Transformer coupled plasma etching of 3C-SiC films using fluorinated chemistry for microelectromechanical systems applications

Di Gao, Muthu B. J. Wijesundara, and Carlo Carraro
Department of Chemical Engineering, University of California, Berkeley, California 94720

Roger T. Howe
Departments of Electrical Engineering and Computer Sciences, and Mechanical Engineering, University of California, Berkeley, California 94720

Roya Maboudian
Department of Chemical Engineering, University of California, Berkeley, California 94720

(Received 6 May 2003; accepted 15 December 2003; published 20 February 2004)

Polycrystalline 3C-SiC films are etched by oxygen-mixed sulfur hexafluoride transformer coupled plasmas for microelectromechanical systems (MEMS) applications. Silicon dioxide is employed as etching masks, which avoids the micromasking phenomena and chamber contamination commonly involved when using metals as masks. The etch rate, selectivity, and profile are characterized as functions of O₂ percentage in the etching gas. Etch rates of SiC remain almost unchanged at about 3600 Å/min up to 50% O₂, but decrease significantly when more than 50% O₂ is used. Etch selectivity of SiC over SiO₂ reaches maximum of 2.6 when using 50% O₂. The chemical composition and the topography of the etched SiC films are also examined. By integrating the etching process with conventional surface micromachining technology, functional SiC-based MEMS resonators are fabricated. © 2004 American Vacuum Society. [DOI: 10.1116/1.1648067]

I. INTRODUCTION

Silicon carbide (SiC) is an attractive semiconducting material for micro- and nanoelectromechanical systems (MEMS) applications from many perspectives. Because of its superior electronic and mechanical stability at high temperatures and its extraordinary chemical inertness, SiC is naturally considered to complement Si as the structural material in MEMS devices operating in harsh environments. As a consequence, SiC-based MEMS has been applied in high-temperature gas, temperature and pressure sensors and micromachined components in gas turbine engines. Recently, SiC has also gained attention as UHF resonator filters for wireless communications. The high Young’s modulus to mass density ratio and the extremely stable surfaces of SiC are advantageous for this application. In addition, SiC has been found to be a biocompatible material, with potential applications in corrosion-resistant bioMEMS devices. Distinguished from more than 200 polytypes, the cubic phase of SiC (3C-SiC) is of the most interest to MEMS, because it can be grown in polycrystalline thin-film form by chemical vapor deposition (CVD) on a variety of substrates, including Si, SiO₂ and Si₃N₄, offering great flexibility for device fabrication.

One of the challenges in fabricating SiC MEMS is the selective etching of SiC films or bulk materials. Because SiC is inert to most aqueous chemicals at temperatures less than 600 °C, wet etching by acids or bases is impractical for SiC. Nonstandard techniques such as laser-assisted photoelectrochemical etching of SiC have been developed, but they require special equipment and have poor process control on the etching uniformity across wafers. Therefore, plasma-assisted etching plays an important role in patterning SiC, and has been extensively studied. Fluorine-based plasmas utilizing various fluorinated gases (such as CHF₃, CF₄, and SF₆, etc.) are commonly employed in the microfabrication process, and have been chosen for most SiC etching experiments. Oxygen and helium have also been applied in combination with the fluorinated gases to increase the etch rate and etch selectivity, and to improve the etch profile. A review of plasma etching of SiC in fluorinated chemistry can be found elsewhere. Conventional etch masks for dry etching, such as hard-baked photoresist, SiO₂, and Si₃N₄, are etched at higher rates than SiC, which has motivated the employment of metals as etch masks. However, metal atoms of the mask material are sputtered by the plasma and deposited into the etch field, producing grass-like structures through a phenomenon called micromasking. Metal masks in plasma etching also cause contamination in subsequent fabrication steps, and hence are commonly prohibited in integrated circuit (IC) processes. Therefore, selective etching of SiC using nonmetallic masks is highly desirable for SiC MEMS fabrication.

Recently, the employment of high-density plasmas in SiC dry etching has shown to significantly improve the etch rate and the etch profile of SiC materials. Such plasmas are generated at low pressures, and have low plasma potential but high ionization efficiencies. High-density plasma systems, such as electron cyclotron resonance and transformer/inductive coupled plasma (TCP/ICP), have been applied in fluorinated chemistry to achieve high SiC etch rates. Deep reactive-ion etching of SiC has also been reported. However, many of these experiments still employ metal masks, which are not permitted in standard microfabrication laboratories. Little work has been done to explore the possibility of...
using nonmetallic etch masks such as SiO₂ to pattern SiC in these plasmas.

In this article, we report a selective etching process for polycrystalline 3C-SiC films (poly-SiC) using oxygen-mixed sulfur hexafluoride (SF₆) TCPs. Nonmetallic standard IC processing materials, such as SiO₂ deposited by low-temperature CVD (LPCVD) and plasma-enhanced CVD, are employed as etch masks. The etch rate of SiC, etch selectivity to SiO₂, and etch profile are investigated as functions of process parameters, specifically the percentage of oxygen in the etching gas. The chemical component and the topography of the etched SiC surfaces are also examined. By using the etch process in a conventional surface micromachining technology, a functional SiC-based folded-flexure comb-drive MEMS resonator is fabricated.

