, SFx+ ions are also able to remove the oxyfluoride by way of the volatile SOxFy gases, but the SF6 flow is fixed on the O2 flow to ensure a vertical wall. Thus, the CHF3 gas is a nearly independent source of oxyfluoride-etching ions.

A more or less contrary mechanism can also explain the directional etching. In this mechanism the CFx* species are passivating the silicon surface which are etched by way of imparting O+ ions (see diagram 1). However, this mechanism is less likely in the pressure regime used in our study as will be clarified further on.

The SF6/O2/CHF3 chemistry is able to etch highly controllable profiles in silicon at very low ion energies (20-90eV) and high etch rates (up to 3μm/min). The low ion energy prevents substrate damage (electronics), mask erosion (the selectivity to metal masks is practically infinite), and makes it easy to change the profile of the trench. The ion energy is ruled by the potential which is developed between the plasma and the powered electrode; the d.c. self-bias. Gases like O2 and CHF3 create high bias voltages whereas SF6 give rise to very low voltages. Thus, when the oxygen flow is increased the d.c. self-bias also increases and ions will gain more energy before colliding with the substrate surface. The d.c. self bias decreases when the power decreases or the pressure increases.

In etching silicon with the SF6/O2/CHF3 mixture there is a constant competition between the etching fluorine radicals and the passivating oxygen radicals. The etching is increased directionally by way of the CFx+ ions. When the SF6 content is increased, the formation of the blocking layer is less pronounced and therefore the profile will be more isotropic (i.e. PE-like, see fig.2). Increasing the oxygen content will decrease the chemical etching and the etch mechanism will become less isotropic. At higher oxygen concentrations the etching will become physical which results in positively tapered profiles (i.e. IBE-like, see fig.3). Increasing the CHF3 content will increase the removal of the blocking layer, thus making the profile less positive tapered. Moreover, the ions are charged positively, whereas the substrate is negatively biased and because of this mechanism it is possible to create negatively (i.e. reverse) tapered profiles (ion bowing, see fig.4). At higher CHF3 concentrations CFx specimens will scavenge the oxygen radicals, thus preventing the blocking layer to form, which results in a more isotropic profile. A higher pressure or lower power results in a more positively tapered profile, because the energy of the impinging ions is lower (d.c. self bias). In these cases, of-normal ions are more likely to reflect from the sidewalls without etching it. When the etching is performed in the isotropic or negatively tapered regime, thus at low oxygen, high CHF3 flow, low pressure, or high r.f. power, micro masks such as native oxide, dust, or resputtered mask material will be constantly underetched and etched surfaces are staying smooth preventing the forming of grass.

3. THE BLACK SILICON METHOD

Of course, it is possible to continue this paper by giving the recipe for the RIE giving a vertical wall profile and show the influence of the various parameters on this profile such as power, pressure, and gas flow. However, this information can't be used in other laboratories, especially when they have a different reactor. This problem has its origin in the difference of the configuration of the etcher (plan-parallel, triode, hexode, etc.) but also in the reactor geometry, the flow handling (turbo-, diffusion pump, etc.), and many others. For this reason a stronger tool is used to find the vertical profile regime. This method uses the fact that the silicon is turned black when the vertical wall recipe is found. From now on, this method will be called the "Black Silicon Method". Another method using response surface methodology has previously been described by the authors [n]; it includes information over the equipment and the actual recipe. Before the Black Silicon Method is formulated, the reason for this effect will be explained and a way to get rid of this blackening will be described.

The origin of black silicon:

As stated in the above there is a constant competition between the fluorine radicals that etch and the oxygen radicals that passivate the silicon. At a certain oxygen content there is such a balance between the etching and the passivation that a nearly vertical wall results. At the same moment native oxide, dust, et cetera will act as micro masks and, because of the directional etching, spikes will appear. These spikes consist of a silicon body with a thin passivating siliconoxyfluoride skin. They will become higher in time and, depending on the etch rate, they will exceed the wavelength of incoming light after some time. This light will be "catched" in the areas between the spikes and can't leave the silicon surface anymore. So, all the light is collected by the etching surface and it is turned into black. In fact, this optical diffuser could be used for all kinds of applications where the reflection of light from the surrounding is not desired, e.g. laser applications or sunlight collectors. In figure 1, a SEM photo is shown from an etched silicon piece under directional conditions. The spikes are 50μm in height and a few μm in width. The origin of micro masks is caused by native oxide, dust, and so on which is already on the wafer before etching. But, it is also formed during the etching because siliconoxide particles coming from the plasma are adsorbing at the silicon surface or because of the oxidation of the silicon surface together with the angle dependent ion etching of this oxide layer. Another source of particles during etching which will act as micro masks is the resputtering of mask material due to imparting ions.

