Depth-dependent mosaic tilt and twist in GaN epilayer: An approximate evaluation*

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An approach based on depth-sensitive skew-angle x-ray diffraction (SAXRD) is presented for approximately evaluating the depth-dependent mosaic tilt and twist in wurtzite *c*-plane GaN epilayers. It is found that (103) plane and (101) plane, among the lattice planes not perpendicular to the sample surface, are the best choices to measure the depth profiles of tilt and twist for a GaN epilayer with a thickness of less than 2 μm according to the diffraction geometry of SAXRD. As an illustration, the depth-sensitive (103)/(101) ω-scans of a 1.4-μm GaN film grown by metal–organic chemical vapor deposition on sapphire substrate are measured and analyzed to show the feasibility of this approach.

Keywords: mosaic structure, tilt and twist, skew angle x-ray diffraction, GaN

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1. Introduction

The heteroepitaxial wurtzite *c*-plane GaN films generally feature a mosaic structure due to the lattice mismatch and the thermal expansion mismatch between the film and the substrate. The threading dislocations along the [0001] direction with a density of about 1×10^8 cm^{-2} $\sim$ 10^{10} cm^{-2}, which include screw-type dislocations with a Burgers vector $b = \langle 0001 \rangle$ accommodating the lattice tilt, edge-type dislocations with $b = (1/3)\langle 11\bar{2}0 \rangle$ accommodating the lattice twist, and mixed type dislocations with $b = (1/3)\langle 11\bar{2}3 \rangle$ accommodating both tilt and twist, form the boundaries among mosaic subgrains.[1] In conventional X-ray diffraction technique, the lattice tilt of *c*-plane GaN wafers can be roughly measured by the width of symmetric ω-scans of $(000l)$ planes, or by the asymmetric ω-widths of some lattice planes inclined to the sample surface (the $(10\bar{1}4)$ plane is typical), and the lattice twist is usually evaluated indirectly by the width of skew-symmetric $(hkil)$ ω-scans (usually the $(10\bar{1}2)$ plane is measured).[2–4] The measured peak widths reflect the depth-weighted average information of mosaicity over the whole epitaxial film since the penetration depth d_p of X-rays (fixed by the X-ray absorption coefficient of the material, the Bragg angle of the lattice plane under measurement, the diffraction geometry, etc.) ranges from several micrometers to tens of micrometers and is usually larger than the thickness of the film. However, the depth-dependent tilt/twist with controllable depth selectivity from the sample surface down to the substrate cannot be achieved by the aforementioned diffraction geometry. This is unfortunate, because such information is important in research on the growth dynamics[5] and the defect formation/control of nitride materials[6–8] and the performance evaluation and reliability analysis of GaN-based optical and electronic devices.[9–12]

The skew angle X-ray diffraction (SAXRD) method[13–15] was developed to characterize the depth-dependent crystal quality of a single-crystal epitaxial film. One can use this method to change d_p gradually from nearly 0 μm (sample surface) by varying the incidence angle of the X-rays, and meanwhile keep measuring the width of a series of ω-scans of the same $(hkil)$ plane to directly evaluate the crystal quality of the material in the range of d_p. The measured widths can be analyzed by an algorithm[16] to extract the real widths of the $(hkil)$ reflection in distinct depths of the film. Specifically, the width results are a mixture of tilt and twist. However, SAXRD can measure many lattice planes, and it is likely to evaluate tilt and twist approximately via distinct lattice planes. According to the principles and the diffraction geometry of SAXRD, we investigated the lattice planes that might be optimal for measuring the depth profiles of tilt and twist, respectively, and analyzed a *c*-plane GaN film grown by metal–organic chemical vapor deposition (MOCVD) on sapphire substrate with these lattice planes.

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2. Principles to choose the lattice planes for depth-sensitive tilt and twist measurement in SAXRD

For mosaic hexagonal nitride films, only the lattice planes that can be accessed by skew symmetric diffraction can be evaluated by SAXRD. As shown in Fig. 1, the diffraction geometry of the lattice plane in SAXRD measurement is between the skew symmetry (with $\phi = 0°$ assumed where ϕ is the rotation angle of the sample surface around its normal n_0) and the asymmetry (with $\phi = 90°$). In order to change d_p, the diffraction plane is rotated around the normal (n) of the lattice plane (from the AOB plane to the $A'OB'$ plane). In this process, to keep the direction of the normal n unchanged, one must simultaneously adjust the ω, χ, and ϕ angles of the X-ray diffractometer, and the way to calculate the increments for these angles can be found in Ref. [14].

