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Anisotropic Wet Chemical Etching of Si for Chemical Analysis Applications

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A convenient starting point for miniaturization in analytical chemistry lies in the use of single crystal Si as a starting material with micromachining as an enabling technology and wet chemical etching as the key micromachining tool. In this feasibility study and learning exercise, Si micromachining will be reviewed and anisotropic wet chemical etching for the formation of channels, columns and other geometric patterns of possible use in chemical analysis applications will be described. Silicon wafers with <100> and <110> orientation were tested and EDP and hydrazine-, NaOH- and KOH-based etchants were evaluated.

1. Introduction

The lack of reactivity toward most chemical species, the desirable mechanical(1) properties and electrical and etching characteristics(1–7) make doped single crystal Si a convenient starting point for miniaturization in analytical chemistry. In such a case, silicon micromachining may be used as an enabling technology for miniaturization and anisotropic wet chemical etching as a key micromachining tool.

The main objective of this work was to evaluate different anisotropic etchants for micromachining of columns, splitters and other variants of geometric patterns that may be used as building blocks for the construction of more complex micromachined structures for possible use in chemical analysis applications. In the following sections, micromachining will be reviewed, the experimental requirements for anisotropic wet chemical etching will be presented and the shapes obtained after etching the types of patterns mentioned above will be discussed in detail.

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2. Review of Micromachining

Several methods, such as wet chemical etching,[1–8] reactive ion or plasma etching,[8–16] laser micromachining,[17–19] micromilling[20] and ultrasonic machining[21] have been used for the micromachining of Si[1–21,24–28] and other materials.[8,22,23] Of these, wet chemical etching of photolithographically 'stamped' patterns on single crystal Si wafers is the least expensive and has been widely used.[1–7,29–73] The cited literature covers the period from 1989 to date. The period prior to this date is reviewed elsewhere.[1–7]

Wet chemical etching can be isotropic or anisotropic (Fig. 1). Isotropic etchants have the same etch rate for all crystal planes. Anisotropic etchants favor one crystallographic plane over another and, typically, their etch rate decreases considerably in areas that have been doped with a high concentration of a dopant, such as boron.[1,2] Although complex patterns have been etched in Si using anisotropic wet chemical etching, their shape and size after etching is governed by fundamental parameters, such as crystallographic planes and depends on wafer orientation and experimental conditions.

Since anisotropic etchants can be used to sculpt trenches, grooves and channels as well as Manhattan-type and regular geometric shapes with well defined edges and sharp corners, they have become a key micromachining tool, especially in the area of micro electro mechanical systems (MEMS).[27–28]

2.1 *Commonly used anisotropic etchants*

Anisotropic etching can be dopant dependent or independent and can have different selectivity toward masking materials. Some key characteristics of selected anisotropic wet chemical etchants are listed in Table 1 (which is adapted from Petersen[1]). All anisotropic wet etchants contain aqueous alkaline solutions of an inorganic or an organic reagent. For convenience, such etchants can be grouped in four main categories.

a) Alkali metal hydroxides,[29–56] such as LiOH,[33] NaOH,[33] KOH[29–51] or CsOH.[52–56]

b) Other inorganic hydroxides, such as NH_4OH.[57–60]

c) Quaternary ammonium hydroxides, such as tetra-methyl ammonium hydroxide[61–64] (TMAH or $(CH_3)_4NOH$).

d) Amines,[65–73] such as hydrazine[65,66] (H_2N-NH_2 or N_2H_4) or ethylenediamine[37,67–71] (H_2N-CH_2-CH_2-NH_2) or ethanolamine[72,73] ($H_2NCH_2CH_2OH$).

Anisotropic etching may occur under an applied bias or under dry rather than wet etching conditions.[8] However, since these were not used here, they will not be discussed further. Moreover, since NaOH-, KOH-, hydrazine- and etlylenediamine-based etchants were tested, only their advantages and disadvantages will be briefly discussed.

2.2 *NaOH and KOH*

NaOH-water and KOH-water mixtures of these inorganic hydroxides are stable, inexpensive and do not generate toxic fumes at room temperature. Due to their desirable etching characteristics, alkali metal hydroxides have been widely used with KOH, being the most widely used and documented etchant.[29–51] Mixtures of KOH and water are highly anisotropic (Table 1), have high etch rates, are well suited for the fabrication of wide a)

b)

c)

d)

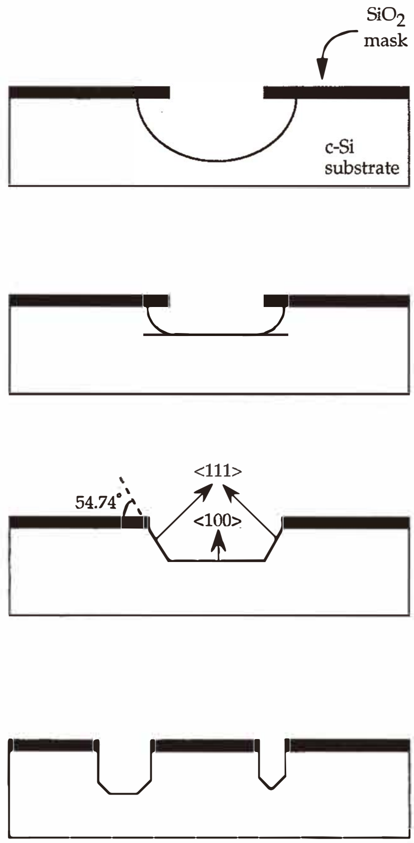

Fig. 1. Isotropic and anisotropic etching for (a) isotropic etching with agitation and (b) isotropic etching without agitation. Undercutting of the SiO_2 mask is also shown in (a) and(b). Anisotropic etching for (c) <100> wafer orientation and (d) <110> orientation.

windows and high-aspect-ratio microchannels and grooves and they provide reproducible results in terms of etch quality and uniformity.

