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Anomalous lattice relaxation mechanics in ZnO/SiC heterostructure

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Anomalous lattice relaxation mechanics in ZnO epitaxy deposited on 6H-SiC substrates has been addressed. Thickness-dependent ZnO epitaxy showed that the lattice/strain relaxes into two steps for the layer thicknesses of (i) 10–20 nm, after overcoming the critical thickness of <6 nm and (ii) 480–1000 nm, after the complete strain relaxation. This lattice relaxation contrast has been represented with a schematic model by emphasizing on the tensile and compressive strains associated with the lattice mismatches in ZnO/SiC heterostructure of <5%. These asymmetric consequences were found also in the optical and structural properties of ZnO layers by squeezing the bandgap energy and disordering the x-ray spectral broadening and reciprocal maps. These anomalous behaviors and the consequences have been attributed to the presence of local mosaics and competition of elastic and plastic deformation kinetics in ZnO epitaxy that finally released by inducing misfit and threading dislocations diffracted in transmission electron microscopy. © 2008 American Institute of Physics. [DOI: 10.1063/1.2919587]

I. INTRODUCTION

II-O materials have attracted a great deal of attention in research for its intense commercial interest in developing the practical semiconductor laser and light emitting diodes (LEDs) from visible to ultraviolet wavelength for the huge market needs. Recent progress in ZnO research has realized a very bright ZnO/ScAlMgO₄ heterostructure LED exhibiting room temperature (RT) light emission.¹ However, to optimize the ZnO-based LED structure, it is important to achieve lower threshold current density and higher differential quantum efficiency. These parameters strictly depend on the control of biaxial strain and stress kinetics of the respective materials system. A lot of efforts have been paid to clarify these limiting factors, especially for the GaN-based materials both theoretically and experimentally.^{2,3} However, few literatures were found on the strain and stress dynamics in ZnO hetero- or homostructures although this material has a potential for applications in a broad spectrum covering from nanoelectronics to spintronics and photonic devices.⁴

In principle, the biaxial strain appears in heterostructure due to two reasons: lattice (δ) and thermal (σ) mismatches. The δ appears while the lattice does not match with the underlayer, say ZnO/Al₂O₃ heterostructure ($\delta \sim 18\%$ that fully relaxes when the epilayer thickness becomes larger than several nanometers of critical thickness h_c). On the other hand, the σ occurs while the postdeposition substrate temperature cools down to RT; a compressive biaxial stress is induced along the out of plane because of the different thermal expansion coefficients in the heterostructures. To minimize these driving forces in ZnO materials system, lattice-matched and/or closely matched substrates can play a vital

role. It is noted that the strain induces, for example, dark-line defects and piezoelectric devices which tend to shorten the device lifetime by containing a huge density of misfit dislocations (MDs) and threading dislocations, including cracking.⁵ These defects, in principle, led to the failure of lasers and breakdown of *pn* junctions.⁶ To avoid/reduce these drawbacks, especially for the ZnO epitaxy, SiC may work out since the δ and σ are $\sim 5\%$ and $<1\%$, respectively. It is noted that SiC is also a wide bandgap material with excellent physical and electrical properties.⁷ Very recently, there are also some literatures demonstrated both theoretically and experimentally on the ZnO epitaxy deposited on SiC substrate⁸ that poise SiC's importance as a suitable substrate for the II-O materials.

To overcome these physical and technical limitations in ZnO materials system for device operation requires clear understanding of the lattice/strain dynamics. Recently, we have investigated the strain relaxation mechanism by varying the ZnO layer thickness grown on SiC substrates.⁹ During these studies, an anomalous lattice relaxation in ZnO epitaxy was found that consequences throughout the structural, surface, and optical properties of the ZnO/SiC heterostructures. In this paper, we have addressed this anomalous lattice/strain relaxation mechanics in ZnO/SiC heterostructures.

