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Sub-50 nm High Aspect-Ratio Silicon Pillars, Ridges and Trenches Fabricated Using Ultra-High E-Beam Lithography and RIE

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We present the fabrication of sub-50 nm Si pillars, ridges, and trenches with aspect ratios greater than 10 using ultra-high resolution electron beam lithography and chlorine based reactive ion etching (RIE). A novel two step etching process involving RIE plus wet etching was developed to achieve 10 nm Si features. Furthermore, photoluminescence studies of these Si nanostructures are also presented.

1. INTRODUCTION

The ability to etch nanoscale features in Si is of great interest for trench isolation[1] and trench capacitors[2], and for novel ULSI quantum effect Si devices. Another attractive aspect of nanoscale Si etching is to study light emission which has been observed in porous Si. Techniques such as wet chemical etching are not suitable for etching nanoscale, high aspect-ratio structures due to undercutting of the mask and sloped sidewalls. Chlorine based RIE, however, is well suited for etching nanoscale features due the ability to control undercutting and etch profiles[3]. To date there have been a number of papers on Cl-based RIE of Si, but few discuss the issue of the ultimate scale to which structures can be patterned.

This paper focuses on fabricating sub-50 nm high aspect ratio Si pillars, ridges, and trenches using ultra-high resolution electron beam lithography and Cl based RIE. We found that with an optimized composition of Cl_2 and $SiCl_4$ we can readily and repeatedly achieve sub-50 nm diameter pillars 520 nm high at a pitch of 100 nm, and gratings with a spacing of 30 nm and a linewidth of 50 nm which are 520 nm deep. Subsequent wet etching in HF was used to remove the Si skin damaged during the RIE process and to passivate the surface for photoluminescence studies.

2. EXPERIMENT

The starting Si wafers, *p*-type with a 10 Ω•cm resistivity and a (100) orientation, were first cleaned using H_2SO_4:H_2O_2:H_2O (1:1:5) for 10 min at 120 °C, DI rinse for 5 min, and BHF:H_2O (1:9) for 30 sec. Then a single 70 nm thick layer of 950 K PMMA was spun on the sample and baked at 165 °C for 12 hours. The PMMA was exposed using a modified JEOL-840A SEM, described elsewhere[4], and developed in a mixture of 2-ethoxyethanol and methanol. Cr was then deposited via electron beam evaporation to a thickness of 50 nm at a rate of 1Å/sec. The residual Cr was removed using a lift-off process.

RIE was performed using a Plasma-Therm parallel plate RIE system operated at 13.56 MHz using mixtures of Cl_2, $Cl_2/SiCl_4$, and $Cl_2/SiCl_4/He$ gases. Chlorine was used because it has been shown to produce vertical sidewalls[3] due to the ion assisted etching mechanism[5], but has the drawback of producing trenches in the bottom corners. $SiCl_4$ and He were added to control trench formation by simultaneous re-deposition[6]. Prior to etching the chamber was always cleaned for 10 min with an Ar plasma, and then pre-conditioned for 10 min using the same etching recipe that was to be used. After inserting the sample, the chamber was pumped below 2×10^{-5} torr.

All samples were etched with the same Cl_2 and $SiCl_4$ flow rates, 76.6 and 13.3 sccm respectively, a power density of 0.32 W/cm^2, and a pressure of 40 mtorr. These parameters were found to produce the sidewall profiles necessary for high aspect ratio nanoscale features without trenching at the bottom corners. The He flow rate was varied from 0 to 60 sccm to further optimize the etch parameters. The substrate temperature during etching was maintained at 32 - 40 °C during etching. After etching, the samples were analyzed using high resolution scanning electron microscopy.

Previously, when etching with Cl_2 chemistries, the formation of roughened Si surfaces, "black Si", was reported[3], but the cause was unclear. Maluf[7], also using mixtures of Cl_2, $SiCl_4$, and He, only observed "black Si" with high He flow rates. We found that proper sample and chamber cleaning resulted in Si surfaces which have surface profile variations of only 10 - 30 nm, but larger surface features, pillars over 250 nm tall and ~50 nm in diameter, were observed at average densities of 50 pillars/100 μm^2. Etched Si surfaces were typically light brown in color.

A series of experiments in which the He flow was varied from 0 to 60 sccm was carried out to determine the effect of He flow on "black Si" formation, etch rate, and sidewall geometry. We found that the He flow in this range did not effect the Si surface or sidewall profiles. As is shown in figure 1, the etch rate was found to increase slightly with increasing He flow. Because the presence of He was not observed to effect the surface or sidewall profiles, our final etching recipe consisted of only Cl_2 and $SiCl_4$ using the previously mentioned parameters. The etch rate was measured to be 260 nm/min.

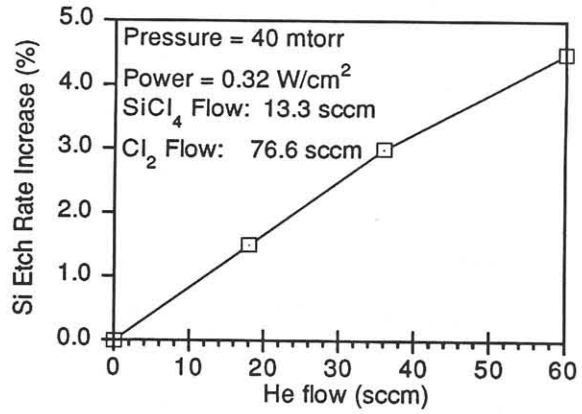

Fig. 1 Percent increase in Si etch rate versus He flow.

