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Control of Trenching and Surface Roughness in Deep Reactive Ion Etched 4H and 6H SiC

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ABSTRACT

An optimized deep reactive ion etching (DRIE) process for the fabrication of SiC microstructures has been developed. The optimized process enables the etching of 4H and 6H SiC to depths > 100 µm with the required characteristics of (1) high rate (>0.5 µm/min), (2) vertical sidewalls, (3) minimal microtrenching at the sidewall base, and (4) smooth etched surfaces. The optimized process was determined based on the results of an experiment (full factorial design) which determined how the etch characteristics are affected by four important process parameters: temperature of the wafer chuck, pressure within the chamber, and concentrations of O_2 and Ar in a gas flow comprised of O_2, Ar and SF_6. This study is believed to be the first systematic investigation of the effect of temperature on SiC DRIE characteristics; substrate heating was found to be a key in producing the desired etch properties.

INTRODUCTION

Single-crystal silicon carbide (SiC) of the 4H and 6H polytypes has attractive characteristics for harsh-environment microelectromechanical systems (MEMS) such as high-temperature pressure sensors [1]. Fabrication of SiC MEMS frequently requires that SiC be etched to depths of 100 µm or greater. Various groups have demonstrated deep reactive ion etching (DRIE) of SiC with a high etch rate (>0.5 µm/min) [2], but a process has not yet been demonstrated which provides high rate together with other required etch characteristics: vertical sidewalls are needed so that lateral dimensions do not vary as a function of etch depth; microtrench depth and roughness of the etched surface must be minimized to achieve high strength. A microtrench, which is caused by a locally higher ion flux (and etch rate) at the base of the sidewall, can significantly weaken a pressure sensor diaphragm by concentrating stress. We report here the development of a SiC DRIE process which enables the high-rate fabrication of SiC MEMS that are largely free of performance-reducing micromachining defects.

EXPERIMENTAL DETAILS

Deep reactive ion etching was performed in a ST Systems Multiplex ICP inductively coupled plasma etcher. Because heating is not required to volatilize the etch products, DRIE of SiC is generally performed with the substrate cooled to about room temperature. Previously, experiments in which the SiC sample was thermally isolated and allowed to heat up by plasma action indicated that increasing the sample temperature might help minimize microtrenching. Therefore, the etcher was modified to provide substrate heating prior to the work reported here.

An experiment was designed to determine how microtrench depth, sidewall slope, surface roughness and etch rate were affected by key process parameters. The process parameters (and the range of values studied) were chuck temperature (20 to 125 °C), pressure (5 to 25 mT), and concentrations of O_2 and Ar (0 to 40%) in a gas flow comprised of O_2, Ar and SF_6. Widely separated low and high values were chosen to produce readily discernable effects. Previous experiments had shown that the etch characteristics depend strongly on these parameters and, in addition, on the coil and platen powers. The coil and platen powers, however, were excluded from this study; instead, they were fixed at values as high as was deemed practical, 2.5 kW and 200 W, in order to produce the highest possible etch rates. Total flow was set as high as practical given system constraints (i.e., 50 sccm for 5 mT pressure, and 100 sccm for higher pressures). High gas flows are generally desired to replenish reactants and remove volatile etch products.

Samples were etched using all 16 possible combinations of low and high values of the studied parameters. Each etch process is represented by a 4-bit sequence (0000 to 1111) where each bit indicates a low (0) or high (1) value for temperature, pressure, O_2 and Ar concentration, in that order. An additional six etches were performed using center-point conditions (72.5 ^oC, 15 mT, 20% O_2, 20% Ar) at the beginning, end, and periodically throughout the experiment. These center-point etches provided a measure of process variability and a test for time-dependent effects. Both 6H n-type and 4H p-type SiC specimens were etched. The 6H-N (4H-P) wafers were research grade, Si-face polished with a resistivity of 0.07 Ω-cm (3.9 Ω-cm) and an orientation of 3.5 ^{o} (8.07 ^{o}) [Cree, Inc., NC]. A 300/500 Å Ti/Ni film served as seed layer for the selectively electroplated nickel etch mask, which was approximately 10 to 15 μm thick. After electroplating, the wafers were diced into ~1.3 cm^2 specimens, each of which contained patterns for about 16 diaphragms of four different designs; some of the diaphragm designs employed a central boss (circular unetched region) so that the etched area was ring shaped.