Preventing black silicon: Spikes which are formed because of dirty wafers before etching are easily controlled by giving the wafer a precleaning step. For instance, native oxide can be removed with the help of an HF dip and dust is less a problem when using the lift-off technique in applying the mask layer, instead of the normally used chemical etching of the mask material with the help of a resist pattern. However, the micro masks which originates during etching must be controlled in a different way. First of all, the resputtering of mask material can be suppressed when the ion energy is low or when the right materials are chosen, but this subject will be treated elsewhere (o). The siliconoxide particles are less a problem when the selectivity between the silicon and the silicon oxide is minimised, but this only occurs when the incoming ions are highly energetic and at these moments the process is not favourable anymore because of substrate damage and the just mentioned mask erosion. As already stated, it is possible to forbid spikes from forming by constantly underetching the micro masks isotropically or etching the features with a slightly negative undercut. The isotropic solution makes only sense when it is used as a post etch, because otherwise the feature density is limited. On the other hand, the negative underetching is an excellent way to control the smoothness of the substrate surface barely limiting the feature size density. In this study the addition of CHF_3 to an SF_6/O_2 plasma is described and its ability to prevent grass. Yet another approach to attack the grass problem is the application of different masks, but this will be published elsewhere (o).

The black silicon method: In this section, an easy way to find the vertical wall regime is described with the help of the information already given. A more or less general tool is reached in which the receipt for any RIE system can be found just by fulfilling the sequence written down below. As can be concluded from point 3 of this sequence purely vertical walls can be achieved for any pressure, power, O_2-, CHF_3-, or SF_6-flow. This is an important conclusion because now we are able to create any d.c. self bias we want without changing the profile. For instance, it is possible to develop very low bias voltages (<20eV) at the higher pressures giving very high mask selectivity, maintaining the profile. In such cases the etched silicon bottom and the sidewalls are nearly perfect as shown in fig.6. It is also observed in diagram 2 that a vertical wall profile is found for zero CHF_3 flow. This means that the passivation with siliconoxyfluorides at the side walls is more likely than the passivation with a fluorocarbon layer, although it is still possible that at different pressure, loading, et cetera, the fluorocarbon layer is more pronounced.. Also, the observation that increasing the oxygen flow gives rise to a more positively tapered profile is a strong indication than siliconoxyfluoride is the sidewall passivator. Auger analysis showed that indeed the sidewalls are covered with silicon oxide; there is no carbonic species found [n].

The black silicon method is tested for three different RIE systems. Most experiments are performed with a plan parallel plate reactor "plasmafab 340" from the STS company [n] and a second plan parallel plate single wafer reactor "plasmatherm 500" showed identical results. A third system, the hexode "AME-8100" from Applied Materials, is used for the batch fabrication of silicon wafers and is also able to achieve vertical profiles. However, the etch rates are approximately one order in magnitude lower than for the single wafer etchers and for this reason less powerful. This is because the wafers are much longer exposed to the aggressive plasma chemistry giving rise to surface roughening when etching very deep trenches in silicon. The etch rate can be increased by decreasing the reactor loading, but this subject will be described elsewhere [o].

The formulation of the Black Silicon Method

1. Place a piece of silicon in the reactor and adjust the preferred power and pressure for an SF6/O2 plasma. Etch ca. 1 micron of silicon, open the process chamber, and look if the silicon is black. If not, do the same again but increase the oxygen flow. Proceed with this sequence until the wafer is black. Increasing the oxygen too much, still will give rise to black silicon since there exists a positive tapered profile without any underetching. Alternatively, it is possible to sense the black silicon with the help of a laser/photodetector set-up.

2. After the black silicon regime is found add some CHF3 to the mixture and increase this flow until the wafer is clean again. Too much CHF3 will make the profiles isotropic (and smooth) because the CFx species are scavenging the oxygen radicals which are needed for the blocking layer.