The main disadvantage of KOH is that it attacks SiO_2 masks (Fig. 1) at an appreciable rate, thus requiring a Si_3N_4 mask, especially if deep etches are to be employed. Additional concerns arise when attempting to incorporate on-chip circuitry along with micromachined structures. For example, Al metalization and bonding pads are attacked by KOH and must be protected (thus requiring an additional mask or use of other etchants) and Na^+ and K^+ released during etching may contaminate the gate of MOS transistors. These concerns have been addressed by etching Si wafers from the back or by using non-alkali metal based etchants such as hydrazine or ethylenediamine.

Table 1
Characteristics of selected anisotropic etchants.

Etchant {Diluent, Additive}	Typical composition(s)	Temp. (°C)	Etch rate*1 (μm/min)	Anisotropy*2	Dopant dependence	Masking film(s) {etch rate of mask}
KOH {H_2O, IPA*3}	44 gr 100 ml	85	1.4	400:1	$\geq 10^{20}$ cm^{-3} B reduces etch rate by about 20	Si_3N_4 or SiO_2 {14 Å/min}
NaOH {H_2O}	10 gr 100 ml	65	0.25–1.0	----	$\geq 3\times10^{20}$ cm^{-3} B reduces etch rate by about 20	Si_3N_4 or SiO_2 {7 Å/min}
CsOH {H_2O}	60%	50	1*4	200:1*5	----	SiO_2{0.25 Å/min}
NH_4OH {H_2O, pyrazine*3}	9%	75	0.5	25:1	Etch rate is B concentration dependent	SiO_2 {1 Å/min}
TMAH {H_2O}	22%	90	1.0	25:1	Etch rate is B concentration dependent	SiO_2 {1 Å/min}
Ethylene diamine {H_2O, Pyrocatechol}	750 ml 100 ml	115	0.75	35:1	$\geq 7\times10^{19}$ cm^{-3} B reduces etch rate by about 50	Si_3N_4 {1 Å/min} SiO_2 {2 Å/min} Au,Cr,Ag,Cu,Ta
Hydrazine {H_2O, IPA*3}	100 ml 100 ml	100	2.0	20:1	Etch stop using 1.5×10^{20} cm^{-3} B	SiO_2{1.6 Å/min} Al*6
Ethanolamine {H_2O, GA, catalysts*7}	3650 ml 1660 ml	117	2.1 (max*8)	36:40	----	----

ED: $NH_2(CH_2)_2NH_2$ (Ethylene diamine)
GA: $C_6H_2(OH)_3COOH\bullet H_2O$ (Gallic acid)
IPA: $(CH_3)_2CHOH$ (Isopropyl alcohol or 2-propanol)
TMAH: $(CH_3)_4NOH$ (Tetra methyl ammonium hydroxide)
Pyrazine: $C_4N_2H_4$
Pyrocatehol: $C_6H_4(OH)_2$
Hydrazine: NH_2NH_2
Ethanolamine: $H_2NCH_2CH_2OH$

*1 <100> orientation
*2 Etch rate of <100>/<111>
*3 Used occasionally
*4 <110> orientation
*5 <110>/<111>
*6 Etching may be suppressed
*7 Plus 3 ml of a 10% surfactant
*8 0.012 M quinoxaline ($C_8N_2H_6$)

2.3 *Hydrazine-water*

The etch rate of SiO_2 or Si_3N_4 is small (Table 1) and etching takes place in a clear solution, thus making visual monitoring of the etching process possible. The etching of Al may be suppressed by adding a small amount of Si to the etchant solution.[66] Bi, Cr, Ti and Pd are not etched,[65] but, Cu, Ni and Zn are etched.[65] The anisotropy is not as good as that for KOH (Table 1). The etching must take place under a blanket of inert gas and even a very thin layer of silicon dioxide is sufficient to prevent etching. It is therefore necessary to dip the wafer in hydrofluoric acid immediately prior to etching.

Since hydrazine is a suspected carcinogen and, at high concentrations, hydrazine solutions are explosive, safety is a key concern. This may be partially addressed by its replacement with ethylenediamine.

2.4 *Ethylenediamine-water*

Although ethylenediamine is colorless in its pure state, it readily absorbs CO_2 and moisture from the atmosphere, and turns yellow when exposed to laboratory air, forming a crystalline deposit. Often, pyrocatehol is added and ethylenediamine-pyrocatehol-water (EDP or EDPW) mixtures have found extensive use in etching polycrystalline[67–69] and single crystal silicon, typically at temperatures around the boiling point of EDP (~100–120°C).

As compared to KOH or NaOH, there is no K^+ or Na^+ contamination of the gates of MOS transistors when using EDP and some of its formulations do not attack Al. Thus, they can be used to etch wafers from the front even when there are unprotected bonding pads. This enables the integration of CMOS electronics along with micromachined structures,[28,74] thus opening a wide range of possibilities for the development of personal or portable analytical measurement tools. Since high concentrations of B can be used as an etch stop (Table 1), a maskless anisotropic etch step also becomes possible. Even though EDP attacks SiO_2 masks very slowly (Table 1), and has been shown to be an excellent etchant, its solutions age rapidly,[75] necessitating frequent replacement and increasing cost. Also, its volatility at room temperature, the toxicity of its fumes and waste disposal considerations limit its wide applicability.

2.5 *Inter-comparisons and etchant selection*

A common disadvantage of these etchants is a white residue[29,31,42,43,51,54] and pyramid formation[29,39,51,57,61,62,66,70,71] on the etched surfaces. Examples are shown in Fig. 2. Although their formation[76–78] depends on experimental conditions, they are typically observed when deep etches are employed. This is an important consideration in this work since such etches are necessary. An example is the fabrication of high-aspect-ratio microchannels that may be used to transport chemical species from an inlet, to the detector, to an outlet. Further inter-comparisons are difficult to make due to the use of differing experimental conditions. In addition, the mechanism of anisotropic etching is not fully understood and, although software-based etch-simulators are beginning to appear,[79–84] etchant selection is mostly based on heuristics. The difficulties in predicting the shape obtained after etching a pattern-on-a-wafer induces trial-and-error studies and one such study is described below.

