

A Review of various Wet Etching Techniques used in Micro Fabrication for Real Estate Consumption

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ABSTRACT

In the 1980's science and technology have pushed towards miniaturization. In order to interface to the microscopic world, miniaturized structures are necessary to bridge the gap to the macroscopic world. Well established and developed methods of micro fabrication applications such as the fabrication of micro structures and circuits can used to micromachine structures in 3D indispensable to interface to the nano world. Micromachining has great significance industrial application such as the accelerometer that triggers air bags in cars and mass storage devices. Bulk materials in micro fabrication are shaped into microstructures using lithography and etching. Different kinds of chemical solutions including acids and bases were used to selectively remove significant amount of silicon from a silicon wafer by chemical reaction of the silicon with the etchant could formed micro structures. Because of the undercutting, etched pits are usually quite different forms the mask at the corners of the structures. The undercutting of convex corners can be compensated by designing suitable mask. By using compensation methods sharp corners can be constructed. Different research group proposed and demonstrated various corner compensation designs using KOH, EDP and TMAH solution. Although other methods have also been reported, they use extra mask and processing steps, which make them more expensive and complex.

Keyword

Micro fabrication, Anisotropic etching, SEM

1. INTRODUCTION

Micro fabrication etching techniques

Micro fabrication techniques uses wet etching for chemically removing layer from the surface of the water. Fundamental etching techniques used in micro fabrication are wet etching (liquid – phase) and dry etching (plasma phase). Dry etching have several disadvantages such as some gases are quite toxic and corrosive. It requires redeposition of non-volatile compounds and it needs specialized and expensive equipment. In comparison, wet etching which is inexpensive has been extensively used for the fabrication of MEMS components such as diaphragms based on single crystal silicon, especially on (100) water and cantilever beam. Key technique for the fabrication of various micromechanical devices is anisotropic etching. The etchrate (ER) in anisotropic etching is much faster in etching plane as etch time progress. Usually the (111) planes of silicon. The important factor of anisotropy etching includes selectivity, handling and process compatibility and anisotropic. Anisotropic wet etching produces a typical etch rate about 1µm/min.

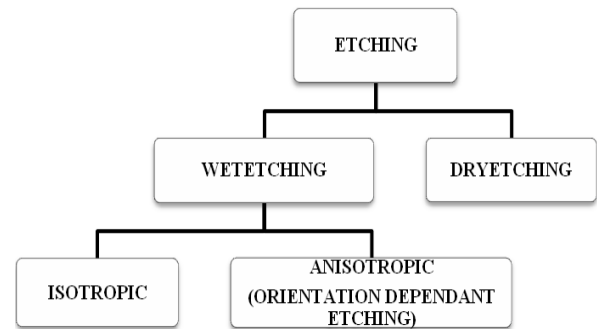


Fig 1: Classification of etching

The important agents used for anisotropic wet etching are potassium hydroxide (KOH), Ethylenediamine pyrocatechol (EDP) and tetra methyl ammonium hydroxide (TMAH).

Table 1. Comparison of Wet Etching and Dry Etching

	Wet Etching	Dry Etching
Method	Chemical Solutions	Ion Bombardment or Chemical Reactive
Environment and Equipment	Atmosphere, Bath	Vacuum Chamber
Advantage	1) Low cost, easy to implement 2) High etching rate 3) Good selectivity for most materials	1) Capable of defining small feature size (<100 nm)
Disadvantage	1) Inadequate for defining feature size < 1µm 2) Potential of chemical handling hazards 3) Wafer contamination issues	1) High cost, hard to implement 2) low throughput 3) Poor selectivity 4) Potential radiation damage
Directionality	Isotropic (Except Crystalline Materials)	Anisotropic

Table 2. Comparison of Isotropic and Anisotropic etching

Isotropic Wet Etching	Anisotropic Wet Etching
<ul style="list-style-type: none"> Etch occurs in all crystallographic directions at the same rate. Most common formulation is mixture of hydrofluoric, nitric and acetic acids (“HNA”: HF + HNO₃ + CH₃COOH). Etch rate may be very fast, many microns per minute. Masks are undercut. High aspect ratio difficult because of diffusion limits. Stirring enhances isotropy. Isotropic wet etching is applicable to many materials besides silicon. 	<ul style="list-style-type: none"> Etch occurs at different rates depending on exposed crystal Usually in alkaline solutions (KOH, TMAH). Heating typically required for rate control (e.g. > 80 °C). Etch rate typically ~1µm/min, limited by reactions rather than diffusion. Maintains mask boundaries without undercut. Angles determined by crystal structure (e.g. 54.7°). Possible to get perfect orthogonal shapes outlines using 1-0-0 wafers.

