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Bulk micromachining of silicon in TMAH-based etchants for aluminum passivation and smooth surface

K. Biswas*, S. Das, D.K. Maurya, S. Kal, S.K. Lahiri

Microelectronics Laboratory, Advanced Technology Centre/E and ECE Department, Indian Institute of Technology, Kharagpur-721302, India

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Abstract

The fabrication of silicon based micromechanical sensors often requires bulk silicon etching after aluminum metallization. All wet silicon etchants including ordinary undoped tetramethyl ammonium hydroxide (TMAH)–water solution attack the overlaying aluminum metal interconnect during the anisotropic etching of (100) silicon. This paper presents a TMAH–water based etching recipe to achieve high silicon etch rate, a smooth etched surface and almost total protection of the exposed aluminum metallization. The etch rate measurements of (100) silicon, silicon dioxide and aluminum along with the morphology studies of etched surfaces are performed on both n-type and p-type silicon wafers at different concentrations (2, 5, 10 and 15%) for undoped TMAH treated at various temperatures as well as for TMAH solution doped separately and simultaneously with silicic acid and ammonium peroxodisulphate (AP). It is established through a detailed study that 5% TMAH–water solution dual doped with 38 gm/l silicic acid and 7 gm/l AP yields a reasonably high (100) silicon etch rate of 70 μm/h at 80 °C, very small etch rates of SiO_2 and pure aluminum (around 80 Å/h and 50 Å/h, respectively), and a smooth surface (±7 nm) at a bath temperature of 80 °C. The etchant has been successfully used for fabricating several MEMS structures like piezoresistive accelerometer, vaporizing liquid micro-thruster and flow sensor. In all cases, the bulk micromachining is carried out after the formation of aluminum interconnects which is found to remain unaffected during the prolonged etching process at 80 °C. The TMAH based etchant may be attractive in industry due to its compatibility with standard CMOS process.

Keywords: MEMS; TMAH; Bulk micromachining; Dual doping; Aluminum passivation; Piezoresistive accelerometer

1. Introduction

The photolithography and etching techniques of silicon integrated circuits have been suitably adapted to realize MEMS for various applications. Silicon has therefore remained as the principal material for MEMS. Silicon has excellent mechanical properties [1]. The wet anisotropic etching of silicon is extensively used to realize complex 3D microstructures. The etchants used in bulk micromachining include inorganic aqueous solutions of KOH [2–4], NaOH [5], RbOH [6], CsOH [7], NH_4OH [8], Hydrazine $(N_2H_4)_n$ [9] and organic etchants like EDP [10] and TMAH [11–13]. The most commonly used etchants are KOH, TMAH and EDP. The choice of a particular silicon etchant for fabricating micromechanical structures depends not only on the etch rate selectivity and anisotropy but also on its compatibility with integrated circuit fabrication processes. Tetramethyl ammonium hydroxide (TMAH) is gaining popularity in MEMS as an alternative to KOH and EDP. Tetramethyl ammonium hydroxide is a quaternary ammonium hydroxide based alkaline solution which has excellent etching characteristics, and low toxicity levels and is easy and safe to handle. TMAH-water solution is compatible with conventional CMOS fabrication process [11] due to the absence of metal ions in its composition. This property as well as the low etch rate of dielectric layers in TMAH makes it a good choice as a silicon etchant for the future MEMS products integrated with on-chip signal processing circuits.

The bulk micromachining requires a higher etch rate which is achievable at a lower TMAH concentration [11,12]. It, however, results in the formation of pyramidal hillocks [11–14] that makes the surface

* Corresponding author. Tel.: +91 3222 28 1479; fax: +91 3222 25 5303.
E-mail address: kanishka@ece.iitkgp.ernet.in (K. Biswas).

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rough and limits the etch rate. This problem has been tackled by adding ammonium peroxodisulphate [12,15–17]. Moreover, TMAH attacks aluminum metallization [11,12]. Thus, bulk micromachining requiring long etching times cannot be performed at the end of IC fabrication. The addition of silicon [14–16] or silicic acid [17,18] along with ammonium peroxodisulphate has been found to prevent aluminum etching. The authors have conducted a systematic study of silicon etching in TMAH under different experimental conditions. The etching characteristics of silicon dioxide and aluminum have also been investigated in detail. Experiments have been repeated by adding silicic acid and ammonium peroxodisulphate and a nearly optimum etchant composition and temperature have been established. The results are reported in this paper.