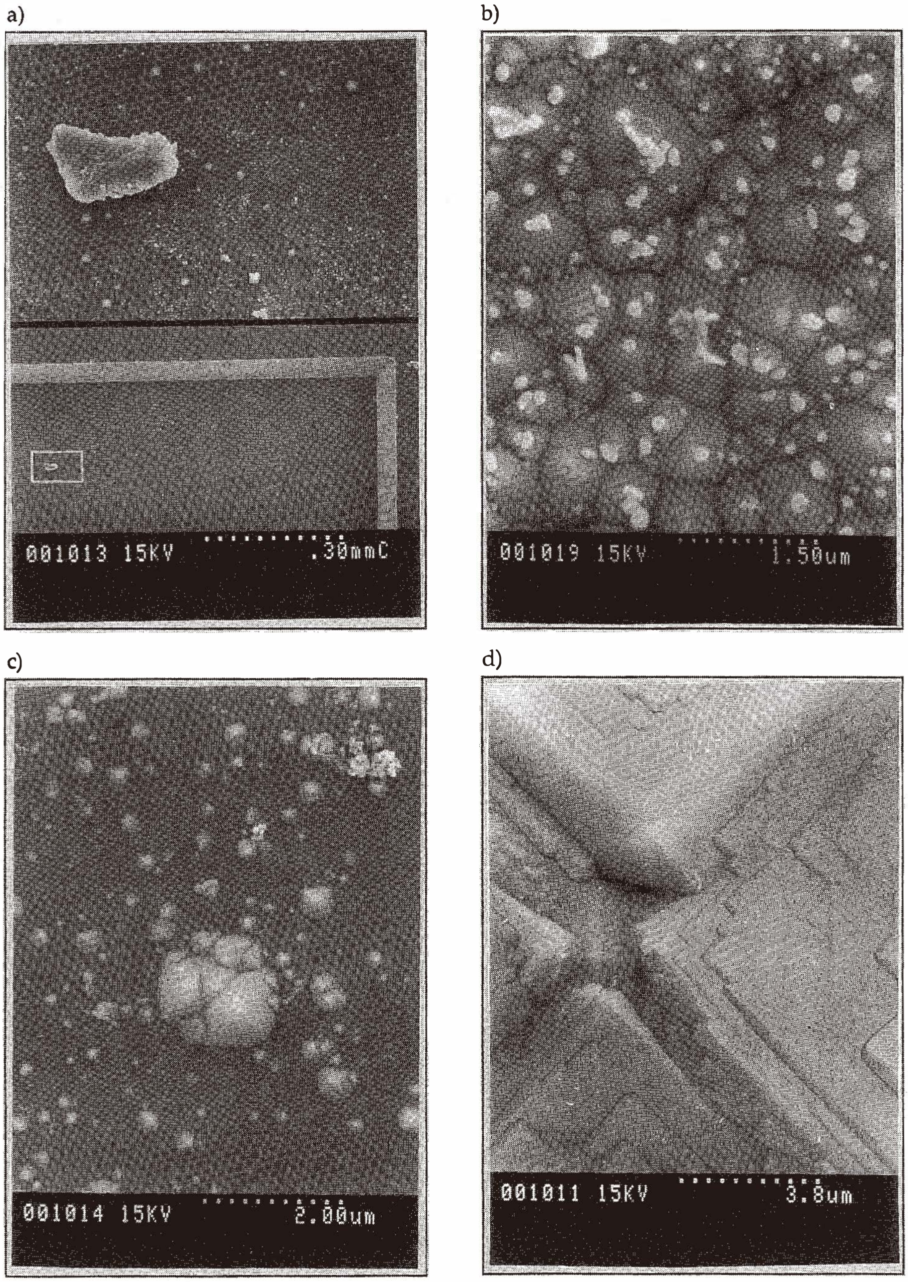

Fig. 2. Examples of white residue ((a) and (b)) on the wafer and of pyramids ((c) and (d)). Close-up of pyramid in (c) is shown in (d).

3. Experimental

Several factors have been reported to affect the choice of an etchant. Among them are etch-composition, -time and -temperature, stirring rate, illumination conditions, wafer quality and crystallographic orientation and doping. In this work, only n-doped wafers were tested using the etch-compositions and etch-temperatures listed in Table 1 and the effect of stirring was studied briefly. Also, only <100> and <110> wafers were tested because <111> wafers typically etch far too slowly to be of any use.

3.1 *Si wafers*

The silicon wafers used in this work were n-type doped (Phosphorous) with a nominal thickness of 376 ± 25 μm and $2-5$ Ω·cm resistivity. A layer of SiO_2 (approximately 1 μm) was grown by oxidation at 1000–1200°C on the top surface of the wafer.

3.2 *Patterns*

Different sizes of straight and cross-connected channels, columns, splitters, regular polygons and circles were designed (Fig. 3). These patterns, most of which are unusual for MEMS or microelectronics, may be used as the basic building blocks for the development of more complex patterns. For instance, the modified ICP torch (top right-hand corner of Fig. 3) combined many of the regular geometric primitives discussed above and was used to evaluate the etchants with a more complex design.