2. SILICON ANISOTROPIC ETCHING IN ALKALINE SOLUTIONS

The etching rate of individual planes defines the structures and their interdependence has its own importance. The etching solution are chosen by considering many factors, such as selectivity of silicon dissolution with respect to silicon dioxide, roughness of the etched surface, anisotropy and etching rate. The resulting surface roughness of the structure is important in determining the components that must be fabricated and applied to microfluidic systems [1] and MEMS structures. Of all the alkaline anisotropic [5-8] etchants used, the inorganic KOH (potassium hydroxide) and organic TMAH (CH₃)₄NOH (Tetra methyl ammonium hydroxide) solutions are the most commonly used. The alkaline KOH [20] etching technique has been used by many research groups to realize the MEMS structure. It is safe, easy to handle, repeatable and etches fast. A key superiority of KOH is low cost, which is fit for batch fabrication. So KOH anisotropic etching has become one of the key technologies in silicon bulk micromachining. KOH etching results in uniform and bright etched surface in a near saturated solution at 80°C. Etching rate of the (1 0 0) silicon plane is 1.4 µm/min at 80°C in 50 wt. % KOH solution [1]. However, the condition of etching and non-uniformity of the etching rate becomes considerably worse above 80°C. However, after aluminum metallization silicon devices cannot be micromachined in KOH solution without proper protection of the metal line. Moreover KOH is not CMOS compatible due to the presence of alkali metal ions in it. Aqueous TMAH solution is compatible with CMOS fabrication process with excellent etching characteristics in terms of etch rate & surface smoothness and low toxicity levels. TMAH can be handled easily and safely and exhibits excellent selectivity to silicon oxide and silicon nitride masks. Slow etching rate of TMAH

and the tendency of formation of pyramidal hillocks are some of the problems causing rough surface. Hydrogen bubbles govern the distribution and population of the hillocks [19] during silicon etching. “Pseudo-mask” phenomenon are caused by these bubbles which annihilate the chemical reaction between the etchant and the silicon atoms at the surface, and increases surface roughness.

The chemical properties variation and modification of etchants by external agitation will detach hydrogen bubbles [1] from the etched surfaces and helps in improving the smoothness and etch rate. Some recent methods of includes ultrasonic agitation to evaluate the wet etching properties of silicon. The method of ultrasonic agitation is very efficient way of achieving a higher etching rate, a uniform etching depth and a smooth etched surface over the whole wafer, but fabricating membrane microstructures using ultrasound is difficult. The structures can be easily damaged by the additives which includes various alcohols, redoxsystem, oxidizing agent and different ion-typed surfactants, have been put into the KOH solutions, and isopropyl alcohol (IPA), IPA-pyrazine system, potassium ion (K₂CO₃), strong oxidizer ammonium persulfate (APS), water glass (WG), non-ionic surfactant and catalyst, have been added to etchants to improve the etching anisotropy, to reduce undercutting or promote the roughness and etching rate. Additives like IPA are used to improve the smoothness quality of the (1 0 0) silicon plane etching and reduce the undercutting of convex corners, but causes decrease in the etch rate in some other crystal plane particularly with higher indices than (1 0 0).

Till now additives used for etching have been selected by trial and error method and the effect and role of them on the anisotropic etching are not yet fully understood. Electroforming procedure are usually used to improve the electrolyte wettability and minimize the adhesion of hydrogen bubbles to the cathode’s electroformed surface. Electrolyte which contains the wetter has the ability to wet the electroformed microstructure surfaces and equip electrolyte to penetrate into deep and narrow patterns. Based on this concept of a using wetters, three on-typed surfactants are commonly used as electroforming agents, thus KOH and TMAH solutions are mainly applied in the anisotropic silicon etching process. IC processing uses mostly the etching characteristics of anionic and cationic surfactants added to TMAH solutions in accordance to the MEMS device fabrication. The etching properties such as etching rate of the (1 0 0) silicon plane and undercutting of convex corners are given more priority during the the selection of the additive [6].

