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Reduction of Hydrogen Annealing Temperature for Shape Transformation of Si Surface by Very Short Water Rinse

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1. Introduction

We have shown that the biosensing using Si ring resonator (see Fig. 1) with the sensitivity of 10^{-9} g/ml will be possible by (1) slot-type waveguide at wavelength of 1.3 µm, (2) improving quality factor *Q* of the ring resonator, (3) specific adsorption of the analyte to the detector surface, and (4) maintaining temperature within $\pm$ 0.005°C [1].

We have been studying the effect of the high-temperature hydrogen annealing to improve the quality factor *Q* of the Si ring resonator(see Fig. 2) which is drastically changed with the surface roughness. Thermal annealing in hydrogen ambient can reduce surface roughness of as-etched silicon surface to levels comparable to commercial polished wafers. Due to the mass transport effect, a sharp corner on an etched step rounded by the H_2 annealing. Sato *et al.* demonstrated several types of buried voids by annealing etched Si trenches in pure hydrogen at 1100°C [4]. So far, most of the previous studies focused on the transformation of deep trenches or mesa structure [2-4]. In this paper, we investigate the effect of hydrogen annealing on the sidewall profiles of Si waveguide on silicon-on-insulator (SOI) to improve the Q factor of the Si ring resonator .

2. Experimental

In order to improve *Q* factor, we have investigated the effect of H_2 anneal temperature from 925 to 800°C which are much lower than that of M. M. Lee *et al.* [5]. The surface migration of Si surface atoms takes an important role and it depends on the annealing temperature, the gas pressure, and the reaction time. The Si wafer was cleaned using SC1 then annealed in pure hydrogen [purity is 99.9999%] at 40 Torr for 10 minutes.

We have found that the final water rinse time drastically affect to the Si shape transformation. After the short water rinsing, the sample was quickly (~10s) dried by N_2 blowing and it was set in the furnace. Additionally we have investigated the effect of thermal oxidation after H_2 anneal.

3. Results and Discussions

3.1 revised method solving thermal etching problem

At the first step, we inserted wafers into the furnace at 900°C. Then, thermal etching occurred as shown in Fig. 3. The thermal etching could be due to the reaction $Si+O_2 \rightarrow SiO_2$ and $SiO_2 + H_2 \rightarrow SiO + H_2O$ [5].The hydrogen atoms react with silicon dioxide to yield flammable SiO and water vapor. In order to protect oxidation, we inserted wafers at low temperature (300°C) as shown in Fig. 4. Then we succeeded in suppression of the thermal etching.

3.2 Effect of water rinse time

Figure 5 shows the cross sectional SEM images of the Si waveguides after H_2 anneal for the water rinse time of 480 s. Anneal temperature is 891 to 925°C. The initial rectangular cross section is deformed to dome shape and the Si surface seems to become smooth. Next, we have investigated the effect of water rinse time before the H_2 anneal. In Fig. 5, the water rinse time is long (480s) which will cause the growth of natural oxide on Si surface. So, we reduced the time to 10 s. Figure 6 shows the SEM images after H_2 anneal with water rinse time of 10 s. At 850°C it deformed to dome shape similar as one at 925°C with water rinse time of 480 s. We have achieved the reduction of the anneal temperature by 75°C. Figure 7 shows annealing temperature versus curvature radius at the corner of deformed Si waveguide. Our results are compared together with the result of Kuribayshi [2] and Lee [5]. Our result with 10s water rinse seems to be positioned on the extrapolated curve after Lee *et al* [5]. Consequently presumably Lee *et al* could also employed short water rinse. However, our work, for the first time, demonstrate the effect of the short water rinse to the deform during H_2 anneal.

3.3 Model of enhanced deformation at short water rinse

The Si deforms by H_2 anneal caused by the reduction of natural oxide layer which mechanism is shown in Fig. 8.The Si oxide is formed during long water rinse and the oxide suppresses the migration of Si atoms.

3.4 Further smoothing by thermal oxidation

After the H_2 anneal, Si waveguide deformed. However, the smoothness is still not enough. So, we have investigated the effect of thermal oxidation after H_2 anneal. The sample is oxidized at 1000°C dry O_2 50 min (SiO_2 50nm). Figure 8 shows the cross sectional SEM images after oxidation and after etched off the oxide layer. The surface of the waveguide becomes more smoothly.

4. Conclusions

We have found that short water rinse (10s) enhances the deformation of Si during H_2 annealing. Previous very high temperature (1000°C) anneal is reduced to ~850°C by this treatment. Suppression of surface migration of Si atoms by covered SiO_2 is proposed as the mechanism. This technology will be widely useful for variety application such an electron device (Si on nothing, SON and Si photonics).

References

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[3] K. Sudoh *et al*.: Jpn. J. Appl. Phys. **43** (2004) 5937.[4] T. Sato *et al*: Jpn. J. Appl. Phys. **39** (2000) 503.[5] M.M. Lee *et al*.: Solid-State Sensor, Actuator and Microsy. Workshop.,(2004) 19.

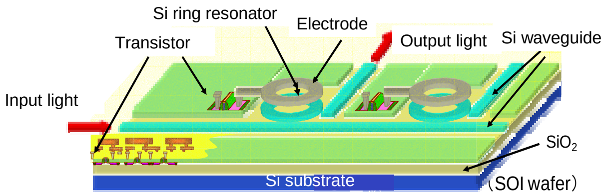


Fig. 1. Proposed biosensor chip with Si ring resonators.