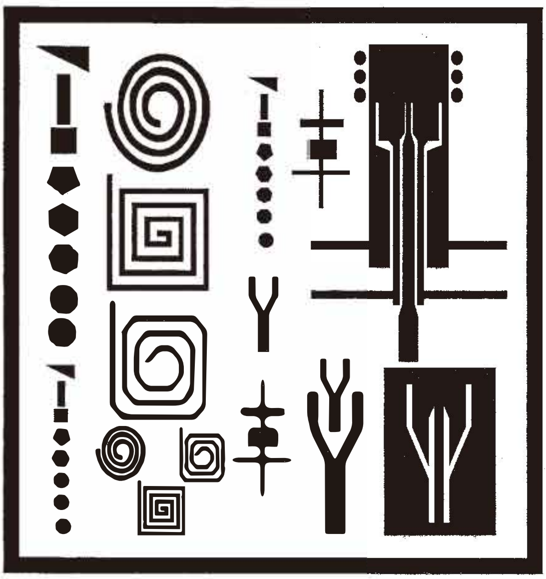

Fig. 3. Mask.

3.3 *Mask*

The patterns were transferred onto the mask (Fig. 3) and the pattern on the mask was transferred onto the 1.1" × 1.1" working area of the wafer using standard photolithographic techniques. The patterned-wafers were etched anisotropically following a low-temperature (*e.g.*, < 120°C) etching procedure (or "recipe" as it is often called), as outlined below.

3.4 *Typical etching procedure*

- The etchants were mixed thoroughly prior to use.
- The etchants were placed in an appropriate etch-bath which was kept at the desired etching temperature.
- The wafer was immersed in 3% HF solution for 60 s.
- At the end of the 60 s time-period, the wafer was removed from the HF solution and it was rinsed for 60 s with deionized water (18 MΩ).
- The wafer was placed on a Teflon wafer-holder.
- The Teflon holder with the wafer in place was immersed into a thermostated etch bath and timing was started.
- At the end of the 2 h etch-period, the wafer was removed from the etch bath and from the wafer holder and it was rinsed with deionized water (for approximately 5 min).
- The etched wafer was blow dried (for approximately 5 min).

All the etching took placed in locally-constructed etch baths.

3.5 *Etch bath*

A simple etch bath for NaOH or KOH etching is shown in Fig. 4(a). For hydrazine or EDP, an etch bath with a condenser and a flow of Ar was used (Fig. 4(b)).

3.6 *Safety and waste disposal*

Because etchants are toxic and corrosive and may be flammable, explosive or suspected carcinogens, the handling and waste disposal instructions described in the material and safety data sheets (MSDS) must be followed closely and the application of standard laboratory-safety practices must be considered a mandatory requirement.

4. From Patterns to Shapes

Selected SEM photographs of the etching results are shown in Figs. 5–17. To enable inter-comparisons, SEMs of the etched shapes obtained for each pattern and etchant are placed on the same figure. To allow a closer examination of the etched surfaces, higher magnification was used in some instances. The photographs were examined visually using subjective evaluation criteria, such as undercutting of the mask, sharpness of the etched corners and edges, quality of the etched surfaces and residue and pyramid formation. Qualitative etching results are summarized in Tables 2 and 3. Since the SEMs show results which are, in many cases, self-explanatory only brief descriptions will be given.

a)

b)

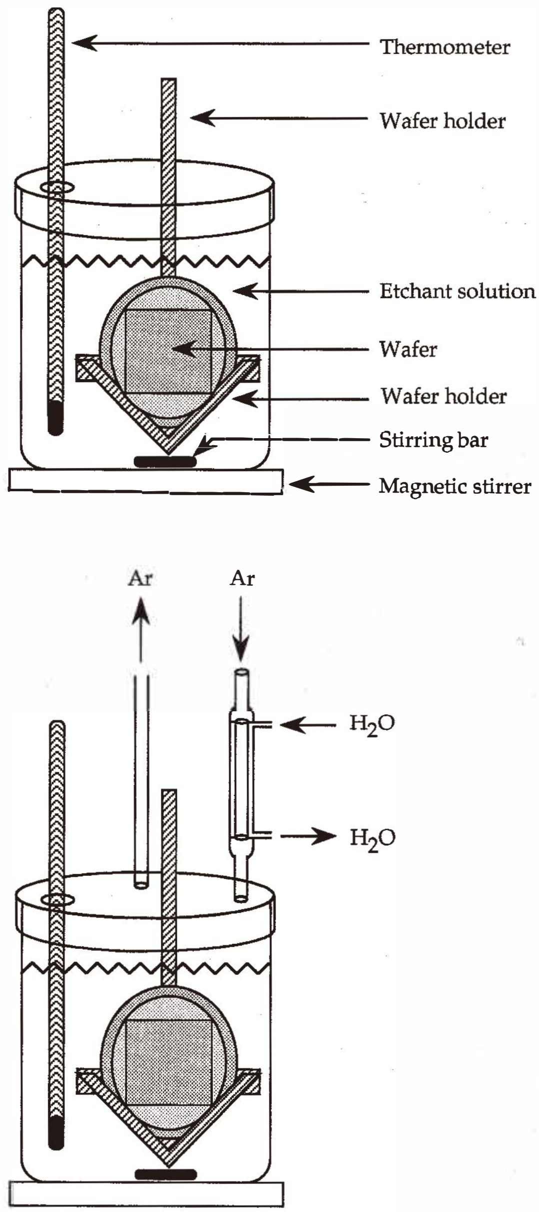

Fig. 4. Etch baths (modeled after refs. 65, 66 and 75) (a) without reflux and (b) with reflux.

4.1 *Brief description of SEMs*

4.1.1 *Cross-connected channels*

The <100> wafers (Fig. 5), formed well defined channels with sharp edges and corners when etched in hydrazine. With NaOH, although sharp edges and corners were obtained, some undercutting of the mask was also noted. With KOH, significant geometric changes occurred and, with EDP, the etched edges and corners became dull and, undercutting of the mask, a white residue and pyramidal protrusions were observed.

For the <110> wafer (Fig. 6), the corners became rounded and some undercutting of the mask was observed when using hydrazine. Significant undercutting was noted when using NaOH or EDP to the point of making the etched shapes unusable. With KOH, the pattern was completely etched away.

