Physics

Physics Research Publications

Purdue University *Year* 2006

Residual strains in cubic silicon carbide measured by Raman spectroscopy correlated with x-ray diffraction and transmission electron microscopy

M. A. Capano B. C. Kim A. R. Smith

E. P. Kvam S. Tsoi A. K. Ramdas

This paper is posted at Purdue e-Pubs.
http://docs.lib.purdue.edu/physics_articles/331

Residual strains in cubic silicon carbide measured by Raman spectroscopy correlated with x-ray diffraction and transmission electron microscopy

M. A. Capano[a)] and B. C. Kim
School of Electrical and Computer Engineering, Purdue University, West Lafayette, Indiana 47907

A. R. Smith and E. P. Kvam
School of Materials Engineering, Purdue University, West Lafayette, Indiana 47907

S. Tsoi and A. K. Ramdas
Department of Physics, Purdue University, West Lafayette, Indiana 47907

(Received 2 June 2006; accepted 12 July 2006; published online 25 October 2006)

Cubic (3*C*) silicon carbide (SiC) epilayers grown on Si substrates by chemical vapor deposition, characterized using transmission electron microscopy (TEM), high-resolution x-ray diffraction (HRXRD), and Raman spectroscopy, reveal the presence of biaxial in-plane strain. Defect (stacking faults, twins, dislocations) distributions revealed by TEM are correlated with peak widths obtained from HRXRD measurements and Raman shifts of the zone center longitudinal optical phonon line. TEM showed defect densities decreasing with increasing distance from SiC/Si interface as the lattice mismatch stress is relaxed. Structural defect densities show the most significant reduction within the first 2 μm of the epilayer. TEM observations were correlated with a monotonic decrease in HRXRD peak width (full width at half maximum) from 780 arc sec (1.5 μm thick epilayer) to 350 arc sec (10 μm thick epilayer). Raman spectroscopy indicates that the residual biaxial in-plane strain decreases with increasing epilayer thickness initially, but becomes essentially constant between 6 and 10 μm. Differences in the observed behavior between HRXRD and Raman spectroscopy are discussed in terms of the fundamental interactions of incident radiation with the 3*C*-SiC epilayers. © *2006 American Institute of Physics*. [DOI: 10.1063/1.2357842]

I. INTRODUCTION

Semiconductor epilayers grown on substrates with small to moderate lattice mismatch, studied as a function of epilayer thickness, reveal high crystallinity below the pseudomorphic limit, but contain high densities of dislocations and other structural defects when this limit is exceeded.[1,2] When the lattice parameter mismatch between the layer and the substrate is large ($>2\%$), the nature of these defects and their influence in regions well removed from the interface are issues of both scientific and technological importance. Such is the case for silicon carbide epilayers grown on Si substrates.[3]

Silicon carbide (SiC) is a promising semiconductor material for solid state electronics. The wide band gaps and large critical breakdown fields of its polytypes make SiC attractive for vertical power devices. For the most part, SiC electronics technology has concentrated on the use of one of the hexagonal polytypes (4*H*) of SiC because this polytype exhibits a band gap energy of 3.23 eV, while possessing a large critical breakdown field and high thermal conductivity. 4*H*-SiC is commercially available as a substrate material.

In comparison with 4*H*-SiC, the cubic polytype (3*C*-SiC) has the advantage of an isotropic electron mobility of 1000 $cm^2/V\,s$ and is the thermodynamically stable polytype of SiC. Consequently, it is the 3*C* polytype which grows on Si substrates. Metal-oxide-semiconductor field-effect transistors built on 3*C*-SiC epilayers have shown encouraging on-state characteristics, with inversion channel mobilities as high as 221 $cm^2/V\,s$ when the epilayer is grown on a 3*C*-SiC substrate[4] and 165 $cm^2/V\,s$ when the epilayer is grown on Si(001).[5] Homoepitaxial growth would be the preferred technical approach with the end goal of realizing 3*C*-SiC devices, but 3*C*-SiC substrates[6] are not widely available and are not commercially viable. Therefore, the most advantageous prospect for realizing 3*C*-SiC devices is to grow epilayers of this polytype on inexpensive Si substrates to take advantage of the physical properties of 3*C*-SiC. The mobility data, coupled with recent advancements in 3*C*-SiC epitaxial growth and processing technology, have stimulated renewed interest in 3*C*-SiC materials and devices. It is with this perspective that structural aspects of 3*C*-SiC epilayers are investigated. The objectives here are to investigate defects and residual strains in 3*C*-SiC epilayers and to evaluates the application of Raman spectroscopy for the purpose of investigating strains in this material.