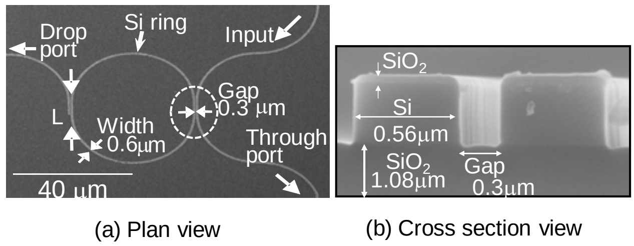


Fig. 2. Typical example of SEM photograph of fabricated Si ring resonator.

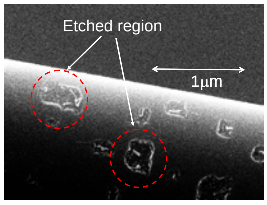


Fig .3. SEM image of thermal etching. The waveguide was annealed at 1000°C and 40 Torr for ten minutes.

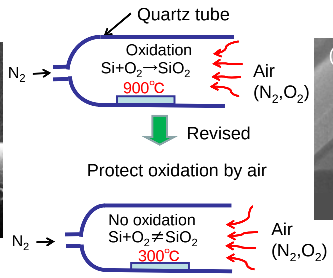
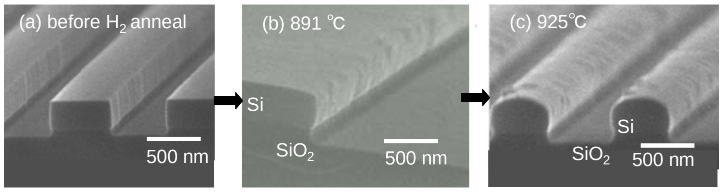
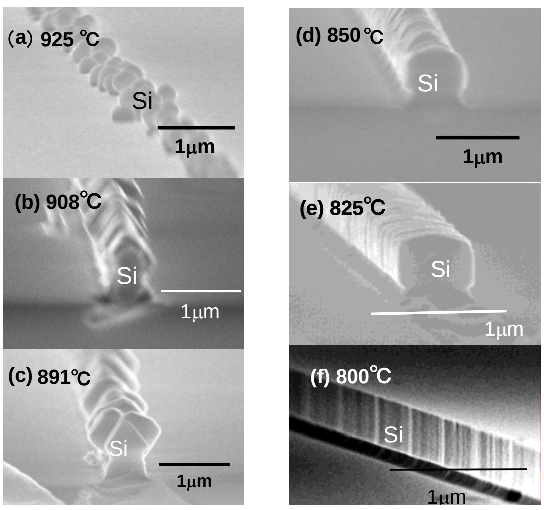


Fig. 4. Revised method solving thermal etching problem.



Water rinse time is 480 s.

Fig. 5. Cross sectional SEM images of the Si waveguides (a) before H_2 anneal, and (b) after H_2 anneal (908°C), and (c) after H_2 anneal (925°C). Water rinse time is 480 sec.



Water rinse time is 10 s.

Fig. 6. Cross sectional SEM image of waveguide. Anneal temperature is 925, 908, 891, 850, 825, and 800°C. Water rinse time is only 10 s.

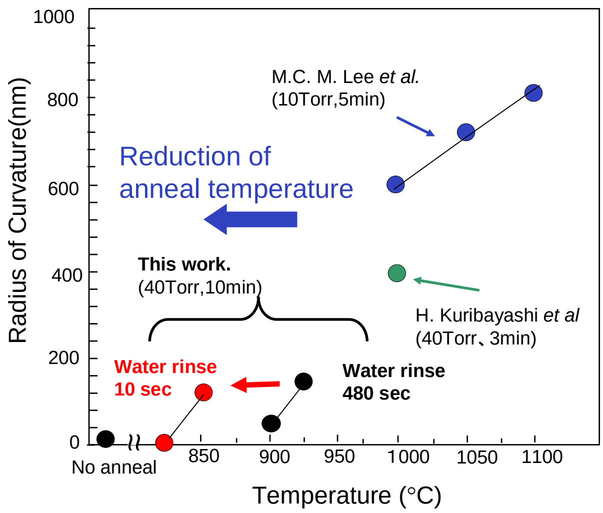


Fig.7. Annealing temperature versus curvature radius of deformed Si waveguide.

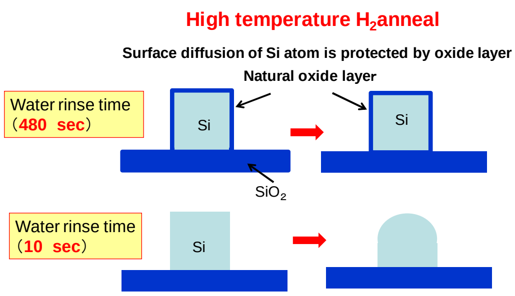


Fig. 8. Effect of water rinse time. Natural Si oxide layer supposed to protect surface diffusion of Si atom.

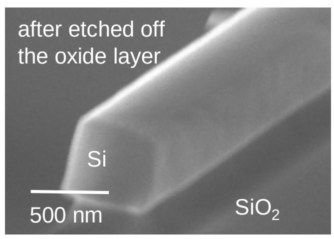


Fig. 9. Cross sectional SEM images after oxidation and after etched off the oxide layer.