

Vapor-phase epitaxial growth on porous 6H-SiC analyzed by Raman scattering

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SiC vapor-phase epitaxy on porous silicon carbide (PSC) substrates formed by electrochemical anodization is reported. Raman scattering indicates that the polytype of the optically smooth SiC grown on PSC formed in both *p*-type and *n*-type 6H substrates is 6H. The Raman scattering selection rules in these films are the same as those observed in the bulk substrate and epilayers grown on bulk, indicating high crystalline quality. The formation of epitaxial 6H-SiC on porous 6H-SiC may open up new possibilities for dielectric device isolation, fabrication, and epitaxial lift-off. © 2000 American Institute of Physics. [S0003-6951(00)01026-3]

Silicon carbide, with its wide band gap, high saturation drift velocity, and high breakdown electric field, has long been recognized as an important semiconductor material for use in high temperature and high power applications, including power transistors, optoelectronics, and sensors. SiC has also been identified as the substrate of choice for the heteroepitaxial growth of important materials, such as GaN and AlN. In particular, the 6H polytype of SiC is of great interest in each of these applications because of its very large band gap and thermal conductivity. In recent years, its use in devices has increased due to better availability of larger sized wafers and higher quality 6H-SiC substrates and epitaxy. The processing of SiC, however, remains difficult, since an efficient wet etchant of SiC does not exist. Sometimes the use of porous materials opens up new processing routes. For example, this has been demonstrated with the formation of low-defect, device-quality homoepitaxial Si,^{1,2} and heteroepitaxy, including GaAs,³ Ge,⁴ and 3C-SiC⁵ on porous Si. SiC is known to dissolve electrochemically and also form porous silicon carbide (PSC), the structure of which resembles porous Si. In this letter, the epitaxial growth of SiC on porous 6H-SiC formed in *p*- and *n*-type substrates is reported.

PSC films were formed in selected regions of the (000 $\bar{1}$) face of 6H-SiC substrates (oriented 3.5° off axis towards $\langle 11\bar{2}0 \rangle$) by galvanostatic electrochemical anodization in an aqueous and ethanolic hydrofluoric acid solution in the dark for all of the samples except one which was prepared with ultraviolet (UV) light (sample N2). The substrate for porous layer samples P1-P4 was *p* type, $N_A - N_D \sim 8.8 \times 10^{17} \text{ cm}^{-3}$, and the substrate for porous layer samples N1 and N2 was *n* type, $N_D \sim 5 \times 10^{17} \text{ cm}^{-3}$. The PSC layers

formed from *p*-type SiC were 70%–75% porous and 1–2 μm thick; these PSC layers were respectively formed with 5, 10, 20, and 20 mA/cm^2 current density for 50, 30, 5, and 15 min. The PSC layers formed from *n*-type SiC were 50%–60% porous and 3–5 μm thick; PSC layers N1 and N2 were respectively formed with 5 mA/cm^2 under dark conditions for 30 min and 5 mA/cm^2 under UV illumination. Electron diffraction from PSC formed in both *n*-type and *p*-type 6H-SiC has shown that PSC is monocrystalline and of the 6H polytype; still, the morphologies of these two types of PSC are different.^{6,7}

Approximately 6 μm thick *n*-type SiC was regrown on all samples by vapor-phase epitaxy^{8,9} during a single run, and all of the regrown samples were optically smooth. A cross-section scanning electron micrograph (SEM) of the cleaved edge of regrown SiC on porous layer N1 atop the *n*-type substrate is shown in Fig. 1. The overgrown layer, macroporous layer, and substrate are discernable, and the surface morphology is smooth (for both N1 and N2). SEMs

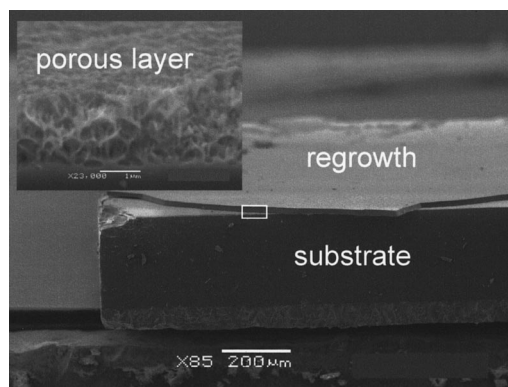


FIG. 1. Scanning electron micrograph of cleaved edge of sample N1 showing overgrown layer, porous layer, and substrate. The inset shows an enlarged view of the porous layer.

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of SiC regrown on the *p*-type porous layers are similar except the pores in the PSC are much finer, as expected. Also, the surface of the regrown SiC atop the P1 layer is smooth; there are hillocks on the surface of layers grown on P2–P4.