Knowledge of structural defects traditionally is gained through material characterization using transmission electron microscopy (TEM) and x-ray diffraction. X-ray diffraction provides high strain sensitivity, but sampling volumes are large. Complementary information is available using TEM where spatial resolution can be very high, but strain sensitivity is poor because of the relatively large divergence of the dispersion surface. TEM analysis is also destructive. An analytical technique which provides reasonable spatial resolution and strain sensitivity, so that residual strains in 3*C*-SiC epilayers can be profiled, would be a valuable asset in understanding the fundamental dynamics of defects and re-

[a)]FAX: 1-765-494-3563; electronic mail: capano@purdue.edu

 © 2006 American Institute of Physics

Downloaded 09 Apr 2007 to 128.210.146.185. Redistribution subject to AIP license or copyright, see http://jap.aip.org/jap/copyright.jsp

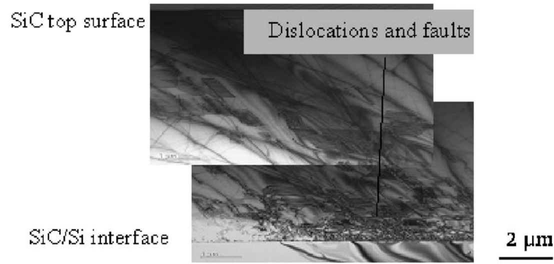

FIG. 1. TEM micrograph of 10 μm thick 3*C*-SiC epilayer.

sidual strain in 3*C*-SiC epilayers. Raman shifts of the longitudinal and transverse optical phonons at the origin of the Brillouin zone (Γ), the zone center optical phonons, have been demonstrated to depend on hydrostatic strain in bulk 3*C*-SiC.[7] With its large band gap, first order Raman spectra of SiC can be obtained using laser radiation in the visible spectral range at reduced incident intensities, thus preventing significant heating of samples. In this letter, Raman characterization of 3*C*-SiC epilayers on (001) Si is presented in the context of the spatial and depth resolutions of the strains present and are correlated with TEM and x-ray characterization.

II. EXPERIMENT

3*C*-SiC *p*-type epilayers of thicknesses of 1.5, 3, 6, and 10 μm are grown on 2.5° off-axis Si(001) substrates using chemical vapor deposition (CVD). The growth temperature is 1350 °C in 100 mbars of hydrogen [25 SLM (standard liters per minute)], silane [6 SCCM (cubic centimeter per minute at STP)], and propane (3 SCCM). A carbonization step is used prior to growth, which consists of flowing propane (3 SCCM) in hydrogen at 1250 °C/300 mbars for 2 min. Trimethylaluminum (TMA) is used as the *p*-type dopant. Structural analysis of epilayers is performed using TEM, high-resolution x-ray diffractometry (HRXRD), and Raman spectroscopy. Raman spectra, recorded in the backscattering geometry at room temperature, are excited by laser radiation from an Ar^+ laser at 514.5 nm. The incident radiation is focused to a 100 μm spot on the sample surface, and its intensity is reduced to 300 W/cm^2 to minimize sample heating. No change in the Raman shift is observed for intensities below 300 W/cm^2, indicating insignificant local heating. The scattered radiation is analyzed by a double-grating SPEX spectrometer and detected with a thermoelectrically cooled photomultiplier tube (PMT) operating in the standard photon counting mode.

