Indentation-induced dislocations and cracks in (0001) freestanding and epitaxial GaN

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Abstract. The (0001) surface of freestanding GaN and an (0001) oriented epitaxial GaN layer of 3 µm thickness on sapphire have been deformed at room temperature using a Vickers indenter. The samples were indented with two different orientations of the indenter with loads in the range from 0.02 to 4.90 N and 0.10 to 4.90 N, respectively. Dislocations and cracks at the indentations were observed by means of scanning electron microscopy, cathodoluminescence, optical microscopy and transmission electron microscopy. Dislocations occur at all indentations for the loads used in the investigations. In both materials, the dislocation arrangement corresponds to the symmetry of the indented surface and the orientation of the indenter has only a marginal influence. Higher loads lead to radial cracks at the corners of the indentations and lateral cracks beneath the surface. The crack system is predominantly determined by the symmetry and orientation of the indenter. The dislocation arrangement and the crack system in freestanding GaN and an epitaxial layer of GaN on sapphire are compared.

1. Introduction

GaN is a III-V compound semiconductor crystallizing predominantly in the wurtzite structure \((a = 0.319 \text{ nm and } c = 0.518 \text{ nm})\) [1]). It is an important material for optoelectronic and high-frequency devices on account of its direct band gap of 3.39 eV [2]. There are several studies of the mechanical properties of GaN single crystals, but there are just a few results of crack formation and propagation [3-6]. One model for the formation of crack seeds is the interaction of dislocations on different glide planes and the pile-up of dislocations at Lomer-Cottrell barriers [7]. If a crack seed is formed, subsequent crack propagation can take place and lead to a macroscopic crack. In addition to a deeper understanding of fundamental processes, a better knowledge of the mechanical behavior would be...
beneficial to optimize processing steps like sawing and grinding, as has been demonstrated for (001) GaAs [8].

The deformation of monocrystalline solids under increasing indenter load can be divided into three stages. In the elastic stage, the deformation is complete reversible. The initiation of the elastic-plastic stage is characterized by the pop-in event. It is associated with the onset of plasticity and the generation of dislocations [9]. In the elastic-plastic stage, the material is deformed by the generation and propagation of dislocations, but the deformation is partly elastic. The third stage is characterized by the formation and propagation of cracks. This can lead to chipping of the material and finally to a total destruction of the sample.

A detailed analysis of dislocations in the wurtzite structure has been given by Osipiyan et al. [10]. At indentations in (0001) GaN, dislocations are arranged in a star-shaped rosette with dislocation segments oriented in the $\pm <2\bar{1}0\bar{0}>$ directions [11]. Transmission electron microscopy investigations indicate $a$-type screw dislocations in the rosettes [12]. The rosette consists of dislocations perpendicular and inclined to the surface [13]. Slip lines occur only in the area within the indentation [14]. GaN has $\{1\bar{1}00\}$ cleavage planes [15]. Drory et al. [3] indented a GaN single crystal using a Vickers indenter. In that work, radial cracks occurred at the corners of the indentations. Most of the investigations deal with GaN epitaxial layers instead of freestanding GaN. The dislocation arrangement or the crack formation has only been investigated separately. In the present study, both dislocations and cracks are investigated in freestanding GaN and in GaN on sapphire. Freestanding GaN and GaN on sapphire are compared. Between dislocations and cracks, geometrical correlations could be found.

In order to investigate the crack formation, the relative crack frequency $n(F)$ is defined as [16]:

$$n(F) = N(F)/N_0,$$

where $N_0$ is the total number of corners of the indentation and $N$ the number of corners exhibiting a radial crack at a certain load $F$. The crack resistance $F_c$ is defined as the load $F$ where the relative crack frequency equals 0.5 [16]:

$$n(F_c) = 0.5.$$

2. Experimental details
In this study, three types of (0001) oriented, not intentionally doped GaN samples have been investigated: as-grown GaN single crystals, a GaN wafer and an epitaxial layer of GaN on sapphire (table 1). As-grown GaN crystals of 0.3 and 1.2 mm thickness have been produced using hydride vapor phase epitaxy on a sapphire substrate and exfoliation. The crystal directions were distinguished by X-ray Laue patterns. The samples were indented at room temperature (RT) using a Vickers indenter. A Hanemann MHP 100 micro hardness tester was used for loads from 0.02 to 1.00 N. In the range from 0.10 to 4.90 N, indentation experiments were performed with a Zwick 3202 hardness tester. For each material, indentations were performed with two different orientations of the indenter (0° series and 45° series). This was made to separate the influence of the indenter geometry from the symmetry of the crystal.