4.1.2 *Splitter*

For the <100> wafer (Fig. 7), although some undercutting was observed, hydrazine gave acceptable results. For EDP, in addition to undercutting, some debris and pyramidal protrusions were noted. With NaOH or KOH, the etched shape for the Y-joint was unacceptable.

For the <110> wafer (Fig. 8), hydrazine provided the best results. Significant undercutting was observed for NaOH and the patterns were etched away when using KOH or EDP.

4.1.3 *Columns*

For the <100> wafer (Fig. 9), hydrazine gave the best results followed by EDP, in particular, for the 90°-cornered columns. With NaOH, the etched shapes were visible but poorly etched and, for KOH, they were significantly distorted or etched away.

For the <110> wafer (Fig. 10), hydrazine gave the best results for the 90°-columns but some of the corners lost their sharpness. The 45°-columns had some undercutting of the mask and the spiral column was etched away in two places. With NaOH, part of the 90°-column was etched properly but undercutting of the mask was noted in the inner part of the column and the 45°-columns and spirals were etched away in many places. With KOH or EDP, the columns were etched very poorly or were etched away.

4.1.4 *Polygons*

For the <100> wafer (Fig. 11), all the polygons etched in hydrazine retained their original geometrical shapes when hydrazine was used. The circle was the exception and was converted to an octagon. For NaOH, the circles, rectangles and squares retained their shapes, but significant undercutting was observed for the triangle. The pentagon and the heptagon were converted to octagons. With KOH, the polygons lost their original shape and formed new shapes, for example, the small-size triangle was converted to a rectangle.

The large-size triangle (top left-hand corner of Fig. 2) etched well in hydrazine (Fig. 12). Some undercutting of the mask and debris/pyramids was observed when using NaOH or EDP and the triangular shape was lost when KOH was used. The quality of the etched walls was examined using SEM photographs taken with a higher magnification: the vertical side had a smooth surface and the oblique side had a rough surface. The small-size triangle lost its original shape and became a rectangle (Fig. 12).

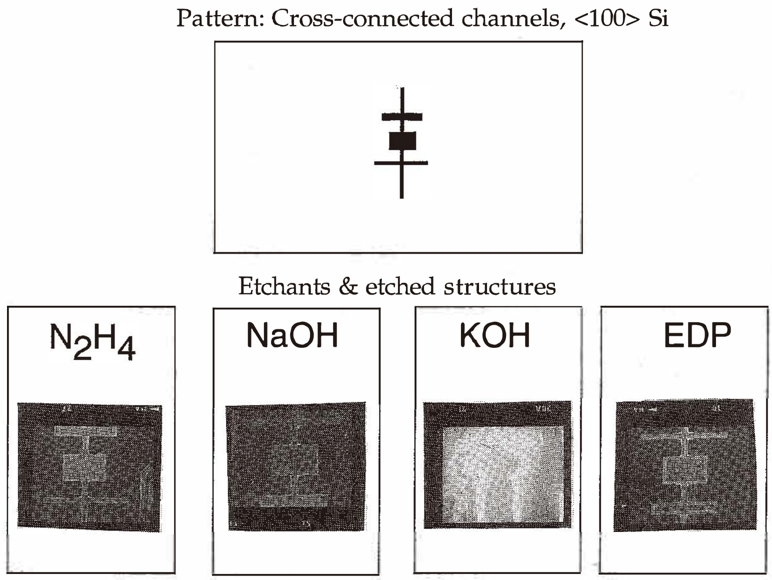

Fig. 5. Shapes obtained on <100> wafers after etching the patterns for the cross-connected channels.

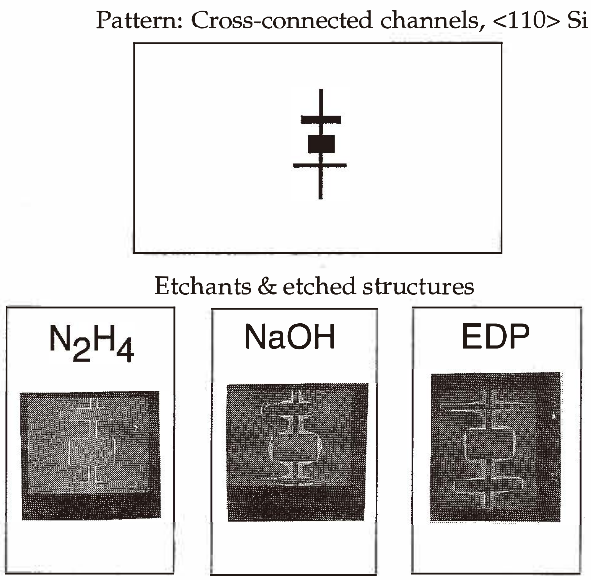

Fig. 6. Shapes obtained on <110> wafers after etching the patterns for the cross-connected channels (a shape was not observed when using KOH).

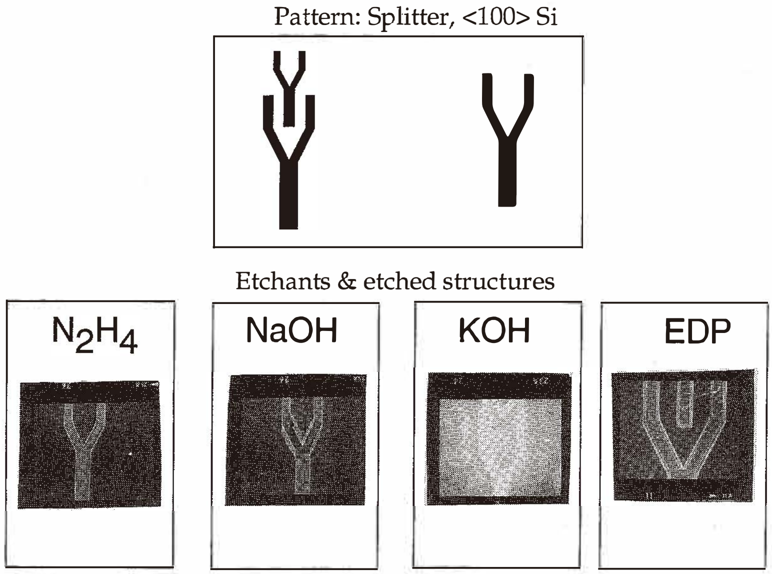

Fig. 7. Shapes obtained on <100> wafers after etching the patterns for the splitters.