Each SiC etch was preceded by in-situ removal of the Ti/Ni seed layer and cleaning of the SiC surface using a 30 min O_2 plasma followed by a 30 min Ar sputter etch. Etch depths in the range of 100-150 μm (typical of what is required for diaphragm fabrication) were obtained by using a SiC etch duration of 4 hrs (3 hrs in the case of high rate processes). After etching, the SiC specimens were cut in half with a dicing saw to obtain cross-sections which were optically imaged to determine sidewall slopes and microtrench depths. Etch depth and average roughness, Ra, of the etched surface were determined using a stylus profilometer. The results were fit by linear models comprised of 16 terms: an intercept, main effects (i.e., first order terms, X_j) and all cross terms of second (X_jX_k) through fourth order ($X_1X_2X_3X_4$).

EXPERIMENTAL RESULTS AND DISCUSSION

Center point measurements showed a large degree of scatter (e.g., $\sigma = 2^o$ for sidewall slope) but no time-dependent effects for microtrench depth, sidewall slope, roughness and etch rate. Fits to the 16 corner points were good, due to the large number of terms employed in the model. Despite the large scatter in the data, conclusions could be made with sufficient validity to derive an optimized process.

Microtrenching

Figure 1 shows how the 6H-N SiC microtrench depth varied as functions of pressure and temperature for O_2:Ar % concentrations of 0:0, 0:40 and 40:0 (i.e., etch processes XX00, XX10, XX01, where X = 0 or 1). The 4H-P results (not shown) were similar. Negative values of microtrench depth are an artifact caused by the use of a linear model and should be interpreted as 0. The results show minimal microtrenching can be obtained with 100% SF_6 at high temperature and high pressure, and with 40% Ar (60% SF_6) at high temperature and any pressure. All etch processes incorporating O_2 were found to produce significant microtrenching. For this reason,

O_2 addition will not be discussed further in this paper. The observation of re-entrant profiles in conjunction with microtrenching, as can be seen in figure 1d, suggests that microtrenching is not caused by ions rebounding from the sidewalls. Charging of the etched structure may deflect ions towards the base of the sidewall [3], a hypothesis that is supported by the experimental observation that the use of O_2 increases microtrenching. The addition of O_2 might result in the formation of an SiF_xO_y layer [4], which would have a greater tendency to charge than SiC.

Contours for 6H-N SiC microtrench depth [µm]