3. ETCHING CHARACTERISTICS OF DIFFERENT ALKALINE SOLUTION

3.1 KOH solution

22 wt. % KOH solution provides maximum silicon etch rates of 89.2 and 88.1 µm/h for n-type and p-type silicon respectively at 80 °C [2]. The dopant type of silicon substrate has little effect on the etch rate of silicon although n-type etches slightly faster than p-type silicon. The silicon dioxide etches rate increases continuously with increase in temperature irrespective of KOH solution concentration. The maximum silicon dioxide etch rate is 450 nm/h at 80 °C using 33 wt. % KOH. The Al etch rate is appreciable in all KOH concentrations with maximum etch rate of 3.0 µm/min. The etched silicon surface smoothens with both increase in KOH concentration and bath temperature [9]. Silicon surface

roughness degrades with increase in etch duration due to the masking of hydrogen bubbles evolved during etching which significantly contributes to surface roughness.

3.2 TMAH solution

The maximum silicon etch rate obtained is 60.2 $\mu\text{m}/\text{h}$ using 3% TMAH at 80 °C. The silicon etch rate decreases with increase in TMAH concentration [8]. Similar to KOH, dopant type of silicon substrate type has negligible effect on silicon etch rate. The silicon dioxide etches rate increases with both increase in temperature and decrease in TMAH concentration. The maximum silicon dioxide etch rate value is almost half in TMAH compared to KOH which is an attractive feature of TMAH. Undoped TMAH solution attacks aluminum film but to a less extent than KOH. The maximum aluminum etch rate is 1.44 $\mu\text{m}/\text{min}$ in 20% TMAH at 80 °C. The etched silicon surface smoothness increases with both increase in TMAH concentration and temperature. The random pyramidal hillocks formed during etching significantly contribute to the surface roughness than other factors. The maximum and minimum silicon roughness values are 3.7 and 0.04 μm which are obtained using 3% TMAH and 20% TMAH, respectively.

3.3 Dual doped TMAH solution

The etch rate of silicon in dual doped TMAH is almost comparable to that of KOH and TMAH but the masking silicon dioxide and aluminum etch rates are quite small which is desirable for MEMS fabrication technology. Dual doped TMAH [2] also improves surface smoothness of the etched surfaces due to the suppression of random hillock formation by the oxidizing agent AP. The silicon surface roughness is three orders less in dual doped TMAH solutions in comparison to KOH and TMAH.

4. CONVEX CORNER UNDERCUTTING WITH DIFFERENT ETCHANTS

The etch rate of individual crystal planes of silicon defining the structure and as well as their interdependency are critical parameters which not only delineate the geometrical shape but also influence the functionality of the microstructure. Convex corner undercutting of crystalline silicon during fabrication of rectangular / square shaped or any right angled edge in a micro structure using anisotropic wet chemical etchant in single crystal silicon is a prime but undesired phenomenon. The undercut ratio depends on several parameters like type of the etchant and its composition, temperature of the etchant, stirring, alcohol additives, surfactants etc. Corner compensation structure can minimize or even completely prevent the corner undercutting and can produce perfect corner. However, this technique requires significant area in the mask layout to accommodate compensation structures resulting in increase of real estate consumption. On the other hand, addition of surfactants and alcohol additives reduced the corner undercutting at the cost of the anisotropy of the etchant.

Generally, convex corner undercut obtained in the first stage decreases with time in the following second stage (KOH and KOH+IPA system) [7] or is at least mitigated (TMAH and TMAH+IPA system). The degree of undercutting is dependent on several factors, such as the etch rate of the specific crystal plane, which is in turn a consequence of the etchant in use, bath temperature, solution concentration and to a lesser extent to stirring conditions. Different samples were first prepared in mask etching mode, having different depths of ridges and undercuts are experimentally determined the

etch rate and can compete and overcome the characteristic undercutting rate of the convex corner which has occurred in conventional micromachining. This is particularly true in the KOH system (which has by default lower initial undercutting compared to TMAH). If the initial undercut is too severe (etched too deep in mask mode without one of the known convex corner compensation [16] schemes) then self-compensation cannot be achieved at the end of the required etching depth. In this case applying masks with additional convex corner compensation structures is mandatory.