Raman scattering has been shown to be an effective, nondestructive means of polytype identification in SiC, and in particular, in differentiating the 6H, 4H, and 3C polytypes.^{10–13} The 488.0 nm line of an Ar-ion laser was used for Raman microprobe analysis; the beam was focused to a diameter of approximately 2 μm and scattered light was collected in backscattering configuration. Initially, polarized Raman spectra were acquired normal to the plane of the regrown layers with $z(x,x)\bar{z}$ and $z(x,y)\bar{z}$ configurations, with z parallel to the c axis. Since 4880 \AA penetrates deeply into the crystalline SiC¹⁴ (\gg epilayer thickness $\sim 6 \mu\text{m}$) and the highly scattering porous layers are relatively thin, some contribution from the porous layer and the underlying substrate is possible even with microprobe analysis. Therefore, all regrown samples were cleaved and examined in cross section with polarized $y(x,x)\bar{y}$, $y(x,z)\bar{y}$, and $y(z,z)\bar{y}$ configurations. With the laser power of 3.4 mW there was no observable shifting and broadening due to laser heating. The backscattered light was dispersed by a 0.6 m single dispersive/double subtractive monochromator and collected by a cooled charge-coupled device array, leading to a resolution of the Raman spectra better than 1 cm^{-1} . Peak positions were determined by Lorentzian fitting.

Figure 2 shows the polarized $y(x,x)\bar{y}$, $y(x,z)\bar{y}$, and $y(z,z)\bar{y}$ Raman spectra of (a) a 6H substrate and (b) a 4H substrate in the cross-section configuration. The 6H–SiC polytype substrate has transverse optical (TO) modes at 767.7 and 788.8 cm^{-1} of E_2 symmetry for $y(z,z)\bar{y}$; a TO mode at 788.0, an axial mode at 888.1 cm^{-1} (inset) and a longitudinal optical (LO) mode at 970.9 cm^{-1} (inset) each with A_1 symmetry for $y(x,x)\bar{y}$; and a TO mode at 798.3 cm^{-1} of E_1 symmetry for $y(x,z)\bar{y}$. The two peaks seen for $y(z,z)\bar{y}$ are formally forbidden, but are observed here even for the substrate. They are allowed and are very strong for $z(x,x)\bar{z}$ and $z(x,y)\bar{z}$ backscattering normal to overgrown layer surface (not shown). The Raman spectrum of 4H–SiC has TO modes at 776.7 and 783.9 cm^{-1} of E_2 symmetry (expected to be forbidden, but seen even for the substrate) for $y(z,z)\bar{y}$; a TO mode at 783.2 cm^{-1} , an axial mode at 838.9 cm^{-1} (inset) and a LO mode at 970.7 cm^{-1} (inset) of A_1 symmetry for $y(x,x)\bar{y}$; and a TO mode at 798.5 cm^{-1} of E_1 symmetry for $y(x,z)\bar{y}$. Polarized $z(x,x)\bar{z}$, $z(x,y)\bar{z}$, $z(x',x')\bar{z}$, and $z(x',y')\bar{z}$ Raman spectra (normal to the top surface) of a 1 μm thick as-deposited (100)-oriented epilayer of 3C–SiC–on–Si (not shown) show that the 3C polytype has an LO mode of T_2 symmetry at 972.0 cm^{-1} which appears for $z(x,x)\bar{z}$ and $z(x',y')\bar{z}$, but not for $z(x,y)\bar{z}$ and $z(x',x')\bar{z}$. [(x',y') are rotated 45° from (x,y).] Given the cubic symmetry of 3C–SiC, this mode would also be seen for the $y(x,x)\bar{y}$ and $y(z,z)\bar{y}$ configurations examined in the cross-section measurements. The TO mode of T_2 symmetry at 796 cm^{-1} is forbidden in backscattering configuration and is not observed. These Raman shifts of each polytype are consistent with published values.^{10–12} Clearly, these polytypes are distinguishable in these polarization configurations.