II. EXPERIMENTS

ZnO layers were deposited on 6H-SiC(0001) substrates by metalorganic chemical-vapor deposition. The details of chemical cleaning and processing have been discussed elsewhere.¹⁰ For ZnO deposition, the flow rate of diethylzinc (DEZn) was controlled by adjusting the flow rate of carrier gas N₂. The substrate temperature was then increased to the selective growth temperature of 500 °C for the successive growth of ZnO layers on 6H-SiC substrates. Prior to ZnO deposition, the DEZn was flown first for 40–80 s and then O₂

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was flown. The flow rate of DEZn was kept to 6 SCCM (SCCM denotes cubic centimeter per minute at STP), while the O_2 flow rate was 10 SCCM.

The c axis and a axis lengths of ZnO layers were determined by x-ray diffraction (XRD) by using the triple-axis ω - 2θ scans on the (002) and (205) reflections, respectively. The reciprocal space mapping was performed along the (002) and (006) planes of ZnO layers and SiC substrates, respectively. Two orthogonal $[11\bar{2}0]$ and $[11\bar{1}0]$ cross-sectional specimens were prepared for transmission electron microscopy (TEM) examination using the standard procedures and thinned by Ar^+ ion milling. Photoluminescence (PL) measurements were conducted by using the He-Cd laser for the excitation wavelength of 325 nm at liquid He temperatures.

III. RESULTS AND DISCUSSIONS

Figure 1 shows the a axis and c axis lengths with the increase in ZnO layers deposited on SiC substrates. For a few monolayers of ZnO, the a axis length is elongated parallel to the substrate surface for the values of 3.259 Å, while the c axis length is compressed for the values of 5.195 Å. In principle, the a axis length in primary stage of nucleation might be smaller since the a_{SiC} is 3.08 Å. This means that a complex and/or an interlayer may be rampant prior to the deposition of ZnO layers. It is well known that in the early stage of deposition, the film is often coherently strained to match the substrate. Therefore, the c axis and a axis lengths are under the corresponding compressive and tensile strains from the nucleation and get relax with the increase in ZnO layer thickness. Throughout this experiment, there are two anomalous lattice relaxation zones due to the ZnO layer thickness: (i) 10–20 nm, just after the h_c of ~ 6 nm (Ref. 11) and (ii) 400–1000 nm, after a complete relaxation. If the strained epitaxial film is sufficiently thick, then it is energetically favorable for a δ to be accommodated by a combination of an elastic strain. The thicker the film is, the more MDs are introduced and thus leading to a lower strain energy. Therefore, in the early stage of growth when the film is less than the h_c , ZnO film is coherently strained by plastic deformation. The strain energy due to δ proportionally increased to the film thickness and, therefore, the epitaxial layer is energetically unstable due to its higher energy compared to unstrained bulk. Above h_c , ZnO film becomes partially coherently strained because the strain begins to relax due to the spontaneous creation of dislocations at the ZnO/SiC heterointerface. As the thickness of the ZnO layer increases, the density of dislocations increases and the strained layer is completely relaxed.

When the ZnO layer increases to the film thickness of 100–150 nm, the strain is almost fully relaxed and the lattice constant of the films are converted to the bulk values. From the measurements of the lattice constant of a thin ZnO layer, the lattice constants of the a axis and c axis lengths were obtained as 3.252 and 5.202 Å, respectively. Compared to the stress-free ZnO lattice constants, $a=3.245$ Å and $c=5.210$ Å, the ZnO epilayer is highly strained. From this experiment, δ is 5.58%, which is almost the absolute lattice misfit for the bulk values. From this observation, h_c can be

FIG. 1. (Color online) (a) The c axis and a axis length relaxations due to ZnO layer thickness and (b) a schematic model of strain relaxation in ZnO/SiC heterostructure. The hatch zones are the anomalous lattice relaxation zones of ZnO layers deposited on the SiC substrates.

calculated by using the form suggested by Kim *et al.*¹² based on van der Merwe's equilibrium theorem,¹³ which is given by $a=a_0+h_c(a_s-a_0)/h$, where a is the in-plane lattice constant of the ZnO epilayer, a_0 is the unstrained lattice constant of ZnO, h is the ZnO layer thickness, h_c is the critical thickness of ZnO, and a_s is the substrate lattice constant. When $h=h_c$, a dislocation is introduced into the film layer to reduce the stress that originates from the δ between the ZnO layer and SiC substrate. Therefore, we expect that the lattice experience the maximum stress when $h=h_c$, and thus, the lattice constant is very close to that of the substrate. By using the van der Merwe's theorem, h_c is estimated to be