5. MASKLESS ETCHING USING VARIOUS SOLUTION

5.1 Maskless etching in KOH

In many KOH anisotropic etching, the central fast etching planes gradually step to (100) planes when the pattern is etched, but the free end is etched as normal. This phenomenon is useful to compensation structure design. If the compensation is finished with (110) fast etching planes, complete convex corner will be formed. The gradual fast etching change cannot be easy calculation. The mechanism is unknown. But in simple calculation, it is acceptable to assume that the fast etching planes abruptly change to (110) planes when half of the pattern is undercut. This assumption is absolutely experiential.

Initial mask etching was performed with KOH at time intervals of 10 min, samples were withdrawn and undercut, ridge width (lateral shrinkage) and depth were monitored under the optical microscope. studies shows a decreasing trend of convex corner undercut meaning roughly that the etch rate dominates over the etch rate of planes exposed at the convex corner. Planes at the convex corner could not be clearly identified by SEM [17] as in the case of mask etching, but consisted of a disordered set of small facets with very smooth transition.

5.2 Maskless etching in KOH+IPA

From the literature and previous work it is known that IPA has a strong influence on etching performances of mask etching, particularly on etching anisotropy and consequently on undercutting of convex corners. The rate of undercut increases up to 20% compared to KOH and varies between 0.83 and 1.17 $\mu\text{m}/\text{min}$. No dependency of initial depth versus undercut behaviour could be observed. Self-compensation was achieved at times between 20 and 30 min depending on the initial undercut. Also, more detrimental undercuts can be compensated if allowing a prolonged etching, but it has to be mentioned that after prolonged maskless etching some rounding can occur at the bottom transition level. This was also observed and reported that this is due to the local etch minimum appearing between these two adjacent etch planes.

5.3 Maskless etching in TMAH

For maskless experiments, an equal temperature of 80°C for all etchants was utilized. As shown in some previous papers, TMAH has a significant undercut of convex corners compared to KOH. Therefore the initial undercuts after mask etching that served as starting points for maskless etching were severe. The undercutting is mitigated to a great extent, but cannot be compensated for a very wide range of conditions. Actually, self-compensation can be achieved for small initial undercuts and long maskless etching times as shown for 5 min initial mask etching that corresponds to a depth of 2.7 μm and

undercut of 20 μm . After onset of etching the undercut [17] increases with the same slope as the above curves do, levels off and then, after 20 min, starts to decrease slowly. This is a peculiarity that was not expected considering the positive slope of the undercut behavior determined for the times longer than 5 min.

5.4 Maskless etching in TMAH+IPA

It is known from the literature that IPA reduces the degree of undercutting in mask etching mode, i.e. changes the etch rate ratio of fast etching corner planes toward rates of (100) and (111) planes. A very slight increase of undercut being of the same order as gained by TMAH, with the exception at the low initial undercuts. No compensating trends were observed in the range of chosen parameters and the undercutting rate was 0.29–0.3 $\mu\text{m}/\text{min}$. In general, the undercut decreases with KOH and KOH+IPA [7] solution and increase in TMAH and TMAH+IPA solutions were determined with the exception mentioned before.

6. ORIENTATION DEPENDANCE OF KOH AND TMAH

A solid hemispherical specimen of single crystal silicon of radius 22 mm, and its sphericity was less than 10 μm was used to evaluate the orientation-dependent etching rate [11–15]. The surface was polished to a mirror finish. The surface roughness was 0.005–0.007 μm in the arithmetical average. All crystallographic orientations appeared on the hemispherical surface. The profile was measured using a 3-D measuring machine. The surface profile was probed every 2° of latitude ranging from 20° to 90° and every 2° of longitude ranging from 0° to 360°. Etching conditions such as temperature and concentration are varied.