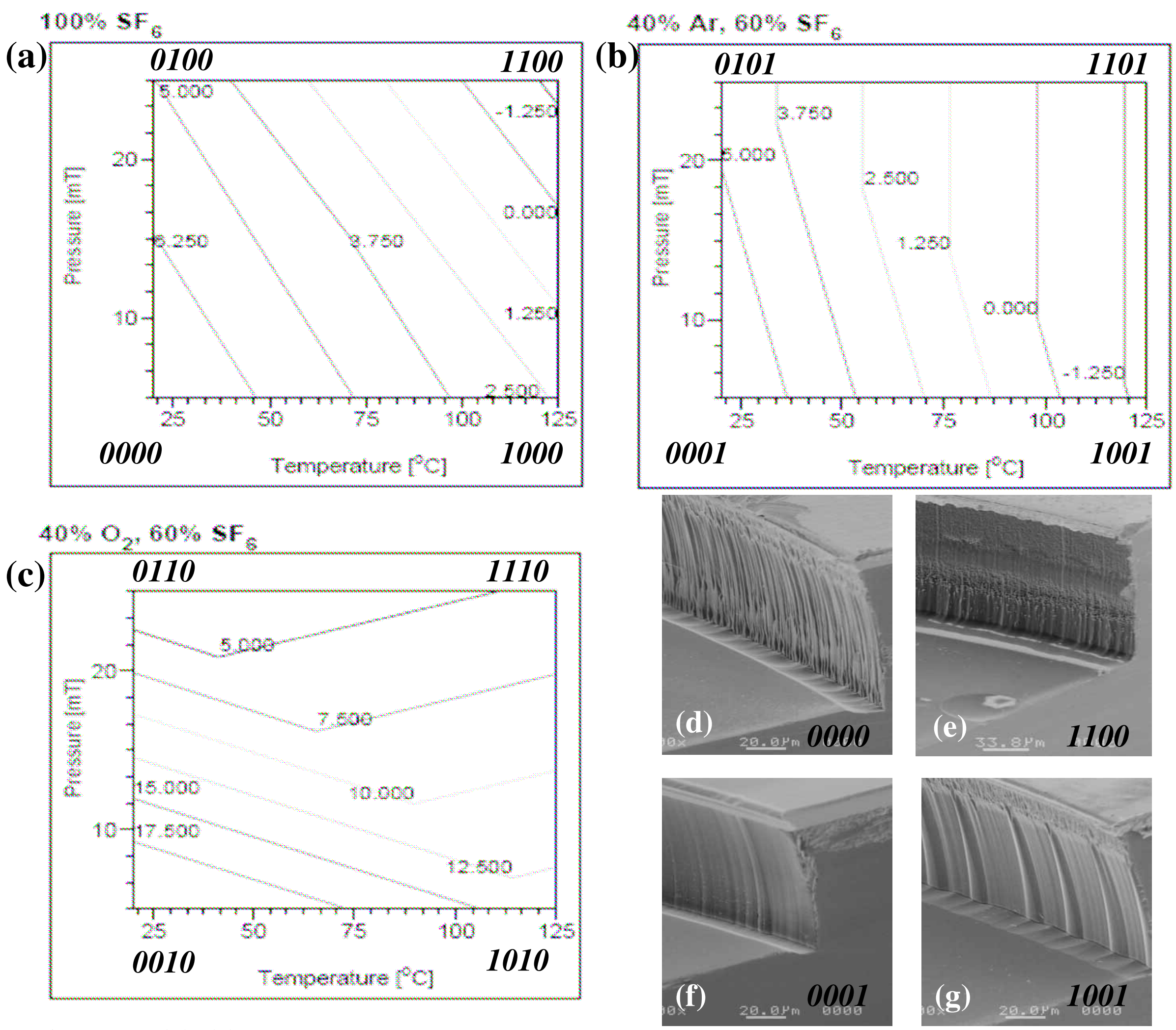

Figure 1. (a)-(c) Least squares fits to microtrench depth data for 6H-SiC as functions of temperature and pressure for various Ar/O_2 % compositions (actual data points are at corners of graphs only). **(d)-(g)** show SEMs of various etch conditions, labeled on graphs.

Sidewall slope

Figure 2 shows how the 6H-N SiC sidewall slope varied as functions of pressure and temperature for Ar % concentrations of 0 and 40. The 4H-P results (not shown) were similar. In general, the sidewall slopes were > 90° (re-entrant profile) and became more nearly vertical with increasing pressure. The addition of Ar produced a greater slope at a given pressure.