$(4.20\text{--}6.28) \pm 0.04$ nm. It has been demonstrated that the h_c of II–VI materials for the δ of $\sim 5\%$ is in the range of 2–6 nm.¹⁴

Considering the experimental observation of lattice relaxation as a function of ZnO layer thickness, it is found that the a axis length elongates to the maximum, while the c axis length gets compressed to the minimum. The complete lattice relaxation mechanism due to layer thickness of ZnO/SiC heterostructure has been sketched in Fig. 1(b). The c axis and a axis lengths of bulk ZnO and SiC substrates, respectively, are as follows: 5.21, 3.25, 15.12, and 3.08 Å. From these data, the δ in the ZnO/SiC heterostructure is $<5\%$, which is responsible for the appearance of MDs marked by arrows, while the ZnO layer thickness is <10 nm. In this stage, the lattice relaxes by plastic deformation and induces MDs. For the ZnO layer thickness of <100 nm, the lattice relaxation takes place both by plastic and elastic deformations by keeping in balance—thereby no anomalous zones. However, after the complete relaxation, only elastic deformation is dominant and again induces anomalous lattice relaxation due to imbalance problems. Finally, a complete ZnO atomic unit forms after the layer thickness of ~ 1 μm .

To understand the state of strain relaxation in ZnO/SiC heteroepitaxy, the in-plane (ϵ_{xx}) and out-of-plane (ϵ_{zz}) strain kinetics have been plotted due to the a axis and c axis lengths. Figure 2(a) shows the lattice parameters a and c versus strain along the parallel and perpendicular planes of ZnO layers grown on the SiC substrates. These structural parameters were evaluated by using the four-axis XRD measurements and details of these results have been reported elsewhere.¹⁵ The plotted results exhibit the linear and non-linear relationships between the lattice parameters and the epitaxial layers elastically and plastically deformed by stress in the epilayers. The ϵ_{xx} is rather linear throughout the relaxation process with respect to the a axis, while the ϵ_{zz} is linearly initially increased but shows an anomalous character when comes close to the bulk values of the c axis lengths. Here, the strain parameters range from -0.3 to $+0.1$ and -0.22 to $+0.3$ for the c axis and a axis lengths, respectively. This anomaly in the lattice parameter relaxation is the result of biaxial strain kinetics and/or associated with complex materials engineering and chemistry. In principle, ZnO belongs to the space group $P6_3mc=C_{6v}^4$, and the material grows along the c axis and relaxes faster perpendicular to the substrate. Since the δ and σ in the ZnO/SiC heterostructure are even larger, the strain relaxation would imply the elimination of dislocations for a few nanometer thicknesses and terminated by big crystallite sizes. Considering these properties, the isotropic spin-orbit coupling, band structure, and electronic properties of ZnO/SiC heterostructures have considerably been modified.

Reciprocal space mapping in the inset of Fig. 2(a) shows that the c axis of ZnO elongates parallel to the substrate surface. For ZnO/SiC heterostructure, although the mosaic spread of planes perpendicular to the surface normal was quite small, the lateral coherence length was quite small, <54 nm, indicating the presence of extended defects with a small spacing. With the increase in the ZnO layer thickness, the $\Delta 2\theta$ and $\Delta\omega$ suddenly decrease and become minima for

FIG. 2. (Color online) (a) The ϵ_{xx} and ϵ_{zz} as a function of ZnO lattice constant and the inset shows the two-dimensional x-ray reciprocal area map of ZnO(0002) reflection grown on the SiC substrate. (b) The $\Delta 2\theta$ and $\Delta\omega$ of XRD of ZnO layers and the inset shows the distribution of an arbitrary reciprocal lattice vector in terms of the tilt and twist distributions. The hatch is the complex zones of the critical lattice relaxation due to the c axis and a axis lengths in ZnO layers.

