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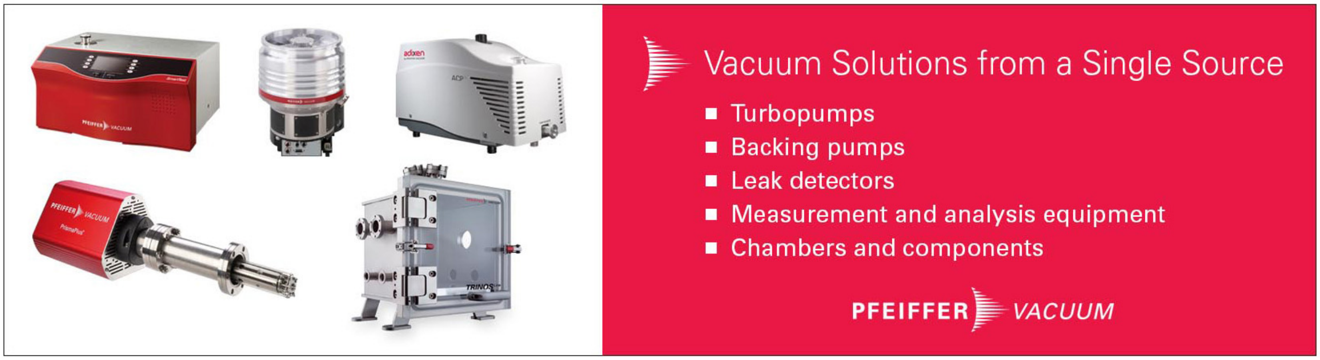

Direct epitaxial growth of submicron-patterned SiC structures on Si(001)

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We report on the direct epitaxial growth of submicron-patterned SiC structures on Si(001) substrates using supersonic molecular jet epitaxy and resistless e-beam lithography. Prior to SiC film growth, an electron beam was scanned on hydrogen-passivated Si substrates in order to produce silicon oxide lines with widths ⩾60 nm. The SiC nucleation and growth rates were significantly reduced on the oxidized regions during the subsequent supersonic jet epitaxial growth of SiC, which yielded epitaxial, submicron-patterned SiC films. The effects of the growth temperature and e-beam dose on the SiC growth and pattern linewidth are discussed. © *1999 American Vacuum Society.* [S0734-211X(99)00906-3]

I. INTRODUCTION

There is much interest in the growth and processing of wide-band-gap semiconductors for high-temperature electronic device applications.[1–4] For device fabrication, submicron patterning of materials is increasingly needed. However, the high chemical stability of the wide-band-gap semiconductors such as SiC and GaN makes it difficult to use conventional etching techniques.[5] Alternative microfabrication techniques have been developed using plasma-based reactive ion etching (RIE) to overcome the high chemical stability.[5,6] However, RIE for SiC often results in surface damage as well as contamination from the cathode material through a micromasking effect.[6]

The residual contamination and material degradation caused by RIE can be eliminated by selective area epitaxial growth.[7] For selective area epitaxial growth of SiC, conventional chemical vapor deposition has been employed using a submicrometer-thick silicon oxide layer to impede nucleation.[8,9] However, high growth temperatures above 1000 °C are required, which resulted in significant degradation of the microstructures.[9] The degradation is more severe for a thinner silicon oxide mask layer, which results in difficulty in making submicron-scale structures. This article reports on the direct fabrication of SiC microstructures with resolution ⩾130 nm using supersonic molecular jet (SMJ) epitaxy and resistless e-beam lithography.

The resistless patterning of semiconductor surfaces is of particular interest for developing all-dry fabrication methods for thin-film structures.[10–12] For the resistless patterning, scanning electron-beam lithography of a surface hydride layer adsorbed on silicon has been developed.[13] The combination of hydride patterning and SMJ growth prevents exposure of the substrate to atmospheric and process-related contaminants and degradation of the microstructures due to the contaminants or attempts at surface cleaning.

SMJ epitaxy has been employed as a hyperthermal growth technique to reduce the SiC growth temperature.[14,15] During supersonic free-jet expansion into a vacuum chamber, individual gas molecules undergo a large number of collisions and in this process the enthalpy of the gas decreases while the kinetic energy of the molecules increases. Higher kinetic energies of film-growth molecules can be obtained by seeding a heavy reactant methylsilane gas (SiH_3CH_3) in a light carrier gas (He). In the expansion, both components of the gas mixture are accelerated to nearly the same velocity and the kinetic energy of the heavy reactant molecules increases. For the 10% SiH_3CH_3 in the He mixture used in this research, the average translational kinetic energy of SiH_3CH_3 molecules is calculated to be 0.36 eV. Due to the hyperthermal kinetic energy and high central flux of the SiH_3CH_3 molecules, the growth temperature for SiC films could be reduced as low as 700 °C.[16]

II. EXPERIMENTAL PROCEDURE

The hydrogen-passivated Si substrates were prepared using the following procedure. The Si substrates were degreased using trichloroethylene, acetone, and isopropylalcohol. After rinsing with deioinized water, the substrates were oxidized in an ultraviolet ozone reactor and then were etched in 4% dilute HF for 30 s to produce a hydride-passivated surface immediately prior to loading them in the scanning electron microscope (SEM) chamber.