Table 1. Summary of the investigated (0001) oriented GaN samples.

| Material          | Density of in-grown dislocations | Thickness | Investigation         |
|-------------------|----------------------------------|-----------|-----------------------|
| GaN wafer         | $10^7 \text{ cm}^{-2}$           | 0.3 mm    | TEM preparation       |
|                   |                                  | 1.2 mm    | Relative crack frequency |
|                   | $1.7 \times 10^7 \text{ cm}^{-2}$ | 0.17 mm   | Cross section preparation |
| GaN on sapphire   | $6 \times 10^6 \text{ cm}^{-2}$  | 3 $\mu$m  | Relative crack frequency |
In the 0° series, one indentation diagonal was oriented in [2\(\overline{1}\)0] direction and the other diagonal pointed into the [01\(\overline{1}\)0] direction (figure 1a). In the 45° series, the sample was rotated in an angle of 45 degrees with respect to the indenter (figure 2b). For each load and orientation, 10 indentations were evaluated.

The indentations and cracks were examined by scanning electron microscopy using a Philips ESEM XL 30 FEG. Bulk GaN was investigated in secondary electron contrast (SE) at an acceleration voltage of 5 kV. GaN on sapphire was examined in the gas mode of the environmental scanning electron microscope (ESEM) at 20 kV in order to reduce the electric charging. The dislocations were observed by cathodoluminescence imaging (CL) in a JSM 6400 scanning electron microscope at an acceleration voltage of 8 or 10 kV in panchromatic mode. Defects beneath the surface in GaN were observed by optical microscopy (OM) in reflection or transmission using a Leica DMR microscope. Selected indentations were inspected by atomic force microscopy using a Nano-R of Pacific Nanotechnology. For TEM investigations in diffraction contrast, a JEM 4000 FX transmission electron microscope was used at an acceleration voltage of 400 kV. In order to fabricate electron-transparent samples, the indented crystal was drilled out with an ultrasonic driller and cleaned in acetone and ethanol. The back side of the sample was ground and dimpled mechanically. Finally, the sample was thinned by 3 keV argon ions to a final thickness of 0.5 to 1 \(\mu\)m in the region of the indentations using a Gatan precision ion polishing system model 691.

3. Freestanding (0001) GaN

Dislocations occur in the surrounding of all indentations for the loads used in the present investigations. In-grown dislocation can be clearly distinguished from indentation-induced dislocations. In-grown dislocations form an irregular network with no preferential orientation (figure 1). Indentation-induced dislocations are arranged in a rosette with six dislocation arms running in the \(\pm <2\overline{1}0>\) directions. Each arm is made up of two branches. Neighboring dislocation arms cut each other. As a consequence, the centre of the rosette appears star-shaped. In the arms, the dislocations are arranged in bands. In TEM micrographs, individual dislocations are visible in the bands. Long bands of straight dislocations indicate surface parallel dislocations. At a diffraction vector \(g\) of 2\(\overline{1}0\), six dislocation arms are visible. At \(g = 10\overline{1}0\), the contrast of dislocations in the \(\pm [2\overline{1}0]\) directions is at minimum (arrows in figure 1). This indicates \(a\)-type screw dislocations in agreement with the results of Albrecht et al. [12].

Figure 1. Dislocations at a Vickers indentation in N-polar (0001) GaN at a load of 0.25 N from the 0° series. TEM bright field images of the same position with a diffraction vector \(g\) of (a) 2\(\overline{1}0\) and (b) 10\(\overline{1}0\).
In GaN, dislocations act mainly as centers of non-radiative recombination. Hence, they appear dark in CL images (figure 2a, 2b). The speckled background in the CL images is caused by in-grown dislocations. The area of the indentation appears dark. No individual dislocations can be distinguished there. Near the indentation, line contrasts dominate in the dislocation arms, which indicate dislocation bands parallel to the surface. The line contrasts in the CL images correspond to dislocation bands found in TEM. In the outer regions, intersection points of dislocations with the surface dominate. The orientation of the indenter has got only a minor influence on the arrangement of the dislocations. However, the density of dislocation bands may vary, as can be seen in the CL images. The extension of the dislocation rosette is increased by electron beam irradiation (recombination-enhanced dislocation glide effect [17]) during the CL measurements. The size of the rosettes rises with increasing load, whereas the characteristic shape remains unchanged.

**Figure 2.** Dislocations and slip lines in the surrounding of Vickers indentations. Investigation of two indentations in Ga-polar (0001) GaN at a load of 0.98 N from the 0° and 45° series, respectively. (a) and (b): CL images at a temperature of 83 K. (c) and (d): SE images. (e) CL image at RT of an indentation from the 0° series at 4.90 N in (01\(1\)0) cross section with three types of dislocation bands (1 – 3).