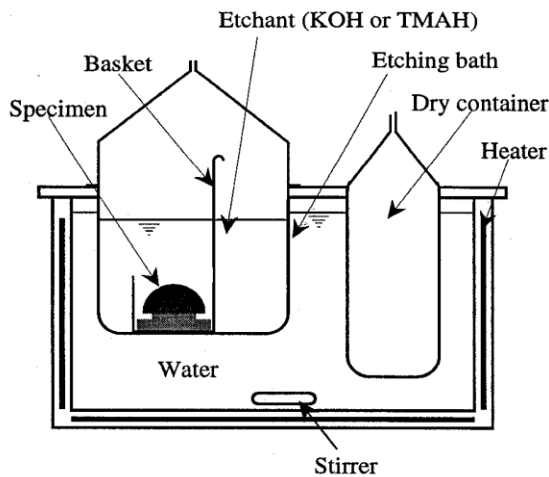


Fig 2: Experimental set up for etching

Although the values [9] in Table 3 varied with etchant concentration and etching temperature, there were difference between KOH and TMAH solutions. The orientation dependence was quite different for the (111).and (221).planes in TMAH and KOH solutions. This suggests that there are differences in the etching mechanisms of the two etchants in terms of crystallographic orientation. The etching rate ratio of (100)/(111) in TMAH was about half that in KOH. The effects of etchant circulation can be ignored for KOH, but not for TMAH solutions. The roughness of etched (100) surface, which is the smoothest of all the orientations, was 0.01 μm

with KOH and 0.4 μm with TMAH solutions. The roughness increased as the etchant concentration decreased.

Table 3. Etching properties of KOH and TMAH solutions

	KOH(34.0wt.%, 70.9°C)	TMAH(20wt.%, 79.8°C)
Etching rate	[μm]	[μm]
(100)	0.629	0.603
(110)	1.292	1.114
(111)	0.009	0.017
Etching rate ratio	[--]	[--]
(100)/(110)	0.49	0.54
(100)/(111)	74	37
(110)/(111)	151	68
Uniformity in etching rates on a hemisphere among four (110)s at the periphery	[%]	[%]
	<1.3	<9.4

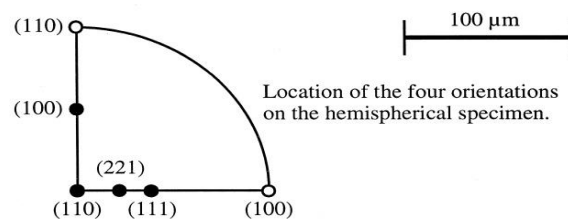
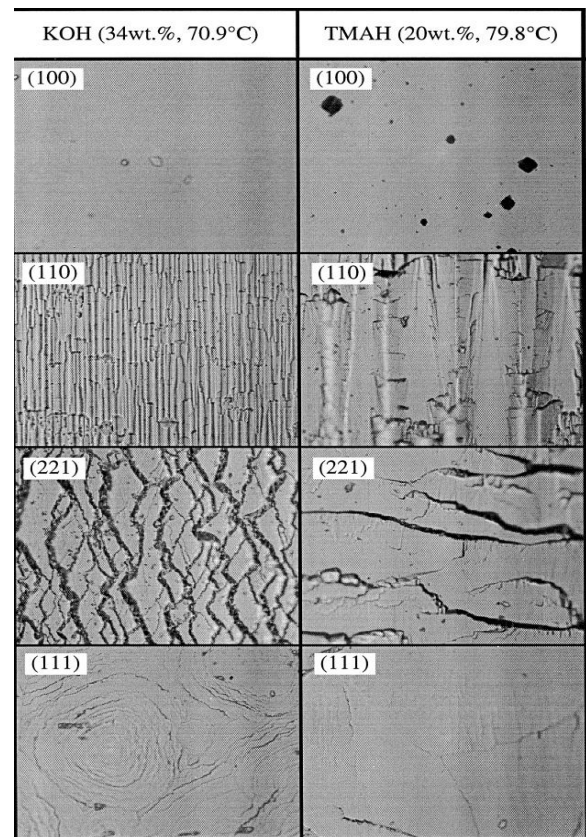


Fig 3: Comparison of the morphologies at four orientations of the surface etched in KOH and TMAH solution.