Contours for 6H-N SiC slope [°]

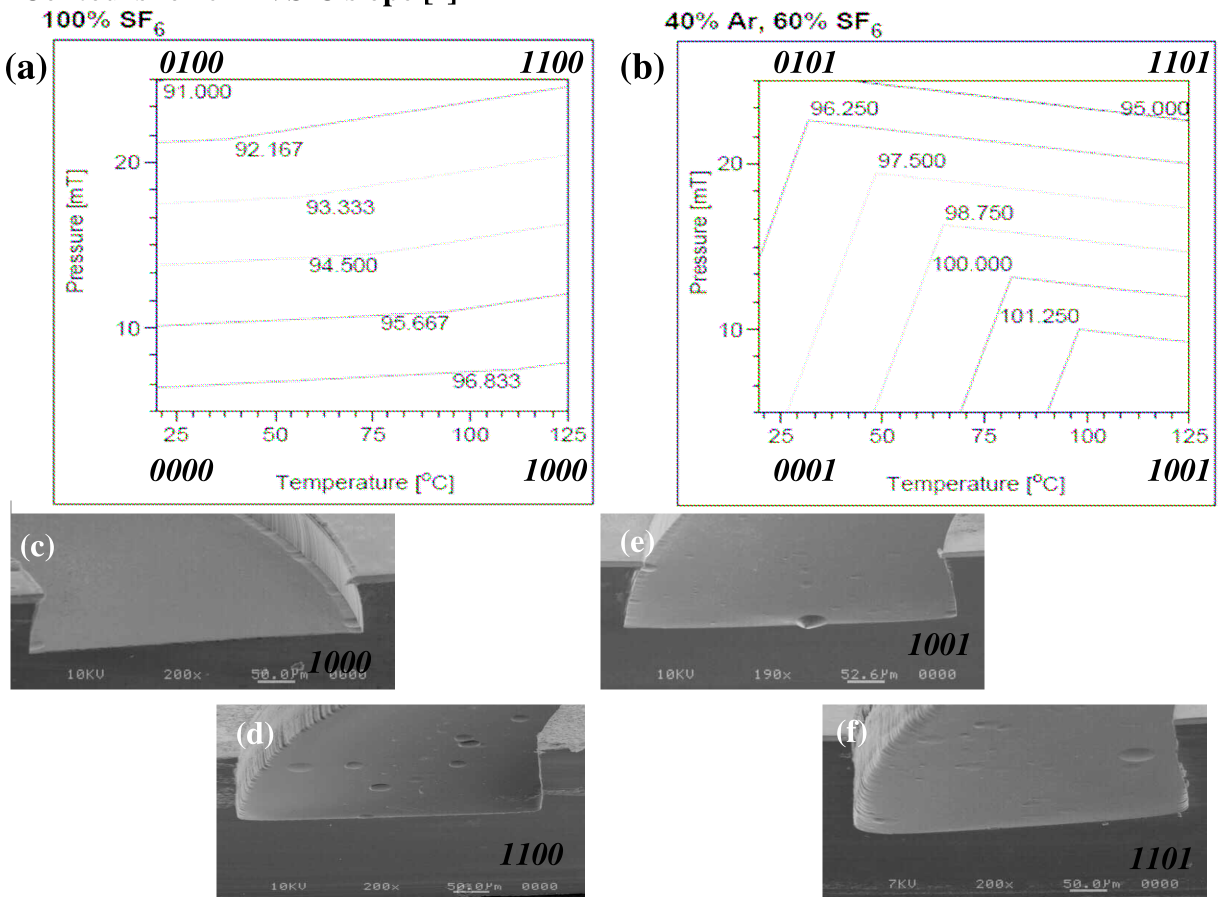

Figure 2. (a)-(b) Fits to sidewall slope data for 6H-N SiC as functions of temperature and pressure for 0% and 40% Ar. **(c)-(f)** SEMs of 6H-N SiC for labeled etch conditions.