II. EXPERIMENTAL PROCEDURE

The poly-SiC films are deposited by LPCVD at 850 °C, and in situ doped by introducing 2% NH₃ (in flow rate) to the precursor 1,3-disilabutane. The film deposition system and procedure have been described elsewhere. The doped poly-SiC film is highly textured in the (111) direction, as shown by x-ray diffraction experiments, and has a resistivity of about 0.02 Ω·cm. The TCP etching is performed in a commercial etching system (TCP 9400, Lam Research Corp.). In this study, the TCP top power, bottom power, the chamber pressure, and the substrate temperature are held constant at 300 W, 200 W, 12 mTorr, and 60 °C, respectively. The total gas flow rate (SF₆ and O₂ gases) is fixed at 20 sccm, while the O₂ percentage is varied from 0 to 100. Two types of SiO₂ films are employed as etch masks, both of which are standard IC processing materials, commonly available in microfabrication laboratories. The low-temperature CVD SiO₂ films (LTO) are deposited using SiH₄ and O₂ gases at 450 °C in a commercial Tylan LPCVD furnace. The plasma-enhanced CVD SiO₂ films (PEO) are deposited using SiH₄ and N₂O gases at 348 °C in a commercial Technics plasma system. To measure the etch rates, poly-SiC, LTO, and PEO films are all deposited on Si(100) substrates. Etch fields of 2 μm line-and-space features are patterned using SiO₂ masks for SiC and hard-baked photoresist for LTO and PEO. The etch rates are then calculated from the step heights of etched lines after removing the masks. The etch profile is examined by cross-sectional scanning electron microscopy (SEM) afterwards. Ex situ x-ray photoelectron spectroscopy (XPS) is employed to study the effect of etching on the chemical composition of film surfaces. An Omicron Dar400 achromatic Mg K x-ray source (15 kV, 20 mA emission current) and an Omicron EA125 hemispherical analyzer are used in the XPS experiment. Atomic force microscopy (AFM, DI Multimode III) is employed to study the effect of etching on the film topography.

III. RESULTS AND DISCUSSION

Figure 1(a) shows the etch rates of poly-SiC, LTO, and PEO films as functions of the O₂ percentage in the O₂-mixed SF₆ plasma. The data are repeatable to within ±10%. It is observed that the etch rate of SiC remains almost unchanged around 3600 Å/min up to 50% O₂, but decreases significantly from ~3500 Å/min at 50% O₂ to ~30 Å/min at 100% O₂. In contrast, the etch rates of LTO and PEO films decrease monotonically as the O₂ percentage increases. As a result, the etch selectivity (etch rates ratio) of SiC over LTO and PEO first increases and then decreases gradually as the O₂ percentage increases, reaching the maximum of 2.6 for SiC/LTO and 1.9 for SiC/PEO at 50% O₂, as plotted in Fig. 1(b). It is worth mentioning that the etch rate of SiC is 3500 Å/min when the etch selectivity toward LTO reaches 2.6, which is favorable in terms of achieving both high etch rate and high etch selectivity.

The effects of O₂ percentage in O₂-mixed SF₆ plasma for the etching of SiC have been discussed elsewhere. The fact that the SiC etch rate does not change significantly with O₂ up to 50% in SF₆ plasma has been reported previously in ECR and ICP etching. Adding oxygen to the plasma is proposed to assist in SiC etching mainly in two aspects: the liberation of F species in the plasma, and removal of C by forming volatile molecules such as CO, CO₂, and COF₂. Oxygen may also inhibit the etching process by producing SiO₂ on the surface. The etch rate of SiC as a function of O₂ percentage (Fig. 1) may be the result of the competition of these possible effects. Figure 1 also shows the lower etch rate of LTO with respect to PEO, which is expected since LTO films are better quality oxide than PEO.

Figure 2 shows the etch profiles of 2 μm line-and-space...
features produced in about 2-μm-thick SiC films on Si(100) substrates using 0%, 10%, 50%, and 80% O2-mixed SF6 plasmas, respectively. Here, LTO films of about 1 μm in thickness are employed as etch masks. The LTO etch masks are totally consumed during the etching when 0%, 10%, and 80% O2 is used, but remain left on top of the SiC when 50% O2 is used due to the higher etch selectivity. It can be clearly seen that the sidewalls of the etched SiC films are strongly tapered, and the line width of the feature shifts from the originally defined dimension. The sidewall slope of the etched SiC films is observed to increase as the O2 percentage is increased. The etch profile of the 50% O2 recipe [Fig. 2(c)] that achieves the highest etch selectivity is acceptable for many MEMS applications.