the complete relaxation of strain, as plotted in Fig. 2(b). It is noteworthy that the strain energy in the initial growth stage is very large as -2.4% . This indicates that ZnO nucleated under the high strain energy on the SiC, mostly due to the layer/substrate misfits. In contrast, the ZnO(0002) rocking curve for the ZnO shows a full width at half maximum (FWHM) of $0.2^\circ\text{--}0.8^\circ$ plotted in Fig. 2(b). Apparent longer lateral coherence length indicates a significant increase in the spacing between the extended defects. This implies that the initial nucleation of dislocations is overwhelmingly edge in nature. The x-ray rocking curve (XRC) of the as-grown film was observed to consistently broaden to 0.04° , presumably due to the dislocation interaction in the growing film which leads to an increase in the average extended interdefect spacing. However, the strain drastically decreases with the increase in the ZnO layer thickness and gradually relieves afterward. It has been demonstrated that the strain energy also critically depends on the layer thickness, and relieves $\sim 94\%$ at larger grain sizes for the ZnO layer thickness of <2 μm .^{10,15}

These asymmetric reciprocal space map and XRD spectra broadening can be defined by considering the model frame of a reference. To describe the distribution of any reciprocal lattice vector in the plane of crystalline reference frame axes, in particular, the surface normal (tilt distribution) and an in-plane (twist distribution) reciprocal lattice vector, as shown in the inset of Fig. 2(b), there are two characteristics of the distribution of typical rocking curves (the rocking curve measures a vertical slice of the two-dimensional distribution function) their FWHM, W , and their Lorentzian fraction f . This is accomplished by making use of rotation matrices as given by the theory of rigid-body rotations by $W_0^{\text{tilt}} = \cos^{-1}[\cos^2(\Gamma_0)\cos(W_y) + \sin^2(\Gamma_0)]$ and $W_0^{\text{twist}} = \cos^{-1}[\cos^2(\Gamma_0)\cos(W_z) + \cos^2(\Gamma_0)]$, where Γ_0 is the angle of inclination (i.e., the angle between the reciprocal lattice vector of interest and the surface normal) and W_y and W_z are the FWHM of the surface normal reciprocal lattice vector (tilt angle) and the in-plane reciprocal lattice vector (twist angle), respectively. Assuming that the tilt and the twist distributions are independent, the angular component of the two distributions can be convoluted to give the resultant misorientation distribution that can be expressed in terms of the W of the initial pseudo-Voigt functions by

$$W_{\text{resultant}} = [(W_1)^n + (W_2)^n]^{1/n}, \quad (1)$$

where W_1 and W_2 are the FWHM of the initial distributions and $n=1-2$ depending on the value of $f[n=1+(1-f)^2]$. By using this theorem, in-plane mosaic is determined to be 0.78° for the ZnO/SiC heterostructure, while 0.94° for ZnO film grown on Al_2O_3 and 0.93° for the AlN film on Al_2O_3 .¹⁶

The biaxial strain relaxation kinetics has also been theoretically assessed by considering the MDs at the heterointerfaces and thereafter into the thin films. The energy cost E_{MD} required for creating a unit length MD at the heterointerface is given by

$$E_{\text{MD}} = \frac{\ln(1 + h/b) \times \mu b^2(1 - \nu/4)}{4\pi(1 - \nu)}, \quad (2)$$

where μ is the shear modulus at the interface between the film and the substrate [$\mu \approx (C_{11} - C_{12})/2$] for the slip system $\langle 011 \rangle \{111\}$, ν is the Poisson ratio, and b is the Burgers vector. For comparison, GaN/SiC heterostructure has been fitted theoretically and plotted in Fig. 3(a). When the ZnO layer thickness is ~ 200 nm, the E_{MD} is 8 meV but 11 meV while it approaches 1000 nm. This means that the possibility of the formation of dislocations in ZnO/SiC is relatively higher than that of the GaN/SiC heterostructures. The fitted parameter was found to be $b=2.253$; absolutely, the a axis length of ZnO. It is found that the MDs are easier to form in the ZnO/SiC heterostructure than in the ZnO/ Al_2O_3 and GaN/SiC materials systems. There is a common tendency to easily form the MDs in II-VI than in the III-V materials.¹⁴ On the other hand, there are systems such as GaN/AlN,¹⁷ with lower δ (2.4%), which are known for exhibiting a clear SK transition with the formation of coherent islands.