Submicron selectivity for the growth of SiC was created by resistless electron-beam lithography. The e-beam lithography system consists of a commercial SEM configured for external scanning mode. The typical beam currents used were on the order of 40–60 pA. The electron dose was in the range of 0.8–1.3 μC/cm. The pressure in the chamber was about 5×10^{-7} Torr. The electron beam was scanned over the hydrogen-passivated Si(100) substrates, resulting in local desorption of hydrogen and subsequent formation of a thin silicon oxide layer due to the presence of residual water vapor in the chamber.[13]

After patterning, the samples were immediately loaded into the SMJ growth chamber. The hydrogen on the Si surface was desorbed by thermal annealing above 500 °C. The SiC films were grown on the patterned Si(001) substrates in an ultra-high-vacuum chamber with a typical base pressure of mid-10^{-9} Torr. The supersonic molecular jet of SiH_3CH_3 was generated via a nozzle with its orifice diameter of 200

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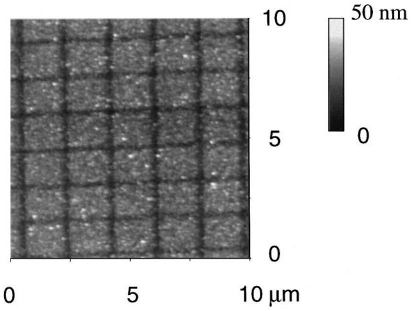

FIG. 1. AFM image of patterned SiC structures grown on Si(100). The microstructures were grown at 750 °C for 15 min at a pressure of 0.5 mTorr.

μm and normally incident on the substrate. During the deposition, the main chamber pressure was monitored using a capacitance manometer. A constant beam flux was maintained at the chamber pressure of 0.5–1.0 mTorr using a needle valve. The substrates were radiatively heated using a BN-coated ceramic heater supported on a Ta holder. The growth temperatures investigated in this research ranged from 680 to 900 °C as monitored by an optical pyrometer.

III. RESULTS AND DISCUSSION

The surface topography of submicron SiC structures was investigated using atomic force microscopy (AFM). After the thin silicon oxide mask pattern was fabricated using an e-beam line dose of 0.9 μC/cm on the H-passivated Si substrate, the SiC film (Yi056) was grown on the patterned Si(100) at 750 °C for 15 min at a pressure of 0.5 mTorr. As shown in Fig. 1, well-fabricated grid SiC microstructures were obtained using the resistless electron-beam lithography and SMJ. The typical thickness (height) of the structures is 10–20 nm and the average surface roughness is 3–4 nm. The AFM image structure with continuous lines is as narrow as 200 nm, as Fig. 1 shows. The growth selectivity results from the lower nucleation and growth rate of SiC on the silicon oxide layer than on Si. Similar behavior was previously observed in the Ge/Si system.[7]

The linewidths of the SiC microstructures were controlled by the e-beam dose. Figure 2 shows AFM images of the SiC microstructures fabricated with doses of 0.8 (Yi052) and 1.3 (Yi055) μC/cm. For the higher dose, the linewidth broadened to 350 nm, which may be due to increased hydrogen desorption caused by increased heating. The variation of the linewidth results presumably from the ±5% instability of the patterned e-beam current.

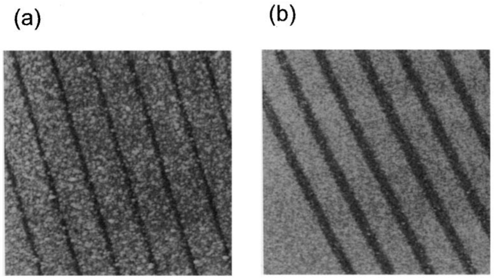

FIG. 2. AFM image of SiC microstructures grown on Si(100) (figure dimension=8.5×8.5 μm^2). Patterning using electron line doses of (a) 0.8 and (b) 1.5 μC/cm resulted in linewidths of 130 and 350 nm, respectively.