HF acid was used to further reduce the size of the RIE etched features. HF was found to etch Si at a rate as slow as 1.9 nm/hr, allowing excellent control in etching nanoscale Si structures, and providing the advantage of passivating the Si surface[8]. Photoluminescence studies were performed on some of the etched Si pillar arrays using a pulsed Nd^{3+}:YAG with a wavelength of 532 nm and an average intensity of 5.7 x 10^7 mW/cm^2.

3. RESULTS AND DISCUSSION

Numerous experiments were conducted to determine the ultimate resolution that could be achieved using this RIE recipe. Si pillars, trenches and ridges which are smaller than that previously reported have been achieved. Figure 2 shows an array of etched Si pillars having diameters of about 40 nm, a period of 100 nm, and a height of 520 nm. The hemispherical Cr dots were not removed for this picture; no undercutting of the mask or trenching at the bottom corners has occured. Figure 3 shows 50 nm wide Si ridges spaced by only 30 nm which are also 520 nm high. The Cr mask has been left in place to again verify that no undercutting of the mask occured during etching. In both cases the aspect ratio is in excess of 10.

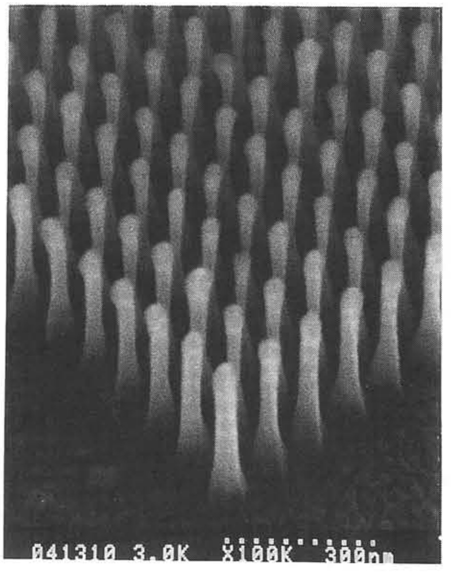

Fig. 2 Sub-40 nm diameter Si pillars with a period of 100 nm and height of 520 nm.

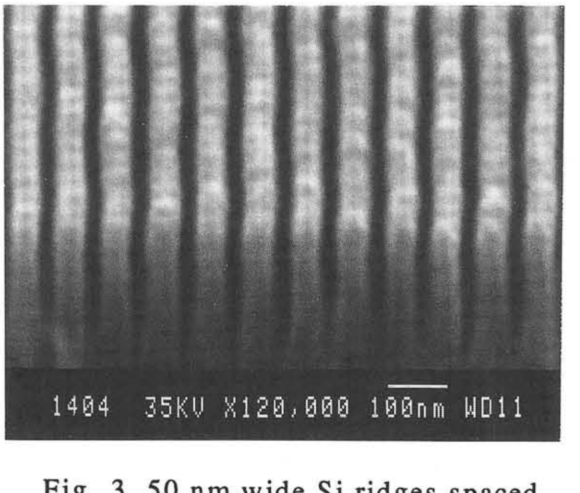

Fig. 3 50 nm wide Si ridges spaced by 30 nm with a height of 520 nm.

It is clear that highly uniform sub-50 nm features with near vertical profiles can be achieved using this technique. These results suggest that the ultimate limit on etched feature size is determined by the ability to pattern nanoscale masks and the fundamental mechanical stability of Si, rather than on the etching mechanism itself.

Figure 4 shows the same pillar array shown in figure 2 after etching in HF acid for 10 hours. The Cr mask was removed prior to HF etching. The columnar structures have been transformed into pyramids with sharp tips less than 10 nm wide. The resulting pyramids with four equal facets indicates that the etching is crystallographically anisotropic, with the [111] planes having the slowest etch rate. Such etching characteristics might be very useful for optical waveguide fabrication.

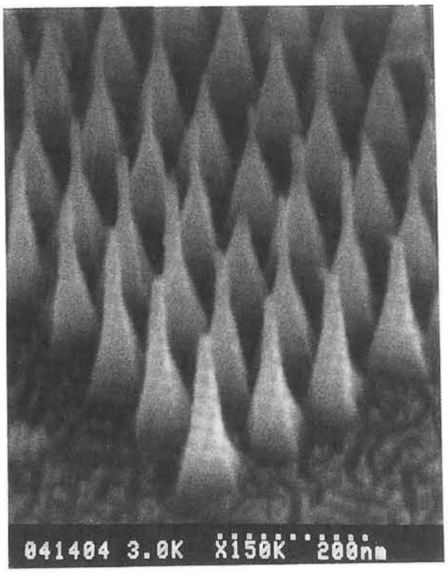

Fig. 4 Same pillar array shown in fig. 2 after etching in HF for 10 hours.

Photoluminescence studies have been performed on etched Si features identical to those shown in figure 4. Samples were illuminated with the 532 nm laser source. Light emission was checked for by using a visual technique with a 532 nm bandstop filter, and also a 0.5 m Zeiss spectrometer. No photoluminescence has been observed. This may be due to the size, shape, and density of the tips. Also, it is possible that the depth of the Si damaged by the RIE process is greater than the HF etch depth so that non-radiative recombination centers are still present on the surface.

4. CONCLUSIONS

Using high resolution electron beam lithography and RIE with Cl_2 and $SiCl_4$ gases we have etched sub-50 nm Si pillars, trenches and ridges with aspect ratios greater than 10. These are among the smallest features ever fabricated using these techniques. We believe that the resolution limiting factor for the RIE etching procedure described here is due to the mask definition process and the mechanical stability of the Si, rather than etching process itself. No photoluminescence has been detected from arrays of pillars with feature sizes on the order of 10 nm that have been passivated using HF acid.

5. ACKNOWLEDGEMENTS

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