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Scalloping removal on DRIE via using low concentrated alkaline solutions at low temperature

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ABSTRACT

The scalloping removal on the silicon via sidewalls after a Deep Reactive Ion Etching (DRIE) by the so-called STiGer process has been achieved using a low concentrated alkaline solution at low temperature. Post-etching to reduce sidewall roughness was carried out in both KOH and TMAH-based solutions where the mass concentrations varied between 2% and 5%. The influence of IsoPropyl Alcohol (IPA) addition and the behavior of alkaline mixtures at two different temperatures (10 °C and 22 °C) were also investigated. The production of silicon surfaces free of micro-pyramids (also known as hillocks) has gone through the optimization of every etching parameter. SEM pictures have qualitatively evidenced the scalloping reduction and the shapes evolution in time, whereas AFM measurements performed on the silicon via sidewalls have allowed a quantification of the smoothing effect of the alkaline solutions. The Root Mean Square roughness (R_q) of the reference (sample with scalloped sidewalls without smoothing treatments) was thus compared with the one for wet alkaline treated samples. The results demonstrate that the etched via sidewalls are 60 times smoother. This value is, to our knowledge, the most important smoothing effect described in the literature.

1. Introduction

Reactive Ion Etching (RIE) is a process based on the synergy of both chemical and physical actions to etch silicon. The chemical action is the injection into the reactor of gaseous fluoride containing molecules (e.g. SF_6), whilst the physical action is provided by the ion bombardment. Deep Reactive Ion Etching (DRIE) is a derived process of the RIE. The DRIE is commonly employed to directionally etch the silicon and leads to the formation of High Aspect Ratio (HAR) structures. We count two main techniques of silicon DRIE: the Bosch [1], the standard cryogenic [2] processes. The Bosch process is basically decomposed in two cycled steps. The alternative injection in the chamber of an etching gas (SF_6) and a polymerising one (C_4F_8) enables the fabrication of HAR structures. For the second technique, the temperature of the silicon substrate is decreased to values close to −100 °C to form a SiO_xF_y passivation layer on the sidewalls. This helps to strongly reduce the etching rate on the sidewalls and thus enhance the anisotropy [2]. The STiGer process is a cryogenic Bosch-like technique [3–5]. The substrate is cooled to cryogenic temperatures and exposed to cycles of isotropic etching steps (SF_6 plasmas) and passivation steps (SiF_4/O_2) plasmas [5]. Like the cryogenic process, a SiO_xF_y inhibiting film is formed on the sidewalls (during passivation cycles) and limits lateral etching. Due to the cyclic passivation steps, the robustness is enhanced in comparison with standard cryoetching, which makes the profiles less sensitive to temperature variations. In addition, the passivation layer desorbs when the substrate is heated back to room temperature. Thus, unlike the Bosch process, there is no need to clean the microstructures and the chamber walls after each process run.

The wide range of possible structures induced by the DRIE is the main reason of its success: the DRIE commonly enables the fabrication of MicroElectroMechanical Systems (MEMS) [1], optical micro-mirrors [6] or Through Silicon Via (TSV) [7]. The Bosch and the STiGer processes present some disadvantages, the most important is the production of scalloping. This corrugation of the HAR structures sidewalls is consecutive to the alternation of etching and passivation steps. For example, the optical properties of micro-mirrors are not fully optimized after the dry etching step because of this persistent scalloping [6,8]. In TSV technologies, the scalloping lessens the deposited layer homogeneity. In a large majority of applications, flat silicon sidewalls are preferred to corrugated ones.

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Many studies have already been performed to lessen the silicon sidewall roughness after DRIE. The wet etching, especially in alkaline solutions, remains the most employed smoothing technique. Every common alkaline solutions i.e. potassium hydroxide (KOH) [6,8–12], Ethylenediamine (such as Ethylenediamine-Pyrocatechol and water (EPW) mixture) [13] and TetraMethylAmmonium Hydroxide (TMAH) [14,15] have already been tested. Furthermore, the addition of surface agents such as IsoPropyl Alcohol (IPA), ionic or non-ionic surfactants has also been studied as smoothing performance improver.

Alkaline solutions are known for a long time in the silicon industry thanks to their anisotropic etching properties. KOH solution is the most anisotropic silicon etching among chemical solutions, but is also banned from microelectronic industry because of potassium contamination of silicon. Nevertheless, this alkali hydroxide is still intensively studied for its notable etching behaviors. EPW is a hazardous mixture and thus rarely employed despite its good etching performances. TMAH is the anisotropic etching chemical commonly used in the microelectronic industry for about 20 years. TMAH based solutions provide smooth surfaces [16], relatively high anisotropy [17–19] and an interesting high silicon/silicon dioxide etching selectivity compared to KOH [20].

Each of these solutions was studied decades ago but only at high temperature (>60 °C). The temperature is known to ensure a higher etching rate, thus limits the etching time. Nowadays, thanks to the evolutions of photolithography and DRIE techniques, the etched structures become much smaller and via sidewalls become smoother [21]. In a near future, highly concentrated alkaline solutions carried out at high temperature will not be adapted any more, because they imply shorter etching durations and thus repeatability issues (e.g. for the emerging nano-devices technology). To reduce the roughness of electrochemically etched macroporous silicon arrays, Lehmann has proved that low concentrated KOH solutions mixed with IPA produced smooth surfaces at low temperature [22].