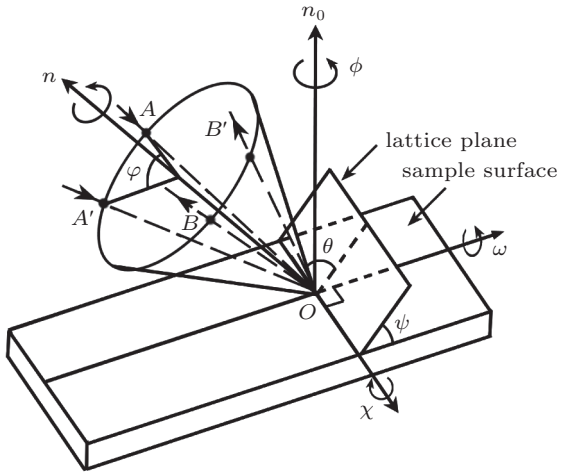


Fig. 1. Diffraction geometry of the SAXRD measurement. The angle φ (ϕ) is the rotation angle of the lattice plane (sample surface) around its normal n (n_0). When the penetration depth of X-rays increases, ϕ and φ decrease while ω and χ increase.

The penetration depth d_p of various lattice planes of wurtzite GaN can be calculated as the function of angle ϕ as shown in Fig. 2. The linear absorption coefficient $u = 0.029$ μm^{-1} is adopted.[16] It can be seen from Fig. 2 that ϕ decreases with increasing d_p, or the diffraction geometry changes from asymmetric to skew symmetric. A starting point of penetration depth from $d_p = 0$ requires the lattice plane with $\psi > \theta_B$, where ψ is the angle between the lattice plane and the sample surface and θ_B is the Bragg angle of the lattice plane. It is noted that the minimum value of d_p could approach 0 μm in theory, but in practice it is decided by the precision of the X-ray diffractometer and could be tens of nanometers.[16]

The selection of a lattice plane from those satisfying the above two requirements to be used in measuring the depth profile of tilt/twist focuses on the planes with diffraction geometry as close to asymmetric/skew symmetric. The plane used for tilt requires both that the angle ϕ approaches 90° at $d_p = 0$ and that the angle ψ is as close to θ_B as possible. The plane for twist requires that ϕ approaches 0° at $d_p = 0$ and that the angle ψ is as large as possible. A requirement suitable for both cases is that d_p changes sensitively with the angle ϕ.

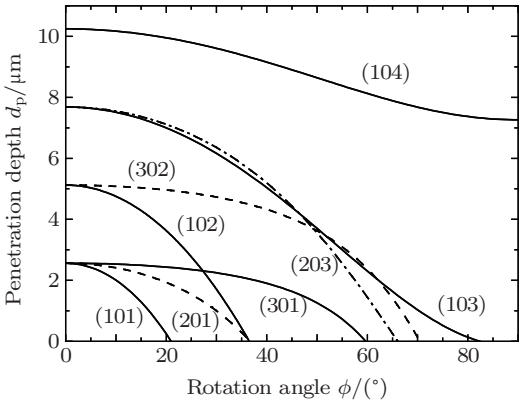


Fig. 2. Penetration depth d_p of various lattice planes of wurtzite GaN as functions of the angle ϕ in SAXRD.

Based on a comprehensive consideration of the above requirements, one can see from Fig. 2 that the optimal lattice planes to measure depth-dependent tilt and twist are the (103) plane and the (101) plane, respectively, for a GaN epilayer less than 2-μm thick. When the penetration depth increases from 0 to 2 μm, the angle ϕ of the (103) plane changes from about 82° to 62°, and the diffraction geometry remains close to asymmetric grazing incidence diffraction. In the same range of d_p, the angle ϕ of the (101) plane changes from 21° to 10°, and the diffraction geometry stays close to skew-symmetric diffraction. Additionally, quite high diffraction intensity can be obtained from these two planes, benefiting the SAXRD measurement at shallow depths.

Issues about errors inherent in the evaluation method cannot be avoided. In order to reduce the error of measurements, it is better to reduce the target depth step by step, and a further fine tuning of the X-ray alignment is usually necessary for each depth on the basis of the calculated ω, χ, and ϕ angles. Especially, poor alignment or instrument broadening is likely to cause great difficulty in the measurement of the (101) reflections at shallow depths. Since the SAXRD diffraction gets closer to skew symmetry for deeper X-ray penetration, in principle the deviation of the width of (101) reflections from the pure twist would decrease, while the error in using (103) ω-width to characterize the tilt would increase. In addition, the practical absorption coefficient of X-rays may differ in different samples, and a larger coefficient reduces the depth that can be detected.