III. RESULTS AND DISCUSSION

Cross-sectional TEM imaging, such as these displayed in Fig. 1, reveals that the total defect densities (comprised of stacking faults, twins, and dislocations) decrease with increasing distance from the SiC/Si interface as the lattice mismatch induced stress is relaxed. Structural defect density shows the most significant reduction in the first 2 μm of growth. Further evidence of the reduction in defect densities as thickness increases is the monotonic decrease in the (004) x-ray diffraction peak full width at half maximum (FWHM),

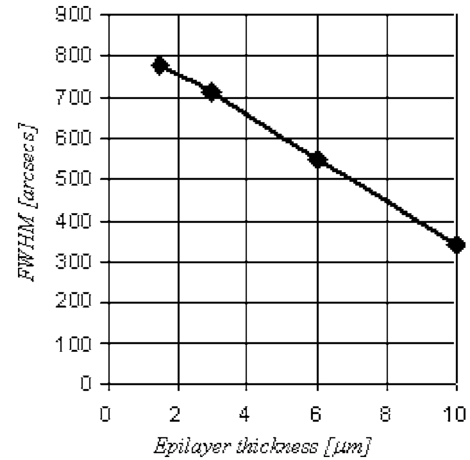

FIG. 2. HRXRD (004) peak width as function of epilayer thickness.

shown in Fig. 2, from 780 arc sec (1.5 μm epilayer) to 350 arc sec (10 μm epilayer). Note that the theoretical FWHM for SiC is about 12 arc sec, which implies further that improvement in epilayer quality is possible because the measured FWHMs are much greater than the expected FWHM calculated for a perfect SiC crystal. Simultaneous with the reduction in FWHM is an increase in peak intensity measured from the (004) diffraction peaks. Measured peak intensities (in counts per second) are 13 982 cps (10 μm), 7316 cps (6 μm), 2452 cps (3 μm), and 1336 cps (1.5 μm). Peak intensities are expected to increase as the quality of the epilayer improves because fewer defects are present to perturb constructive interference of x rays diffracted from (004) atomic planes. The observed trend in peak intensity provides additional confirmation that defect densities decrease as the distance from the SiC/Si interface increases and that scattering from defects is diminishing as the layer thickness increases.

Figure 3 compares Raman spectra of a 1.5 μm thick epilayer, a 10 μm thick epilayer, and a 10 μm thick freestanding epilayer with the Si substrate removed by a KOH etching solution. Biaxial in-plane strain within the epilayers

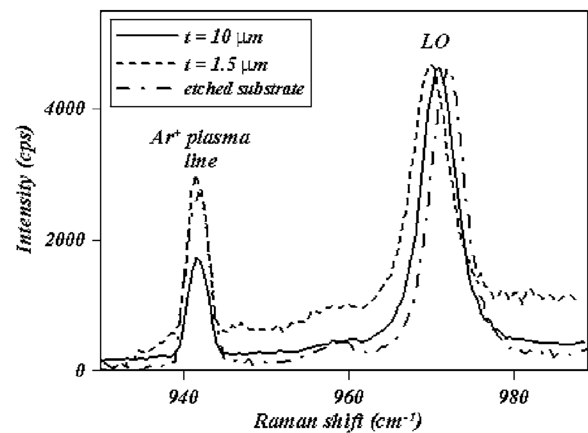

FIG. 3. Raman spectra from 1.5 and 10 μm thick 3*C*-SiC epilayers as well as a free epilayer with etched Si substrate. Ar^+ plasma line at 18 488.22 cm^{-1} (with an apparent Raman shift of 941.5 cm^{-1}) suitable for calibration of Raman spectra was fitted with the Gaussian line shape, whereas the longitudinal optical (LO) Raman peak was fitted with the Voigt line shape. cps ≡ counts per second.

Downloaded 09 Apr 2007 to 128.210.146.185. Redistribution subject to AIP license or copyright, see http://jap.aip.org/jap/copyright.jsp

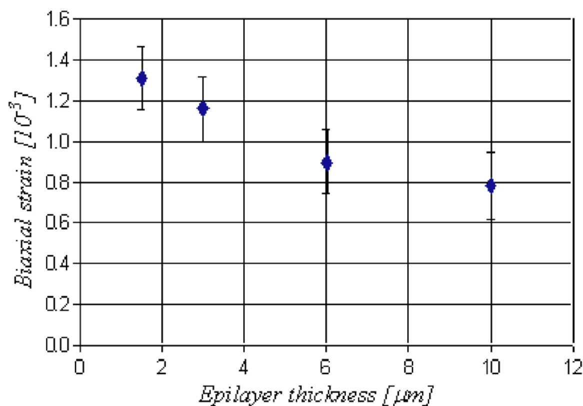

FIG. 4. Biaxial in-plane strain in 3*C*-SiC epilayers.