The sidewall tapering in plasma etch processes has been reported previously,20,22 and is known to be strongly dependent on the etch process parameters, etch features, and etch aspect ratios. The tapered sidewall is the result of many possible effects, including mask erosion, deposition of the etch products desorbed from the surface and etch inhibitor from the plasma to the sidewall and the substrate, and angular dependencies of the etch yield and etch rate. The etch profiles also show an enhanced etch rate at the edges of the pattern, a phenomenon called trenching. This most often occurs when the sidewalls of the pattern are tapered at a steep angle, where some of the low-angle ions and reactive species reflect off the tapered surface toward the pattern edge, resulting in trenches. An in-depth analysis of the etch profile in our experiment requires detailed knowledge of the TCP etch process, as well as the etching sidewall chemistry, which future research will explore.

The chemical composition of etched SiC surfaces is examined by ex situ XPS. Figure 3(a) shows the survey scans of SiC films etched by different recipes for 1 min. Various peaks are identified on the figure. The spectrum obtained on the film prior to etching is dominated by Si 2p (101 eV), Si 2s (152 eV), C 1s (283 eV), and O 1s (532 eV) peaks. These peak positions are consistent with an earlier publication on the 3C-SiC films.23 The O 1s peak is attributed to sample exposure to air prior to introduction to the XPS chamber. As expected, F and O are present on the surfaces after etching. In addition, the relative intensities of the O 1s and O Auger (745 eV) peaks increase, while the intensities of the F 1s (687 eV) and F Auger (597 eV) peaks decrease as more O2 is used in the etching gas. The F peaks appearing in the sample etched in the 100% O2 plasma is most likely due to the etch chamber contamination. High-resolution spectra are recorded for each element and used to calculate the elemental composition. The Si/C ratio is found not to change significantly after etching by different recipes. Furthermore, the high-resolution scans of C 1s photoemission peaks presented in Fig. 3(b) show no significant contribution due to CFx bonding, indicating that there is no carbon fluoride polymer formed on the SiC surface during the etch process.

To examine the effect of etching on film topography, SiC films of 2 μm in thickness are deposited on Si(100) substrates and etched without patterning. AFM images of SiC films taken before and after etching in the 50% O2-mixed SF6 plasma are presented in Fig. 4. The rms roughness of the sample from a 10×10 μm² area is 28 nm before etching.
and decreases to 22 nm after 1 min etching, and further decreases to 12 nm after 4 min etching, which means the etch process actually smoothes the surface as etching proceeds. Smoothening of etched surfaces has been observed previously in anisotropic dry etching of SiC films and other semiconductor materials. This behavior may be explained by the fact that inclined surfaces tend to be etched more quickly than flat surfaces due to the angular dependence of the etch yield and etch rate in a reactive-ion etching process. The smoothening of SiC surfaces also indicates that there is no significant preferential loss of silicon from the SiC surface during etching, which is in agreement with the XPS results.

To demonstrate the use of the etch process in a conventional surface micromachining technology, a functional SiC double-flexure comb-drive resonator is fabricated. A schematic cross section of the resonator is shown in Fig. 5. Briefly, the microfabrication process proceeds as follows: (i) Si(100) wafers are thermally oxidized and coated with Si$_3$N$_4$…
layer by CVD; (ii) 0.5 μm doped polycrystalline Si film and 2 μm LTO sacrificial layer are deposited on top of Si₃N₄; (iii) ~1.8 μm doped poly-SiC film is deposited; (iv) poly-SiC film is etched by 50% O₂-mixed SF₆ TCP using LTO as the etch mask; and (v) the SiC resonators are released by time-etching of the sacrificial LTO in HF. The SEM images of the released resonator and a close-up of the interdigitated comb fingers are shown in Figs. 6(a), and 6(b), respectively. To observe the SiC etch profile in the resonator fabrication process, one of the folded beams is manually broken after testing and examined using cross sectional SEM [Fig. 6(c)]. The sidewall profile is fairly consistent with the result shown in Fig. 2(c). The resonator resonates both in atmospheric pressure and in vacuum. A frequency response of the resonator actuated in a vacuum probe station (30 μTorr, room temperature) is plotted in Fig. 7. The signal is detected by an HP 4195A network analyzer after feeding the current from the sense probe to an off-chip transimpedance amplifier. It is tested and examined using cross sectional SEM.

IV. CONCLUSIONS

In summary, poly-SiC films have been etched using fluorinated chemistry in a commercial TCP system. Nonmetallic SiO₂ is used as the etch mask, which has rarely been reported. By varying the O₂ percentage in the O₂-mixed SF₆ etching gas, the etch rate of SiC, etch selectivity over SiO₂, and etch profile have been characterized. An etch selectivity of 2.6 for SiC/SiO₂ can be achieved using 50% O₂, while still maintaining a relatively high etch rate of 3500 Å/min for SiC. Carbon fluoride polymer is not observed on the etched SiC surface when examined by XPS. A functional SiC MEMS resonator has been fabricated as a demonstration of integrating the etch process into conventional surface micro-machining technologies.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support of DARPA MTO program and the Sandia National Laboratories. The author would also like to thank C. W. Low for helping test the SiC comb-drive resonators.

#References#