3. Sample analysis

The *c*-plane nitride sample under investigation was grown in a self-built low pressure MOCVD system on a 2-inch (1 inch = 25.4 mm) sapphire substrate. The sample is grown under a pressure of 40 Torr (1 Torr = 1.33322×10^2 Pa), and except for the 10-nm low temperature AlN (LT-AlN) nucleation layer grown at 550 °C, all the other layers including 500-nm GaN, 20-nm high temperature AlN interlayer (HT-AlN IL), and subsequently 900-nm GaN followed by a 20-nm $Al_{0.3}Ga_{0.7}N$ layer, are grown at 1000 °C. Trimethylgallium (TMG), trimethylaluminum (TMA), and ammonia (NH_3) are used as Ga, Al, and N sources, respectively, and purified hydrogen is used as the carrier gas.[4] The XRD measurement is performed using Bruker D8-discover system equipped with a Ge (220) monochromator, delivering a pure Cu $K\alpha$ line of wavelength $\lambda = 0.15406$ nm.

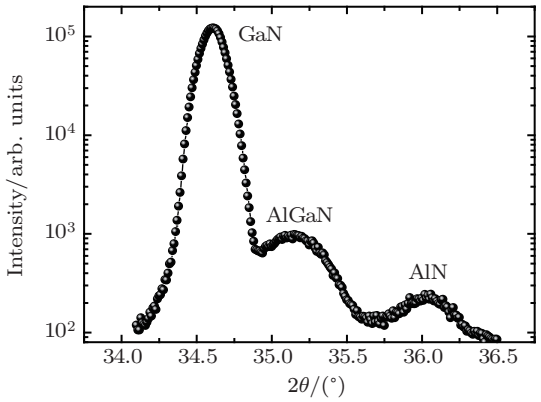


Fig. 3. Measured 2θ–ω curve of the sample.

Figure 3 depicts the 2θ–ω curve of the sample measured in double-crystal diffraction condition. The peaks for GaN, AlGaN, and AlN are all distinct, implying good crystal quality of these materials. To obtain the tilt β_t and the twist α_t of the sample, the rocking curves of the symmetric (002) plane and several skew-symmetric planes are measured before SAXRD measurement, and the data of width w are fitted as a function of the angle ψ (Fig. 4) and extrapolated by using the approach proposed in Ref. [17], resulting in $\beta_t = 297$arcsec and $\alpha_t = 864$ arcsec. The tilt is smaller in magnitude than the twist, which is a typical feature of GaN epilayers with a good crystal quality. The penetration depths of these lattice planes in the (skew) symmetric diffraction are larger than the thickness of the nitride film (about 1.4 μm), and thus β_t and α_t present the mosaic effect of the whole film.

Considering the thickness and the layered structure of the sample, we took ω-scans of the (103) plane and the (101) plane of the nitride epilayers from the top surface down to the depth of 1350 nm with a constant depth step of 150 nm by using SAXRD and the real width of each 150-nm slice is extracted with the lower triangular coefficient matrix of normalized X-ray intensities, as proposed in Ref. [16]. The measured width and the extracted one are given in Fig. 5, and also shown for comparison are the extrapolated values of tilt and twist and the ω-width of the (101) plane in Fig. 4.

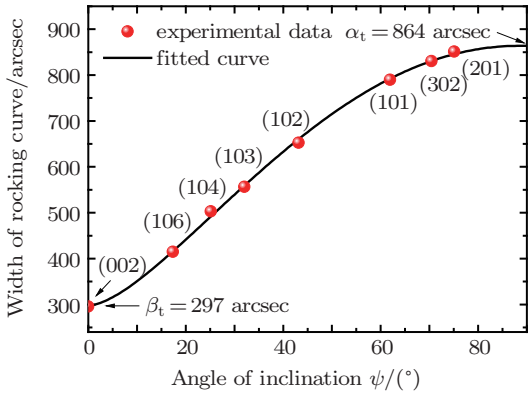


Fig. 4. (color online) Measured widths of (skew) symmetric X-ray rocking curves of some lattice planes versus angle ψ between the plane and the wafer surface, with a fitted curve.