is determined from the position of the longitudinal optical (LO) phonon peak using the slope of the linear dependence of Raman shift on strain,[8] and its value of 972 cm^{-1} is observed in the present study from the freestanding epilayer. The variation of biaxial in-plane strain with epilayer thickness is shown in Fig. 4. Figure 4 shows that the biaxial strain initially decreases, then is nearly constant for 6 and 10 μm thick epilayers. This behavior differs from the trend observed from high-resolution x-ray diffraction in Fig. 2. One reason for the difference in the observed behavior relates to differences in how deeply each technique probes the 3*C*-SiC epilayer. The x-ray penetration depth is tens of microns when limited by photoelectric absorption, and may be further limited by primary extinction effects as the epilayer quality improves. Diffraction is attenuated least from the surface and the measured intensities fall off exponentially with depth. Considering these factors, a reasonable estimate of the x-ray "sampling" depth in these specimens is about 5–10 μm. For the sub-band-gap (514.5 nm) laser line used in Raman experiments, the sampling depth is larger than the x-ray sampling depth. The biaxial strain Raman measurement on a 10 μm thick epilayer is more strongly influenced by the highly strained, highly defective regions close to the 3*C*-SiC/Si interface than are the x-ray measurements from the same 10 μm thick epilayer. This conjecture is strongly supported by the observation of a strong Raman line at 521 cm^{-1}, associated with the zone center optical phonon of the Si substrate, in all samples investigated. Furthermore, Raman spectroscopy and HRXRD probe different physical features. The so-called double-crystal x-ray measurements used to collect FWHM data are sensitive to both normal strains (those strains with displacements perpendicular to the interface) and lattice tilts. It is possible to separate the effects of normal strains from lattice tilts using triple-axis techniques, but that is not relevant to the present discussion. Raman spectroscopy is sensitive to biaxial strains for a phonon propogating perpendicular to the interface. Normal and in-plane strains are related through the Poisson ratio, but the insensitivity of Raman spectroscopy to lattice tilts suggests that differences between x-ray and Raman data are to be expected.

IV. CONCLUSIONS

In closing, this article reports structural data pertaining to 3*C*-SiC grown on Si(001) substrates. Significant reduction in defect density within the first 2 μm of epigrowth is observed. Continued improvement in epilayer quality is seen as thickness increases up to 10 μm. This is relevant for device fabrication and provides valuable insights regarding the appropriate epilayer thickness for either vertical or lateral device structures. Raman spectroscopy is shown to be a valuable tool in the characterization of epilayer crystalline quality and residual strains. Differences between data collected by Raman spectroscopy and high-resolution x-ray diffraction are explained in terms of the interaction of incident radiation and 3*C*-SiC. Raman spectroscopy is highly sensitive to residual strain and has a spatial resolution that is intermediate between transmission electron microscopy (low strain sensitivity) and high-resolution x-ray diffractometry (very high strain sensitivity) and is nondestructive.

ACKNOWLEDGMENTS

The authors wish to acknowledge support for this project from the Office of Naval Research (Grant No. N00014-03-1-0196) and the Indiana 21st Century Fund (Award No. 503040742). Two of the authors (A.K.R. and S.T.) received support from the National Science Foundation (DMR 0405082).

[1]J. W. Matthews and A. E. Blakeslee, J. Cryst. Growth **27**, 118 (1974).
[2]R. J. Thomas *et al.*, Phys. Rev. B **49**, 2181 (1994).
[3]H. Nakazawa, M. Suemitsu, and S. Asami, Mater. Sci. Forum **338–342**, 269 (2000).
[4]K. K. Lee *et al.*, IEEE Electron Device Lett. **24**, 466 (2003).
[5]J. Wan, M. A. Capano, M. R. Melloch, and J. A. Cooper, IEEE Electron Device Lett. **23**, 482 (2002).
[6]H. Nagasawa, T. Kawahara, and K. Yagi, Mater. Sci. Forum **389–393**, 319 (2002).
[7]D. Olego, M. Cardona, and P. Vogl, Phys. Rev. B **25**, 3878 (1982).
[8]S. Rohmfeld, M. Hundhausen, L. Ley, C. A. Zorman, and M. Mehegany, J. Appl. Phys. **91**, 1113 (2002).

Downloaded 09 Apr 2007 to 128.210.146.185. Redistribution subject to AIP license or copyright, see http://jap.aip.org/jap/copyright.jsp