In this paper, we study and compare the effect on scalloping of low concentrated KOH or TMAH solutions at low temperatures. We exhibit the significance of IPA addition for HAR via smoothing. The via shape evolution (i.e. anisotropic behavior of alkaline solutions) is also observed. Finally, SEM and AFM analyses are performed to quantify the smoothing of the via sidewall.

2. Experimental

For the different experiments, 500 μm thick, (1 0 0) orientation, single side polished 6″ silicon wafers were used. First, 1.2 μm thick of silicon dioxide (SiO_2) was grown on the silicon wafer by wet thermal oxidation. After a photolithographic step, round shaped via with several diameters were formed using an industrial etching tool dedicated to cryo-etching. The configuration of the Alcatel 601E DRIE (inductively coupled plasma) equipment is described elsewhere [5]. As illustrated in Fig. 1, the scalloping height is about 500 nm maximum at the via opening repeated every 1.4 μm.

Prior to the wet etching, the samples were dipped into a HF solution (50 vol.%) and immediately rinsed in deionized water in order to completely remove the SiO_2 from the surface. The oxide was removed to avoid any underetching during the alkaline etching. The different etching solutions were prepared from 2 or 5 wt.% KOH or TMAH mixed with either 10 or 20 wt.% of IPA. Two etching bath temperatures were also studied: 10 °C (±1 °C) or ambient temperature (i.e. 22/23 °C). The alkaline solutions were not stirred during the etching process. After the etching step, the samples were rinsed with deionized water.

The results were observed by SEM (top, tilted and sliced views of via) to determine the evolution of the holes shape and the smoothing effect. The silicon samples were observed after 1, 2, 4 and 6 h of immersion in the different solutions. The samples presenting the smoothest sidewalls were then analyzed by AFM to measure the roughness reduction regarding DRIE via. Before every AFM measurement, the etched silicon samples slices were polished, cleaned by a Piranha solution (H_2SO_4 96 wt.% (2 vol.)/H_2O_2 30 wt.% (1 vol.)) during 10 min, dipped into a HF (50 vol.%) solution and then rinsed with deionized water to eliminate the polishing wastes.

3. Results and discussion

3.1. Via etched with the STiGer process

The 12 μm in diameter hole represented in Fig. 1 has been etched with the STiGer process. The etching and deposition cycle parameters (gas flows, pressure, source power, bias voltage, and time) have been carefully adjusted to get these vertical sidewalls. A depth of 55 μm has been reached within 10 min, which represents an average etching rate of 5.5 μm/min. This performance is typical of pulsed DRIE process for a final aspect ratio of 3 [23]. Moreover, no defect like bowing or extended scalloping can be observed as they appear for aspect ratios above 15–20. The profile only exhibits a standard scalloping of 500 nm due to the consecutive isotropic cavities etched during each etching cycle. It appears in Fig. 1 that the depth of the cavities and the ripples amplitude decrease with the depth of the via. It must be due to the Aspect Ratio Dependent Etching effect (ARDE effect) which involves a reduction of the etch rate with the depth leading to smaller cavities. It is not possible so far to avoid the appearance of this sidewall roughness with conventional DRIE techniques. It can only be reduced by, for example, decreasing the pulse time. In this last case, it depends on hardware capabilities.

The corrugation resulting of the dry etching (illustrated Fig. 1a and b) can be significantly reduced thanks to a soft and low temperature alkaline solution post-treatment. Several factors imputed to the anisotropic etching can influence the shape of the via and the quality of the silicon surface.

3.2. Behavior of low concentrated alkaline solutions at low temperature

As stated in the introduction, KOH or TMAH solutions containing a small amount of hydroxide ions have not been extensively studied because of the rough surface development at high temperatures. On the contrary, at low temperature the etching rate strongly decreases in these mixtures whereas the surface remains smooth. Besides temperature, the hydroxide ion concentration and the addition of IPA are the two other critical parameters affecting the (1 0 0) and (1 1 0) planes silicon etching rates. KOH in solution is known to etch Si{1 1 0} faster than Si{1 0 0} [24,25] whatever the hydroxide ions (HO^-) concentration. Unlike KOH, the (1 1 0)/(1 0 0) etching rate ratio into TMAH solution depends on the HO^- concentration. Below 15 wt.%, (1 0 0) tends to be etched faster than (1 1 0) plane [26,27]. This behavior has been observed during our experiments in 2 and 5% TMAH without IPA.

The addition of IPA is the main parameter affecting the via shape. Fig. 2 illustrates the evolution of a via shape with the immersion duration into a TMAH (5%)–IPA (10%). As we can see, the (1 1 0) planes are the remaining ones. We can conclude that the (1 1 0)/(1 0 0) etching rate ratio is lower than 1. This behavior has been observed into every (KOH or TMAH) alkaline solutions mixed with IPA. Moreover, these observations are coherent with the literature for highly concentrated alkaline solutions [27] which means that hydroxide ions does not inverts the (1 1 0)/(1 0 0) etching rate ratio even at low concentration. If IPA is added to the solution,