**Figure 3.** Cracks at Vickers indentations in (0001) GaN. (a) and (b): OM images in reflection corresponding to figure 2a and 2b, (c) and (d): Scheme of dislocations (dotted), slip lines (dashed) and radial cracks (solid) at an indentation. (e) Cross section transmission OM image of a Vickers indentation from the 0° series at 4.90 N, R radial crack, L lateral crack.
Slip lines occur in the area within the indentations (figure 2c, 2d). There are three sets of slip lines lying in the ±<100> directions. In the 0° series, one set of slip lines is parallel to an indentation diagonal. The other sets are oriented at an angle of 60° to this diagonal and intersect at the other indentation diagonal. In the 45° series, one set of slip lines is parallel to the edges of the indentation in the [01T0] direction. The other sets of slip lines cut each other at a face of the indentation.

For cross section investigations, a set of indentations in the 0° orientation was aligned in [2T10] direction. Subsequently, the sample was cleaved though the indentations along the (01T0) plane. In the cross section CL images, three types of dislocation bands are visible (figure 2e). The area beneath the indentation appears dark. Near the indentation, no individual dislocations are visible, but in the deeper regions, dislocations inclined to the surface occur (1). Next to the indentation, dislocations perpendicular to the surface are visible (2). Beneath the indentation, dislocations parallel to the surface occur (3). The area of these dislocations reaches from the surface into deeper regions.

Higher loads lead to radial cracks at the corners of the indentations and lateral cracks beneath the surface (figure 3). The brightening at indentations in the OM images is caused by specular reflections at lateral cracks (figure 3a, 3b). The system of radial cracks is predominantly determined by the symmetry of the indenter. It changes with the rotation of the indenter. The crack patterns at Vickers indentations in (0001) GaN can be characterized as follows: Radial cracks emanate from the corners of the indentation. At higher loads, lateral cracks occur beneath the surface. At the highest loads used, radial cracks can emanate from the edges of the indentation in some cases. The characteristic features of the dislocation arrangement and the radial crack system are illustrated in figure 3c and 3d.

In the 0° series, the formation of radial cracks starts at a load of 0.05 N (figure 4a). In the 45° series, it begins at 0.1 N. The relative crack frequency $n$ (equation 1) rises with the load. The crack resistance $F_c$ for radial cracks (equation 2) obtained for measurements with the MHP 100 micro hardness tester amounts to $(0.28 \pm 0.12) \text{ N}$ in the 45° series and $(0.36 \pm 0.14) \text{ N}$ in the 0° series. The differences in the crack resistance between the 0° series and the 45° series are not significant. In addition to radial cracks, the formation of circular lateral cracks beneath the surface begins at 0.50 N in the 45° series and at 0.98 N in the 0° series (figure 4b). The crack resistance $F_c$ for lateral cracks amounts to 0.81 N in the 45° series and 1.39 N in the 0° series. For a load of 4.90 N, radial cracks emanate from every corner of the indentations. Additionally, at two indentations from the 0° series, a radial crack emanates from one edge of the indentation.

![Figure 4. Relative crack frequency at Vickers indentations in Ga-polar (0001) GaN (a) of radial cracks and (b) of lateral cracks with a MHP 100 micro hardness tester (empty symbols) and a Zwick 3202 hardness tester (filled symbols).](image-url)
4. Investigation of (0001) GaN on sapphire and comparison with freestanding (0001) GaN

Similar to freestanding GaN, the speckled background in the CL images is caused by in-grown dislocations (figure 5a, 5b). Indentation-induced dislocations are arranged in a rosette with six dislocation arms running in \( \pm 2\overline{1}00 \) directions. Each arm is made up of two branches. The orientation of the indenter has got only a minor influence on the dislocation arrangement. In contrast to freestanding GaN, the dislocation arms are broader. Line contrasts dominate and no intersection points of dislocations with the surface are visible. This indicates dislocations parallel to the surface. Slip lines in the different \( < \overline{1}00 \) directions occur in the area within the indentations (figure 5c, 5d), similar to freestanding GaN. Higher loads lead to lateral cracks beneath the surface (figure 5e, 5f). The frequency of lateral cracks is zero for loads up to 0.49 N (figure 5g). At 0.98 N, lateral cracks occur. For higher loads, the relative crack frequency equals 1. The crack resistance for lateral crack can be estimated to about 1.5 N. The differences between the 0° series and the 45° series are not significant. In contrast to freestanding GaN, radial cracks occur only marginally even at the highest load used.