Roughness of etched surface

The average roughness, Ra, was the single etch characteristic for which significant differences between 6H-N and 4H-P SiC were observed. Figures 3 (a) and 3 (b) show how Ra varied as functions of pressure and temperature for 6H-N and 4H-P SiC, respectively, using 100% SF_6. At high pressure (the regime of interest because it provides vertical sidewalls), significantly rougher surfaces were obtained for 4H-P SiC; this can be observed by comparing figures 4 and 2d, which show 4H-P and 6H-N SiC samples, respectively, etched with process 1100 (producing Ra = 4300 and 300 Å, respectively). The addition of 40% Ar was found to significantly reduce the roughness of 4H-P SiC for all temperatures and pressures (for 6H-N the effect was not significant). At high temperature and pressure, the addition of 40% Ar reduced Ra of 4H-P SiC from 4300 Å (process 1100) to 200 Å (1101). The addition of Ar has previously

Contours for SiC surface roughness [log (Ra[Å])]

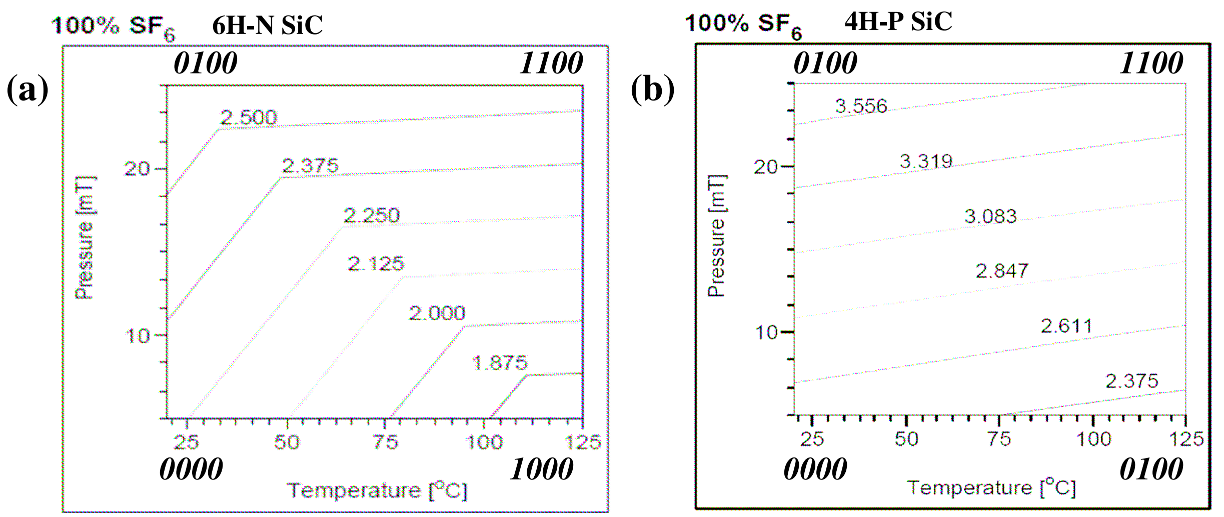

Figure 3. Least squares fit to surface roughness [log (Ra)] data of **(a)** 6H-N SiC and **(b)** 4H-P SiC as functions of temperature and pressure for 100% SF_6.

been shown to provide smoother etched surfaces, possibly by sputtering away etch resistant materials and by creating an amorphous SiC surface layer, which eliminates dimpling caused by enhanced etching in the vicinity of a crystal defect.

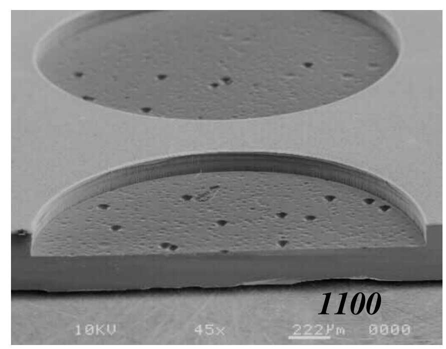

Figure 4. 4H-P SiC etched with process 1100.