3. Now a wafer with the mask pattern of interest is inserted in the reactor and the etched profile is checked. Increasing the SF6 content will create an isotropic profile (fig.2). Adding too much oxygen will make the profile positively tapered (fig.3) and extra CHF3 will make it more negatively tapered (fig.4). Adding at the same time O2 and CHF3 with the correct balance will create very smooth and nearly vertical walls (fig.5). Increasing the pressure or decreasing the power will make the profile more positively tapered. In diagram 2 the influence of the O2/CHF3 flow and the pressure/power on the profile is given. Increasing at the same time the O2 and CHF3 flow, pressure and CHF3 flow, power and O2 flow, or power and pressure will hardly change the profile. However, such an increase will increase the d.c. self-bias and a higher d.c. self bias will give the of-normal ions enough energy to etch the sidewalls, thus changing the profile a little. Structure heights of 100 micron with an undercut of less than 1 micron are achieved.

5. APPLICATIONS AND CONCLUSIONS

Wafers which are purposely not cleaned or even oxidised in an oxygen plasma and etched in the Black Silicon Regime can be used as an optical diffuser for e.g. laser applications. It is possible to create spikes at well-defined locations in order to form a tip for the use in AFM applications. In our study we are mainly interested in the use of the Black Silicon Method for MEMS applications. In figure 7, a micromachined xy-stage is shown. The structure is etched during one run with standard RIE. After the directional etching, the sidewalls are passivated using a low pressure CHF3 plasma and the xy stage is etched free with the help of an isotropic SF6 plasma. In the same run the structure is passivated with a fluorocarbon layer using a high pressure CHF3 plasma [p].

Although the Black Silicon Method is tested for the SF6/O2/CHF3 plasma only, it will also work for other silicon etch gases e.g. CF4, NF3, SiF4, CF3Br, or Cl2. In fact, every plasma mixture which consists of a chemical etchant, a passivator and an ion source can be used for the Black Silicon Method., even when the substrate is not silicon et all but e.g. a polymer. All together it is shown that the Black Silicon Method is a very strong tool for etching high structures with excellent profile control using an SF6/O2/CHF3 plasma

6. REFERENCES

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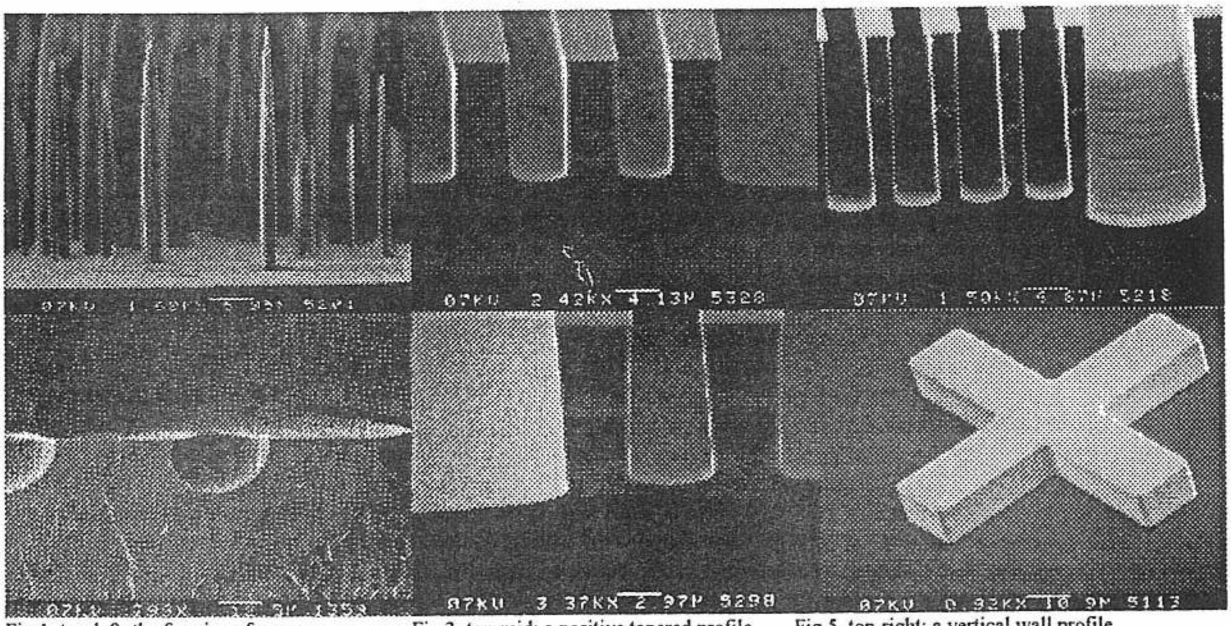