TEM diffracts the MDs and threading dislocations in ZnO/SiC heterostructures plotted in Fig. 3(b). The cross-sectional image clearly shows that ZnO layer has three steps: initially with high density of dislocation defects for the ZnO

FIG. 3. (Color online) (a) Theoretical calculation of MD formation energy E_{MD} as a function of ZnO layer thickness and (b) TEM cross section of the ZnO/SiC heterostructure where the MD and threading dislocations are evident. There are three steps in ZnO layers marked by straight lines for the thicknesses of 100–200 nm and <1000 nm.

layer thickness of 100–150 nm, later on relatively low density of defects, and finally, columnar growth modes with fresh edges. This observation nicely matched with the observation in lattice and strain relaxation mechanics since there were also three steps in covering the relaxation with the increase in the ZnO layer thickness. The clear dark line almost along the c axis in the ZnO layer represents a threading dislocation. The threading dislocation does not significantly affect the growth of ZnO on it. A closer look at the image reveals that near the ZnO surface, smaller domains of a few tens of nanometers in width exist. Such smaller domain structures extend for around 100 nm along the growth direction, after which the larger domain structures at the size order of 100 nm are formed by connecting smaller domains. The columnar growth mode has been subjected to the imbalance of interface chemistry in the II–VI/IV materials system.¹⁸

PL measurements showed the dominant 3.3762, 3.3638, and 3.330 eV peaks originated from free-exciton (FX), neutral-donor bound exciton (D^0X), and neutral acceptor-bound exciton (A^0X) bands, respectively.⁹ It is added that the deep-level emission was completely absent even at RT in the ZnO/6H-SiC heterostructures. Figure 4 shows the band edge emission peaks as a function of the cell volume ($\sqrt{3}/2a^2c$)

FIG. 4. (Color online) Photon energy as a function of the cell volume of ZnO layers deposited on the SiC substrates. The PL measurements were performed by using the He–Cd laser for the excitation wavelength of 325 nm at liquid He temperatures.

for the ZnO epitaxial layers. In principle, the photon energy does not change due to the cell volume especially for the wurtzite structure. If we carefully observe, the band edge actually does not change due to the change in the cell volume since the (A^0X) band is constant due to the cell volume. However, for the band edge emission, it is almost the same except the zone where the anomalous lattice relaxation was found. This simply reflects that it might not be related to the band edge shifting with respect to the cell volume but be in a trap. It is noted that the FX_A band is observed only for the layer thickness of $6 \leq t \leq 1000$ nm. This FX_A band dominance can be initially attributed to the defect-free ZnO layers since h_c has been experimentally assigned to be 4–6 nm. In addition, there were no FX_A bands for the layer thickness of >10 – <80 nm due to defects possibly contributed by δ , as has been clarified in TEM and secondary ion-mass spectroscopy.¹¹ However, with the increase in the ZnO layer thickness, the FX_A , D^0X , and A^0X bands were blueshifted by the energies of 2.5, 4.8, and 5.8 meV, respectively. This blueshifting is consistent with the systematic biaxial strain relaxation as a function of ZnO layer thickness.

It has been demonstrated that δ starts to relax for the layer thickness of 4–6 nm and then becomes constant in strain for a fluctuation of 0.1%. However, it relaxes faster for the layer thickness of <10 nm and suddenly gets down in the layer thickness of 80–100 nm. If δ and h of the epilayers are small, the misfits between the two semiconductors are accommodated by the trigonal strain in the epilayer. For a given δ , if the thickness h of the epilayer exceeds the h_c , the strain is partly accommodated by the MDs. The strain was relieved as the film thickness increased, while columnar

seeds started to nucleate on parts of the film. The strain in ZnO film was completely relaxed when the film thickness was thicker than 150 nm. It has been attributed that the improvement in the crystal quality is related both to dislocation density and to the reduction in the degree of misorientation in the ZnO/SiC heterostructures.

IV. CONCLUSIONS

The anomalous lattice/strain relaxation mechanics in ZnO/SiC heterostructure has been demonstrated. The anomalous lattice relaxation is the result of complex interface chemistry, local mosaics resulting in tilting and twisting of the c axis and a axis lengths that finally reflected by the asymmetric plastic and elastic lattice deformation kinetics and terminated by the formation of MDs and threading dislocations. The overall observation has been represented by a schematic model.

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