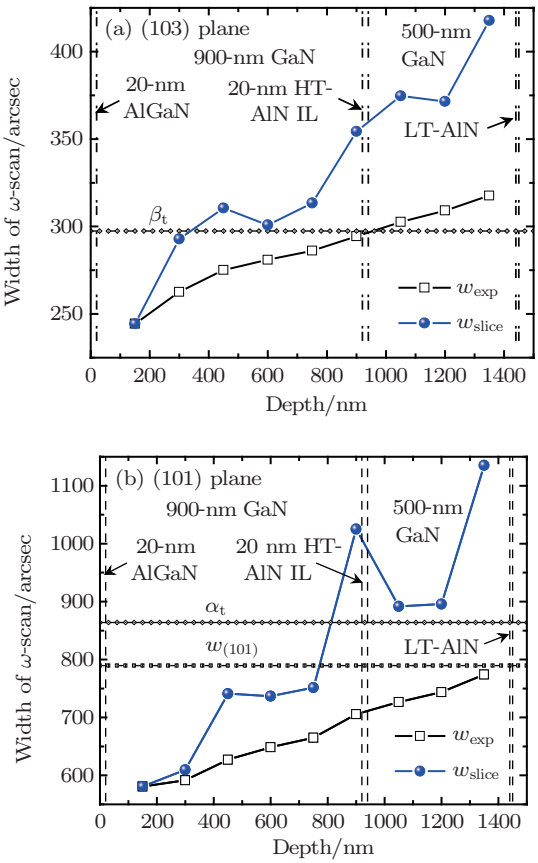


Fig. 5. (color online) Peak widths of SAXRD ω-scans of (a) (103) plane and (b) (101) plane as functions of penetration depth of X-ray. Both the measured width w_{exp} and the extracted one w_{slice} are plotted, and the tilt/twist and ω-width of the (101) plane in Fig. 4 are presented as horizontal lines labeled β_t/α_t and $w_{(101)}$. The location of each layer in the nitride film is also shown.

The fact that the measured width decreases with decreasing depth implies that the crystal quality becomes better and better toward the surface, and it is a reasonable trend according to the growth process of nitride hetero-epitaxy. At the maximum penetration depth of $d_p = 1350$ nm, the width of the (101) plane (774 arcsec) measured by SAXRD is slightly smaller than the skew-symmetric one of the same plane (790 arcsec) and smaller than the twist ($\alpha_t = 864$ arcsec); and that of the (103) plane (318 arcsec) is slightly larger than the tilt ($\beta_t = 297$ arcsec). So when the penetration depth approaches the total thickness of the considered material (which should be less than 2 μm, as pointed out in Section 2) the error in evaluating the mosaic tilt/twist by the SAXRD peak width of the (103)/(101) plane is quite small. What is more, the error in using the (103) plane to evaluate the depth-dependent tilt increases at greater depths. As a result, the above comparison shows that the (103) plane can be used in the SAXRD method to evaluate the tilt effectively, although it cannot be accessed in conventional asymmetric diffraction because its ψ (32.04°) is larger than θ_B (31.72°).

Compared with the measured width w_{exp}, the extracted width w_{slice} for each slice reflects more sensitively the density variation of different threading dislocations along the material's growth direction. The steep decline of w_{slice} versus d_p curves for both (103) and (101) planes in the range of 1200 nm $\sim$ 1350 nm shows a strong annihilation of dislocations appearing at about 100 nm $\sim$ 150 nm above the LT-AlN nucleation layer,[18,19] a region where the growth mode of GaN begins to change from vertical island growth to island coalescence. While in the subsequent 300 nm (750 nm $\sim$1050 nm), a distinct difference in the evolution of tilt and twist emerges. The twist increases clearly across the HT-AlN IL and then reduces quite greatly, but the tilt keeps reducing continuously and seems unaffected by the HT-AlN IL. This difference should be closely correlated to the cracking of HT-AlN IL.[20] The interlayer undergoes a tensile strain on the GaN template and its thickness of 20 nm far exceeds the critical thickness of AlN to crack (about 7 nm observed in Ref. [21]). The cracks of the HT-AlN layer, usually along $[11\bar{2}0]$ axes, intersect each other and form net-like boundaries, through which strain is partially relieved, while the surrounding AlN areas (with quite a good crystal quality) keep coherently strained with the GaN below.[22] Consequently, in GaN grown on the HT-AlN IL, almost no additional tilt comes into being due to the high quality of the HT-AlN layer. But more twist-related edge dislocations appear (increased twist at $d_p = 900$ nm) originating from the densely distributed cracks or the turned misfit dislocations at the AlN/GaN heterointerface,[23,24] although some are blocked by AlN IL, and the interplay among these chaotic edge dislocations quickly leads to another strong dislocation annihilation (decreased twist at $d_p = 750$ nm). Thereafter, a mild decline of tilt and twist continues toward the sample surface, which is a natural result in view of the layered structure of the sample. Additionally, it is reasonable that the tilt/twist lies in the midst of the spans of w_{slice} data of the (103)/(101) plane for the GaN layer with an HT-AlN IL inserted.

4. Conclusion

A fast, low-cost, non-destructive approximate evaluation method for depth-sensitive mosaic tilt and twist in wurtzite GaN hetero-epilayer is given based on skew-angle X-ray diffraction (SAXRD). It is found that the (103) /(101) plane is optimal to investigate the depth-dependent tilt/twist for GaN films less than two micrometers thick. The measurement and analysis of SAXRD ω-widths of these two planes are carried out on a GaN sample with a high temperature AlN interlayer (HT-AlN IL), showing the effectiveness of the method. The principles for choosing the lattice planes for depth-sensitive SAXRD measurement of tilt and twist are also applicable to the other mosaic hexagonal semiconductor films such as AlN and ZnO.

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