2. Experimental

2.1. Experiments using undoped TMAH aqueous solutions

Four-inch diameter, (100) orientation, 525 μm thick single-side-polished p-type (resistivity 10–40 Ω-cm) and n-type (resistivity 4–6 Ω-cm) silicon wafers were used for the present study. Initially, a silicon dioxide layer of thickness around 1 μm was thermally grown on silicon wafer by the cyclic oxidation (dry–wet–dry) process. Rectangular and square windows of various dimensions were opened on the front surface oxide layer of the wafer by photolithography process. Aluminum layer of thickness 0.4 μm was deposited on the front side by the thermal evaporation technique. Suitable patterns of aluminum film were formed by photolithography. The TMAH–water solution was prepared by diluting commercially available TMAH (25%, Merck, Germany). All experiments were carried out in a closed glass vessel with a constant temperature bath. A water cooled reflex condenser was used in the etching bath to avoid variations in concentration of the etchant during etching. Samples were immersed vertically in the etching solution. Before insertion of the samples in TMAH solution, native oxide was removed with no damage to the aluminum metal patterns using a special native oxide etchant followed by rinsing in de-ionized water. A magnetic stirrer rotating at a speed of 100 rpm was used constantly during etching experiments to facilitate reactant transport to the surface and removal of reaction byproducts for achieving uniform reaction rate.

Etching experiments were performed on both p-type and n-type substrates for different TMAH concentrations and etching temperatures. The TMAH concentration in the solution was varied from 2 to 15% and the temperature from 50 to 80 °C in steps of 10 °C. Two samples were used for each experiment. The same experiment was repeated twice to check repeatability and consistency of results. The etching was carried out over a span of 30 min wherein the second sample was inserted 15 min after the first. As the etch rate of silicon dioxide is very small in TMAH [11,13,19], an etching time of 60 min was used to determine etch rate. The etch rate of silicon and aluminum, and the surface roughness after etching was measured by Dektak^3 surface profilometer by averaging the readings obtained from the *x*- and *y*- scans. The silicon dioxide thickness was measured using an ellipsometer and hence the etch rate was determined.

2.2. Experiments using dual doped TMAH water solution

For this study, 5% TMAH solution was used at 80 °C with different concentrations of silicic acid (10–44 gm/l). In the present study instead of pure silicon, silicic acid has been added in TMAH solution for aluminum passivation because silicic acid requires very less time to dissolve in TMAH solution compared to pure silicon. The aluminum etch rate was measured in each case. The variation of aluminum etch rate was also measured for both, ammonium peroxodisulphate (AP) addition of different concentration (upto 10 gm/l) to 5% TMAH solution mixed with silicic acid of concentration 30 gm/l and silicic acid addition of different concentration (10–44 gm/l) to 5% TMAH doped with 7 gm/l ammonium peroxodisulphate (AP) at 80 °C. Next, ammonium peroxodisulphate (AP) was added in different concentrations (upto 10 gm/l) to 5% TMAH solution mixed with silicic acid of concentration 38 gm/l. In all the experiments using dual doped TMAH solution silicic acid was first dissolved in TMAH solution and subsequently ammonium peroxodisulphate (AP) was added. The variation of etch rate of silicon and the roughness of the etched silicon surface with the concentration of AP were measured. The etched surfaces were also observed by optical microscope and SEM.

3. Results and discussions

3.1. Variation of silicon, aluminum and silicon dioxide etch rates in undoped TMAH

Fig. 1 shows the etch rates of p-type and n-type (100) silicon wafers for four different TMAH concentrations (2, 5, 10 and 15%) at four different bath temperatures (50, 60, 70 and 80 °C). In general, n-Si etches slightly faster than p-Si. The etch rate increases with temperature and decreases with TMAH concentration. A maximum etch rate of about 60 μm/h is obtained with 2–5% TMAH concentration. For such etching conditions, the scanning electron microscopy and surface profilometry reveal rough surface with hillocks. At higher TMAH concentrations the surface becomes smoother. Fig. 2 shows the surface roughness vs temperature for different TMAH concentrations. The SEM photograph of the surface