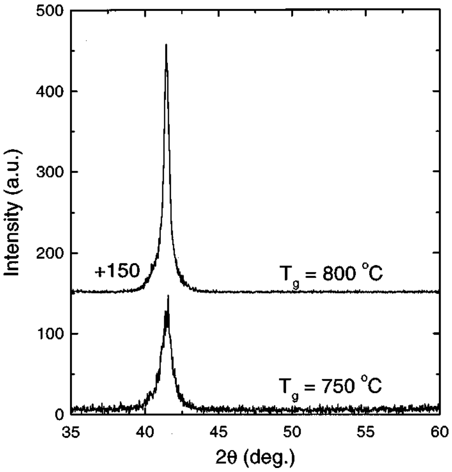

FIG. 3. Typical XRD data of SiC films grown at temperatures (T_g) of 750 °C and 800 °C. Only an XRD peak of 3C–SiC(200) is shown.

The crystallinity of the as-grown patterned films was characterized using x-ray diffraction (XRD) measurements. Figure 3 shows typical XRD results for the films grown at 750 and 800 °C. The only XRD peak observed was at 41.4°, corresponding to 3C–SiC(002). This shows that the submicron-patterned SiC has crystallized at 750 °C, with the low-temperature crystallization presumably assisted by the hyperthermal kinetic energy of the supersonic jet.[16]

The submicron patterns of 3C–SiC on Si(100) depended significantly on the growth temperature. At temperatures below 700 °C, the patterns were difficult to find because the films were very thin everywhere. Increasing the growth temperature to 750–780 °C produced thicker films with easily observable patterns. This suggests that the silicon oxide patterns generated by resistless lithography maintain their integrity up to nearly 800 °C. However, SiC films grown above 800 °C did not show any pattern. The degradation of the patterns at temperatures above 800 °C occurs presumably by decomposition of silicon oxide and formation of volatile SiO. According to Edgar *et al.*,[9] a 284-nm-thick silicon oxide layer was degraded at 950 °C. In our experiments, the degradation of the much thinner (~few nm) oxide layers started at the relatively low temperature of 800 °C.

IV. CONCLUSIONS

In conclusion, direct epitaxial growth of submicron-patterned SiC was carried out on Si(001) using supersonic molecular jet epitaxy and resistless electron-beam lithography. The optimum growth temperature range was from 730 to 780 °C. For temperatures above 800 °C, the microstructures could not be fabricated due to degradation of the silicon oxide mask. SiC microstructures with linewidths of 130–350 nm were achieved using scanned e-beam doses of 0.8–1.3 μC/cm.

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[1] J. H. Edgar, J. Mater. Res. **7**, 235 (1992).
[2] H. Morkoç, S. Strite, G. B. Gao, M. E. Lin, B. Sverdlov, and M. Burns, J. Appl. Phys. **76**, 1363 (1994).
[3] H. Matsunami and T. Kimoto, Mater. Sci. Eng., R. **20**, 125 (1997).
[4] T. Fuyuki, T. Hatayama, and H. Matsunami, Phys. Status Solidi B **202**, 359 (1997).
[5] P. H. Yih, V. Saxena, and A. J. Steckl, Phys. Status Solidi B **202**, 605 (1997).
[6] A. J. Steckl and P. H. Yih, Appl. Phys. Lett. **60**, 1966 (1992).
[7] G. Eres, F. Y. C. Hui, T. Thundat, and D. C. Joy, J. Vac. Sci. Technol. B **15**, 2934 (1997).
[8] Y. Ohshita and A. Ishitani, J. Appl. Phys. **66**, 4535 (1989); Y. Ohshita, Appl. Phys. Lett. **57**, 605 (1990).
[9] J. H. Edgar, Y. Gao, J. Chaudhuri, S. Cheema, S. A. Casalnuovo, P. W. Yip, and M. V. Sidorov, J. Appl. Phys. **84**, 201 (1998).
[10] J. W. Lyding, T.-C. Shen, J. S. Hubacek, J. R. Tucker, and G. C. Abeln, Appl. Phys. Lett. **64**, 2010 (1994).
[11] K. Masu and K. Tsubouchi, J. Vac. Sci. Technol. B **12**, 3270 (1994).
[12] E. S. Snow and P. M. Campbell, Appl. Phys. Lett. **64**, 1932 (1994).
[13] F. Y. C. Hui, G. Eres, and D. C. Joy, Appl. Phys. Lett. **72**, 341 (1998).
[14] L.-Q. Xia, M. E. Jones, N. Maity, and J. R. Engstrom, J. Vac. Sci. Technol. A **13**, 2651 (1995).
[15] S. A. Ustin and W. Ho, Mater. Res. Soc. Symp. Proc. **482**, 313 (1998).
[16] G.-C. Yi, G. Eres, and D. H. Lowndes (unpublished).