7. TMAH SOLUTION FOR ANISOTROPIC ETCHING OF SILICON

Anisotropic silicon etching is a key technique for the fabrication of various micromechanical devices. The important properties of anisotropic silicon etchants are anisotropy[18], selectivity, handling and process compatibility. The process compatibility becomes especially important, since sensors and actuators need to be utilized within circuits on the same chip to realize a new transducer system with high performance. Tetramethyl ammonium hydroxide (TMAH, $(\text{CH}_3)_4\text{NOH}$) [4] solutions with various concentrations from 5 to 40 wt % and temperatures can be used for wet anisotropic etching purpose. The main feature of TMAH is its compatibility, since it has been utilized in the etching process as the developing solution of positive photoresist. Therefore, semiconductor-grade solutions can be easily obtained. The dependences of the etch rates of (100), (110) and (111) crystal planes on temperature and concentration, the selectivity to SiO_2 and Si_3N_4 , the dependence of aluminum etch rates on the amount of dissolved silicon, the dependences of polysilicon etch rate on boron concentration and the electrochemical etching characteristics can also be evaluated. Experiments show that etch rates decrease with increasing concentration. At 5 wt %, the etched surface was sometimes covered with pyramidal hillocks and the etch rate became very low. As the concentration increases, the smoothness of the etched (100) surface changed drastically. At 5 wt %, the surface was covered with pyramidal hillocks having high densities. With an increase in concentration from 5 to 15 wt %, the density of the hillocks decreased and smaller hillocks were obtained. The hillocks [16-20] were bounded by (111) crystal planes. Above 22 wt %, a very smooth surface was obtained. The measured roughness for the 22 wt % solution was within 100 nm. There is no significant dependence of roughness on temperature. The dependences of the (110) etch rates shows a similar tendency to those of the (100) plane. The etch rates are higher than those for the (100) plane. As the concentration increases, the smoothness of the etched (100) surface changed.

Etching of silicon(110) surface revealed that at 10 wt %, the surface was covered by an irregular pattern and a ridged pattern in the $\langle 110 \rangle$ direction. With an increase in concentration from 5 to 22 wt %, the roughness became smaller and rather a smooth surface was obtained at 30 wt %.

The aluminium [14] etch rate shows that an increase in the amount of dissolved silicon, the aluminium etch rate decreased. At more than 40 g/l dissolved silicon, the aluminium etch rate decreased rapidly. An aluminium etch rate of 0.01 was obtained for 67 g/l of dissolved silicon.

The relative etch rate of boron decreases with increasing boron concentration above approximately $1 \times 10^{19}/\text{cm}^3$. The rate of decrease of the relative etch rate depends strongly on the concentration of the etching solutions. In the solution with a boron concentration of $4 \times 10^{20}/\text{cm}^3$ and a TMAH concentration of below 22 wt %, a relative etch-rate ratio of below 0.01 value.

8. CONCLUSION

Bulk micromachined piezoresistive accelerometer developed by several research groups have mostly used potassium hydroxide(KOH) and TMAH based wet etching. KOH wet etching technique has been used by many research groups to

realize the MEMS accelerometer structure. However, after aluminum metallization silicon devices cannot be micromachined in KOH solution without proper protection of the metal line. Moreover KOH is not CMOS compatible due to the presence of alkali metal ions in it. Aqueous TMAH solution is compatible with CMOS fabrication process with excellent etching characteristics in terms of etch rate & surface smoothness and low toxicity levels. One of the major disadvantages in using a wet anisotropic etching based micromachining process is convex corner undercutting of square/rectangular structures.

The etchants, modified by adding various ion-typed surfactants and non-ionic to KOH and TMAH solutions, were used to evaluate the etching properties under various operating parameters including the etching rate and roughness quality of the (1 0 0) silicon plane, the selectivity of silicon dissolution toward silicon dioxide and reduction of the undercutting at convex corners.

Compensation by (100) oriented beams provides sharp convex corners in pure 25% TMAH, while in the case of low concentration (10% or 20%) (110) planes are exposed at the corners between the bottom (100) and sidewall (010) planes. In the case of surfactant added TMAH, it is difficult to make sharp edge convex corners using the corner compensation method.

The etch rates of (100) and (110) decrease with increasing concentration. As the concentration increases, the roughness of the etched surface is reduced and a very smooth surface is obtained above 22 wt %. Etch rates of 10 gm/mm for the (100) plane and 14 gm/mm for the (110) plane at 90 °C were obtained using a 22 wt % solution.

The etch rates of aluminium were reduced by dissolving silicon in TMAH solution. It was confirmed that the etch-stop techniques using a heavily boron-doped layer or p-n junction were applicable to TMAH solutions. It can be concluded that TMAH is a promising solution for silicon micromachining as one of the useful etchants having good compatibility.

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