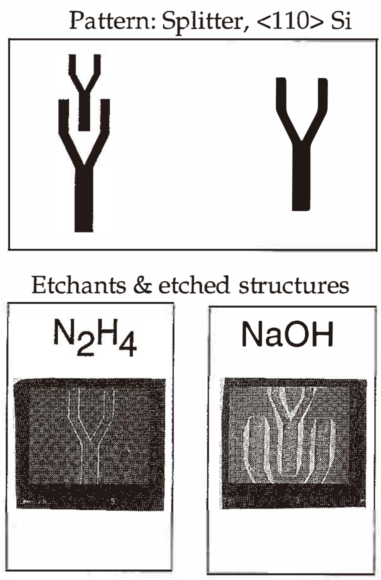

Fig. 8. Shapes obtained on <110> wafers after etching the patterns for the splitters (a shape was not observed when using KOH or EDP).

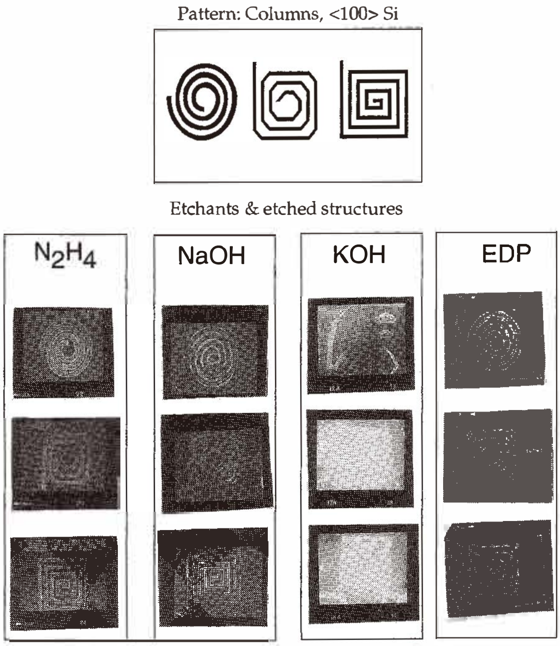

Fig. 9. Shapes obtained on <100> wafers after etching the patterns for the columns.

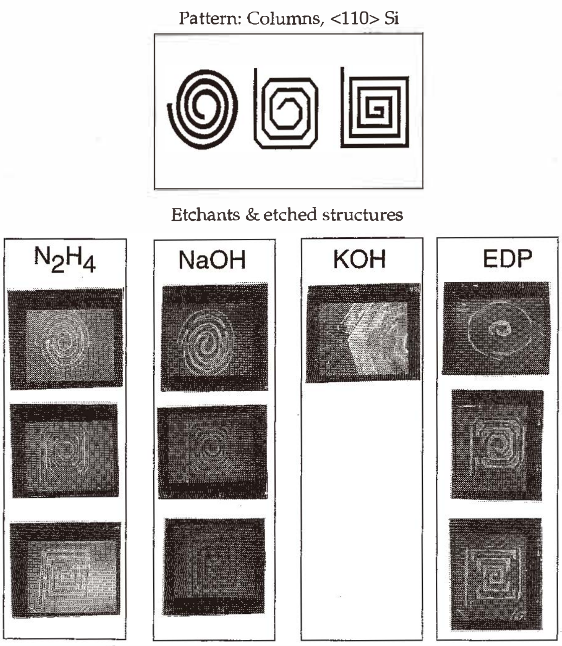

Fig. 10. Shapes obtained on <110> wafers after etching the patterns for the columns.

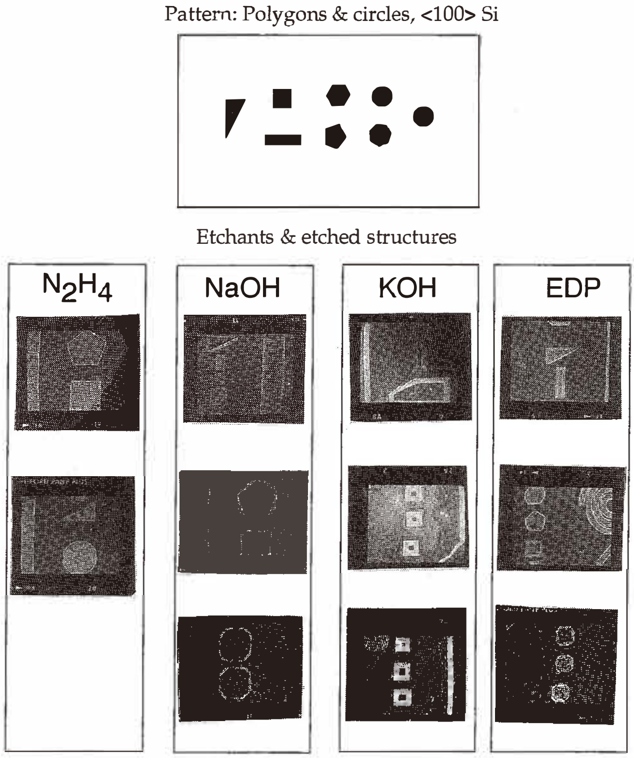

Fig. 11. Shapes obtained on <100> wafers after etching the patterns for the polygons and the circles.