Fig.1, top-left: the forming of grass.
Fig.2, bottom-left: an isotropic profile
Fig.3, top-mid: a positive tapered profile
Fig.4, bottom-mid: a negative tapered profile
Fig.5, top-right: a vertical wall profile
Fig.6, bottom-right: an etched structure

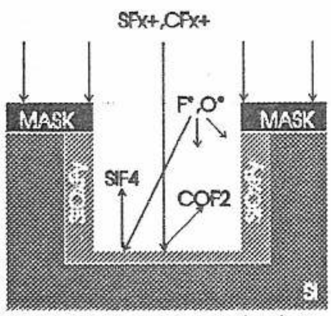

Diagram 1: the SF6/O2/CHF3 chemistry

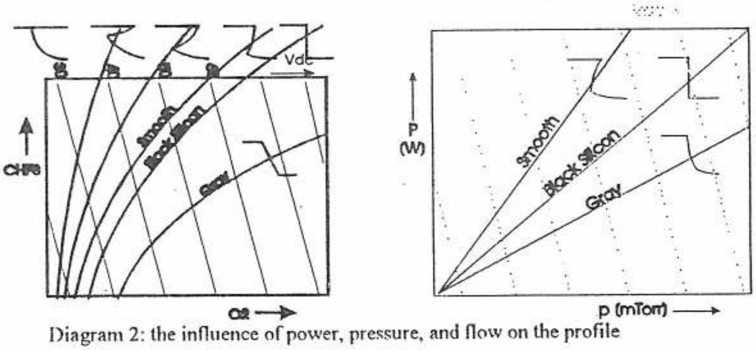

Diagram 2: the influence of power, pressure, and flow on the profile

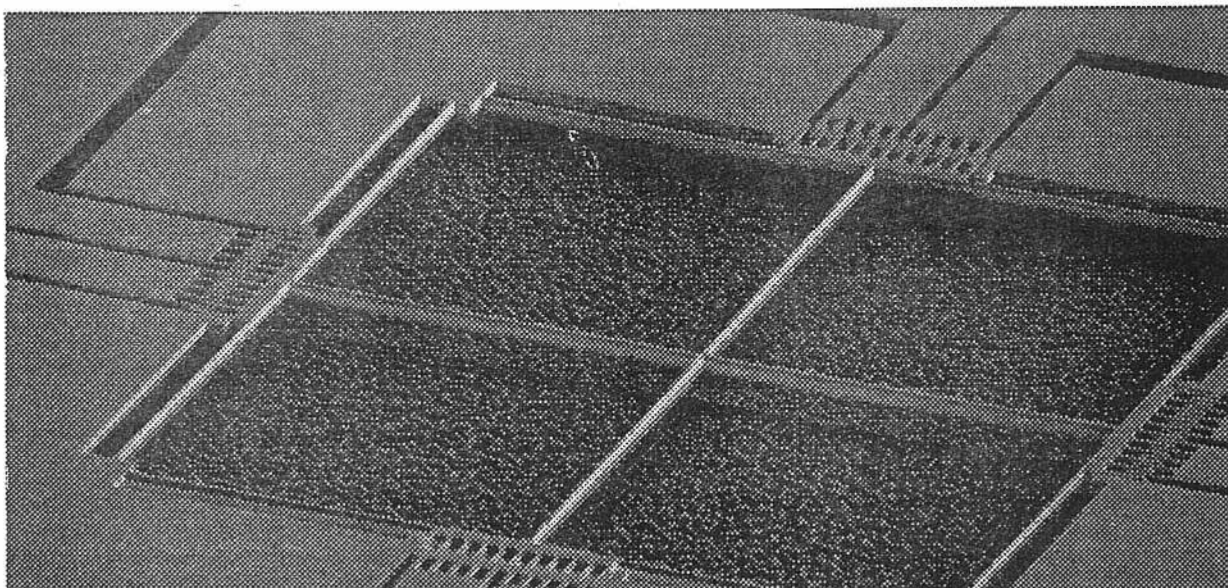

Fig.7: A micromachined xy stage etched with the help of the Black Silicon Method in an SF6/O2/CHF3 plasma.