Etch rate

The use of high power on the coil (2.5 kW) and platen (200 W) provided high etch rates (> 0.5 µm/min) for all conditions studied. Etch rates of 4H-P and 6H-N SiC were similar. High coil power increases the density of ions and reactive neutral species in the chamber, while high platen power increases the energy with which ions bombard the surface. Both these factors helped maintain high etch rates, over the wide range of temperatures, pressures and gas compositions studied. Etch rate was generally found to increase with increasing pressure, and the maximum etch rate, 0.81 µm/min, was obtained with process 0100.

Determination of optimized process

Determination of the optimized process is driven by the need to simultaneously provide minimal microtrenching, vertical sidewalls, and smooth etched surfaces. High pressure (>25 mT) is required to obtain nearly vertical sidewalls, as shown by figure 2. However, as shown in figure 3, high pressure produces unacceptable roughness for 4H-P SiC, unless Ar is incorporated at a sufficiently high concentration (e.g., 40%). Using 40% Ar concentration and high pressure, microtrench depth can be minimized by using an elevated chuck temperature (e.g., 125 ºC), as shown in figure 1. These etch conditions (high temperature and pressure, 40% Ar) optimize all the considered etch characteristics. An extrapolation of the model of figure 2 was used to determine that the pressure should be 35 mT to produce a 90° sidewall, at 125 °C and 40% Ar.

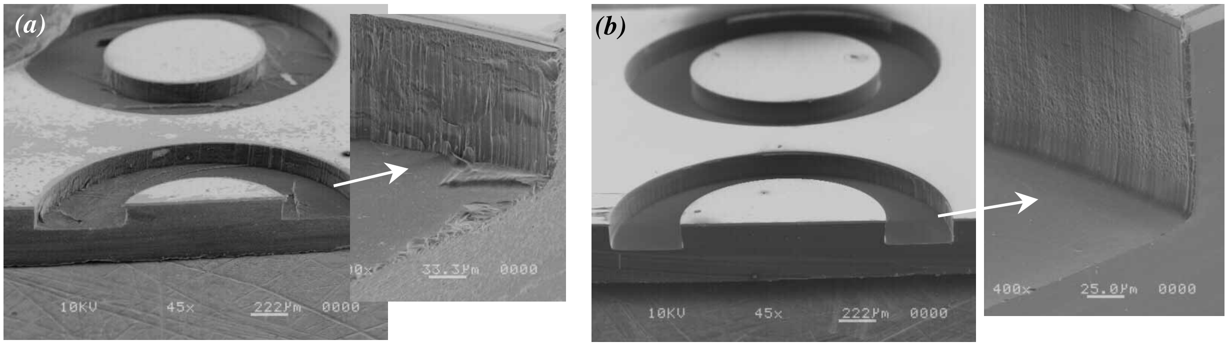

Figure 5. 6H-N SiC etched 160 µm using optimized process at **(a)** 125 °C, **(b)** 180 °C.

Figure 5a shows an SEM image of a 6H-N specimen etched for 4 hrs at these conditions (125 °C, 35 mT, 40% Ar). The sidewall slope was approximately 90°, as predicted; however excessive polymer generation resulted in rougher surfaces than expected. Another sample was etched using the same conditions but at 180 °C. This sample was found to have smoother surfaces, as can be seen from figure 5b. The higher temperature may have the beneficial effect of increasing the reactivity of etch products and reducing adsorption of species on the surface, thereby preventing polymer buildup. Similarly good results were obtained when this optimized process was used to etch 4H-P SiC. Etch rates and Ra for 4H-P/6H-N SiC were 0.7/0.7 µm/min and 290/40 Å.

CONCLUDING REMARKS

The effects of chuck temperature, chamber pressure, and O_2 and Ar concentrations on the DRIE characteristics of 6H and 4H SiC were studied. Key factors to obtain minimal microtrenching (high temperature), vertical sidewalls (high pressure), smooth surfaces (Ar incorporation) were determined. An optimized process for DRIE of 6H and 4H SiC meeting the defined requirements was found. The repeatability of this process is now being studied.

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