For the <110> wafer (Fig. 13), hydrazine gave the best results and even the circle almost retained its shape. With NaOH or KOH, the original geometry changed significantly, for example, the small-size triangle lost its shape. With EDP, significant undercutting of the mask was observed.

For the larger-size triangle, sharp edges and well defined corners were obtained when using hydrazine (Fig. 14). With all the other etchants, significant undercutting was observed and, for the small-size triangle, a new shape was obtained.

4.1.5 *Simplified ICP torch*

This pattern (Fig. 2) is a combination of some of the simpler geometrical variants discussed above and, for the <100> wafer (Fig. 15), only hydrazine gave an acceptable etched shape.

For the <110> wafer (Fig. 16) and similar to the <100> orientation, only hydrazine gave an acceptable etched shape.

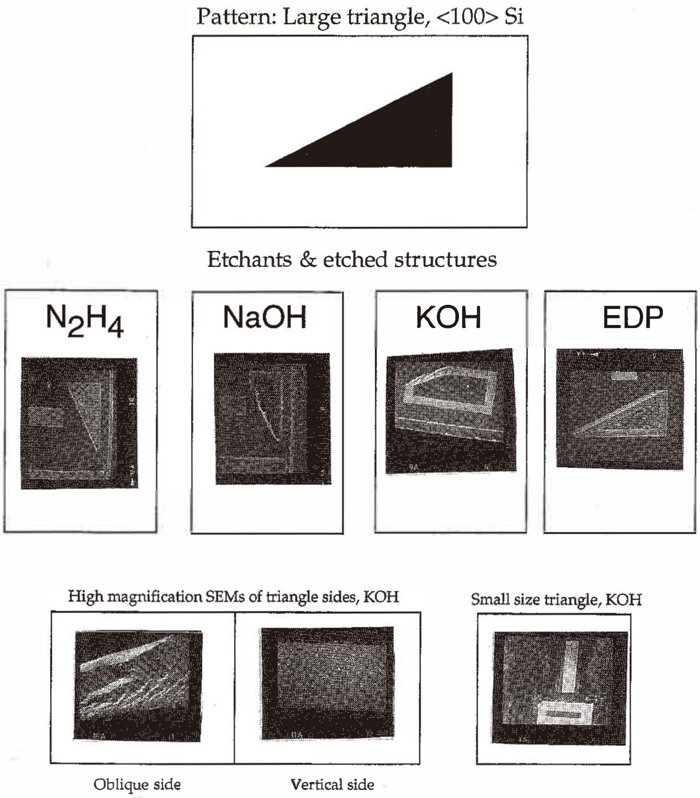

Fig. 12. Shapes obtained on <100> wafers after etching the patterns for the large-size and the small-size triangles.

4.2 *Agitation effects*

The effect of stirring was also studied, albeit briefly, and <100> wafers were etched using NaOH. As expected, agitation had a significant effect on the etched shapes (Fig. 17). Without agitation, interconnected channels showed sharp, 90°-corners (Fig. 17(a)) but, with agitation, the 90°-corners were destroyed and the center of the interconnected channels was converted to a star-like shape (Fig. 17(b)). However, not all 90°-corners were destroyed. For example, stirring had a small effect on the squares with 90°-concave corners (Figs. 17(c) and 17(d)), most likely because these corners are bounded by the <111> planes.

The circles retained their shape without agitation (Fig. 17(e)). With agitation, however and, depending on size, the circles became rectangles or polygons and an example is shown in Fig. 17(f).

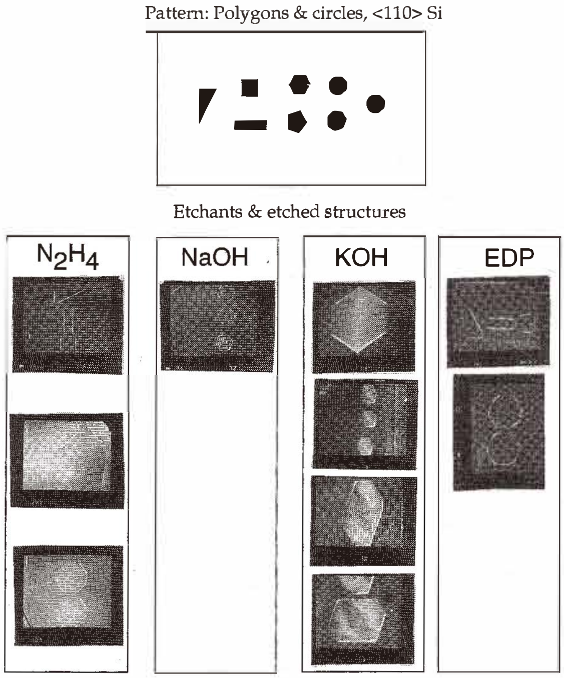

Fig. 13. Shapes obtained on <110> wafers after etching the patterns for the polygons and the circles.

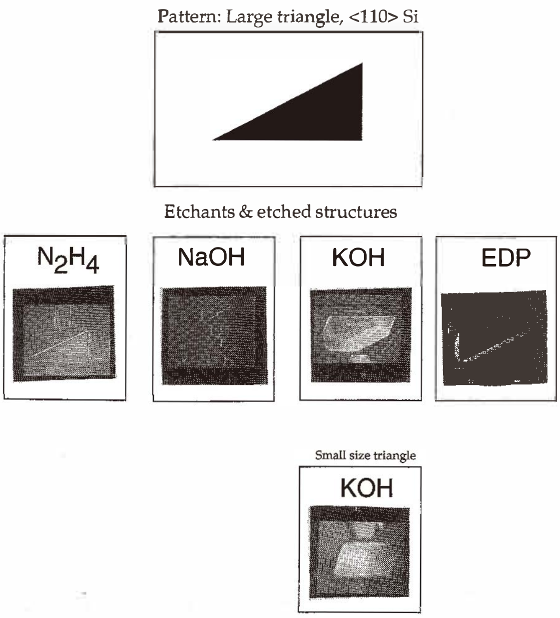

Fig. 14. Shapes obtained on <110> wafers after etching the patterns for the large-size and the small-size triangles.