Polarized Raman spectra of SiC regrown on the bulk

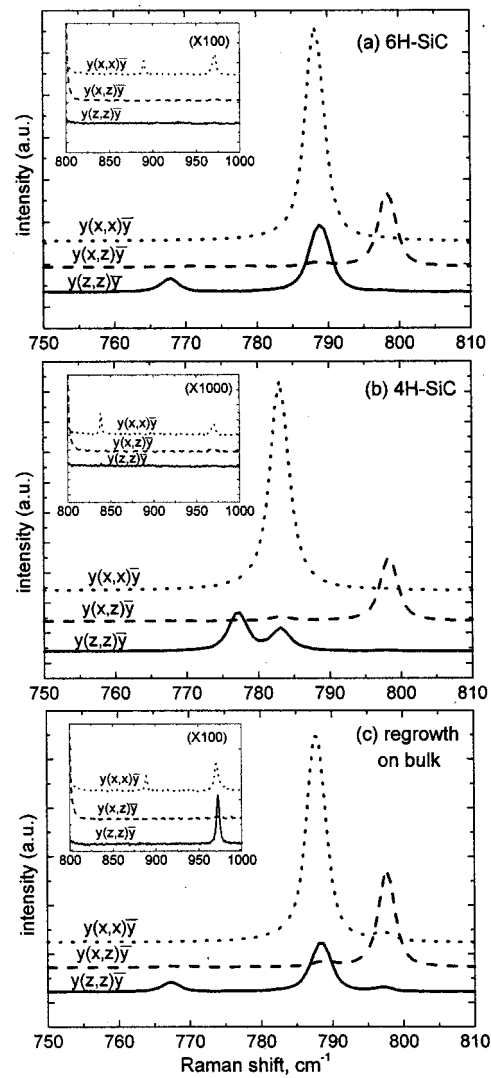


FIG. 2. Polarized Raman spectra of (a) bulk 6H–SiC, (b) bulk 4H–SiC, and (c) regrown SiC on bulk *n*-type 6H–SiC with $y(z,z)\bar{y}$ (solid lines), $y(x,z)\bar{y}$ (dashed lines), and $y(x,x)\bar{y}$ (dotted lines) configurations. The insets show the weaker peaks at higher frequencies.

n-type substrate are plotted in Fig. 2(c); the spectra of the regrowth on the bulk *p*-type substrate are identical. These Raman spectra are identical to those of the bulk 6H–SiC for both *n*- and *p*-type material.

Figure 3 shows polarized Raman spectra of SiC regrown on porous layer P1 taken (a1) near the top surface of the epilayer, (a2) near the center of the epilayer, and (a3) in the underlying bulk *p*-type substrate, and of sample N2 collected in a similar manner (b1)–(b2) and in the underlying *n*-type substrate (b3). The Raman spectra of the SiC layers grown on porous layers P1–P4 and N1–N2 all look the same. They all appear to be essentially identical to those of the 6H substrate and the SiC regrown on the 6H substrate (even in the spectral range of the inset in Fig. 2(a)—not shown). Phonon peaks seen in 4H–SiC and 3C–SiC are not seen in any of the regrown samples. This suggests that the SiC regrown on each porous layer (and on the 6H substrate itself) is a high quality 6H–SiC epilayer. One difference in the spectra of each epilayer overgrown on a porous layer is the Raman peaks all become a bit weaker near the top surface of the overgrown layer (with the focused beam still entirely hitting

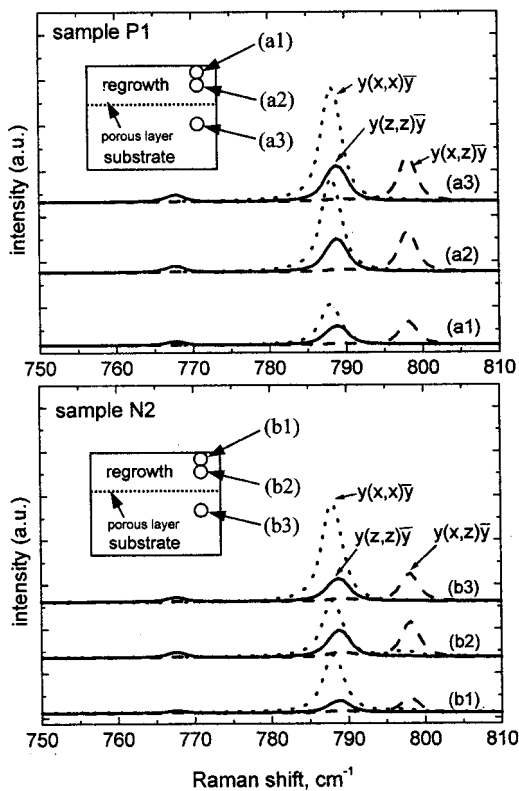


FIG. 3. Cross-section polarized Raman spectra of SiC regrown on porous (a) sample P1, and (b) sample N2. The solid, dashed, and dotted lines denote the $y(z,z)\bar{y}$, $y(x,z)\bar{y}$, and $y(x,x)\bar{y}$ configurations, respectively. The spectra have been taken near the top surface in (a1) and (b1), near the center of the regrown layer in (a2) and (b2), and within the underlying bulk substrate in (a3) and (b3).

the layer) while the peaks decrease only a little near the top of the epilayer grown directly on the substrate.

The successful growth of 6H-SiC on PSC, along with the selective oxidation and removal of PSC, suggests new ways to process porous/crystalline 6H-SiC structures to achieve dielectric isolation, fabricate micromachined structures such as microelectromechanical structures, and achieve lift-off for subsequent wafer bonding, similar to existing methods involving porous Si.¹⁵ The successful growth of 6H-SiC on PSC suggests the possibility for growth of other

materials on PSC including GaN and other III-N materials, and the application of the above methods to these other materials.

In summary, epitaxial growth of high-quality monocrystalline SiC on porous silicon carbide is reported. Raman scattering has shown that the epitaxial growth of optically smooth SiC on porous silicon carbide formed in both *p*-type and *n*-type 6H substrates is of the 6H polytype. This opens a new process route for preparing dielectrically isolated and micromachined structures and devices in 6H-SiC. Further characterization of these layers and optimization of the crystalline quality by varying substrate choice and preparation conditions are underway.

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