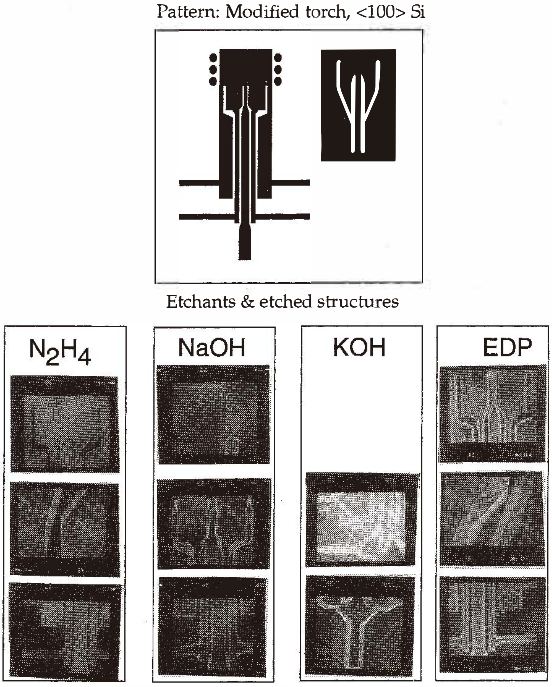

Fig. 15. Shapes obtained on <100> wafers after etching the patterns for the modified ICP torch.

5. Conclusions and Future Prospects

Similar to MEMS and microelectronics, experimental conditions have been found to exert a significant influence on the shape of etched patterns. In addition, the geometry of a pattern on a wafer was found to be an important variable in defining etched shapes and should be taken into consideration when micromachining structures for chemical analysis applications. Overall, it can be concluded that extensive experimentation is required before usable micromachined structures can be obtained.

The developments reported in this work may be used in mask design, for instance, for corner compensation, may help add an analytical chemistry component to a MEMS curriculum[85] and may aid in future miniaturization of analytical tools, such as, vials, beakers, sensors, instrument components, modules or even entire chemical analysis microinstruments. Even though such futuristic extensions are intellectually appealing and conceptually straightforward, their implementation may prove to be challenging. However, work continues[51,86,87] in our laboratory along these lines.

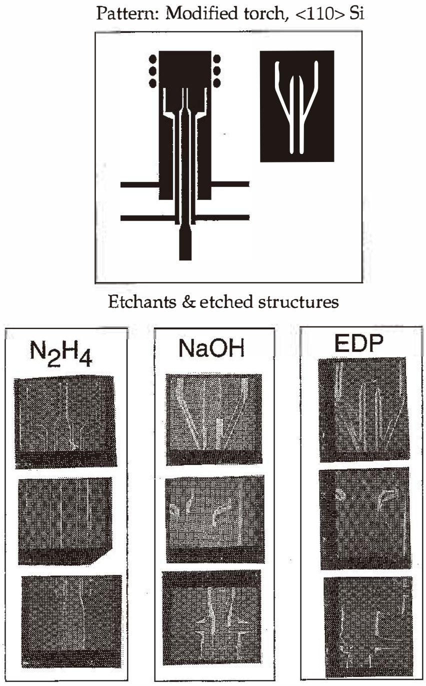

Fig. 16. Shapes obtained on <110> wafers after etching the patterns for the modified ICP torch (a shape was not observed when using KOH).

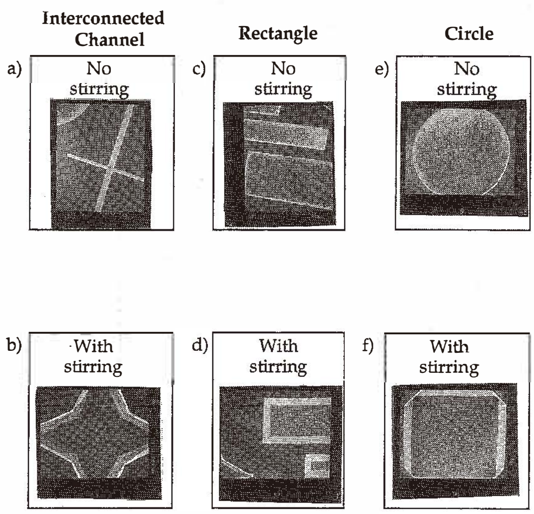

Fig. 17. Agitation effects (see text for discussion).

Table 2
Summary of etching results for the <100> Si wafer.

Patterns	N_2H_4	NaOH	KOH	EDP
Regular polygons	@@@	@@	×	@@
Triangle	@@	@	×	@
Cross-connected channels	@@@	@@	×	@
Column (spiral)	@@	×	×	@
Column (45°)	@@	@	×	@@
Column (90°)	@@@	@@	×	@@@
Splitter	@@@	@@	×	@@
Modified ICP torch	@@@	@	×	@@@

@@@: Excellent @: Poor
@@ : Good × : Unacceptable

Table 3
Summary of etching results for the <110> Si wafer.

Patterns	N_2H_4	NaOH	KOH	EDP
Regular polygons	@@	@@	×	@
Triangle	@@	@	×	@@
Cross-connected channels	@@	@	---	@
Column (spiral)	@	×	×	×
Column (45°)	@@	@	×	×
Column (90°)	@@	@@	×	×
Splitter	@@	@	---	@
Modified ICP torch	@@@	×	×	×

@@@: Excellent × : Unacceptable
@@ : Good ---: Not available
@ : Poor

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