Figure 5. Dislocations and cracks at Vickers indentations in (0001) GaN on sapphire at a load of 4.90 N from the 0° and 45° series, respectively. (a) and (b): CL images at RT, (c) and (d): ESEM images. (e) and (f) OM images in reflection. (g) Relative crack frequency of lateral cracks at indentation in (0001) GaN with a Zwick 3202 hardness tester.

5. Discussion

In the wurtzite structure, there are two types of smallest translation vectors: The \( a \)-type (\( a/3 < 2\overline{1}00 \) ) and the \( c \)-type (\( c < 0001 \) ). This leads to simple dislocations with a Burgers vector of \( al3 < 2\overline{1}00 \) and \( c < 0001 \). These Burgers vectors lead to five types of perfect dislocations: \( a \)-type edge dislocations, \( a \)-type screw dislocations, \( a \)-type 60° dislocations, \( c \)-type edge dislocations and \( c \)-type screw dislocations [10]. The most densely packed planes in the wurtzite structure are the \{0001\} basal planes and the \{\( \overline{1}00 \)\} and \{2\( \overline{1}00 \)\} prismatic planes. These planes are assumed to be the glide planes in the wurtzite structure [10].

For the comparison of the dislocation rosette (figure 1) with glide prism models, the most important results of the investigations are summarized as follows. The dislocation rosette at indentations in (0001) GaN consists of six arms running in the \( \pm 2\overline{1}00 \) directions. TEM investigations showed long bands of straight dislocations with an \( a \)-type Burgers vector in direction of the dislocation arms. In the outer regions of the rosette, CL images showed intersection points of dislocations with the surface. These factors indicate a dislocation arrangement for one rosette arm as sketched in figure 6. This model explains the experimental data for surface parallel dislocation arms.
The glide planes of these dislocations are perpendicular to the surface. As a consequence, the screw dislocation segments occur as line contrasts both in TEM and CL images. In the outer regions, intersection points of dislocations at the surface are visible in CL images. The slip lines in $<\overline{1}100>$ direction correspond to traces of the $\{2\overline{2}0\}$ planes.

Dislocations can interact with each other during their propagation. At the intersection of neighboring surface parallel dislocation arms, three different types of interactions of perfect edge and screw dislocations are conceivable (figure 7): 1. The intersection of two $a$-type screw dislocations. 2. The intersection of an $a$-type edge and an $a$-type screw dislocation. 3. The interaction of two $a$-type edge dislocations [18].

**Figure 6.** Glide prism configuration of dislocations in the rosette arms.

**Figure 7.** Possible interactions of dislocations on neighbouring dislocation arms.

1. The intersection of two $a$-type screw dislocations leads to jogs on both dislocations, which have a $60^\circ$ character (figure 7-1). The intersection of individual dislocations leads to elementary jogs, whereas the intersection of dislocation bands can lead to jogs with the height of several Burgers vectors. These jogs can easily move along the screw dislocations, but for a movement in the $\pm [0001]$ directions they have to climb. If the screw dislocations move in the $\pm [0001]$ directions, the $60^\circ$ segments will lag behind. Subsequently, elementary jogs can lead to point defects and higher jogs to prismatic loops. The edge dislocation segments are not affected by the cutting process, and they can propagate unhindered in the $\pm <2\overline{2}0>$ directions on $(1\overline{1}00)$ slip planes. 2. The intersection of an $a$-type edge and an $a$-type screw dislocation leads to jogs on both dislocations, which have a $60^\circ$ character (figure 7-2). The behavior of a jog on a screw dislocation was described above. The jog on the edge dislocation can move easily in the $\pm <2\overline{2}0>$ directions, but in the $\pm [0001]$ directions it can only climb. Hence, the edge dislocation can propagate unhindered in the $\pm <2\overline{2}0>$ direction of their slip plane. 3. Two $a$-type edge dislocations ($b_1 = a, b_2 = a$) of neighboring dislocation arms include an angle of $60^\circ$ (figure 7-3). The reaction of these dislocations would result in a dislocation with a Burgers vector $b_1 = a <\overline{1}100>$. According to the $E \propto b^2$ criterion ($E$ is the elastic energy of the dislocation), this dislocation is expected to be unstable, since $b_1^2 + b_2^2 = 2a^2$ but $b_3^2$ equals $3a^2$. This leads to repulsive forces between the edge dislocations and can result in a pile-up of dislocations.

The crack system is influenced by various factors. These are the cleavage planes of the crystal, the geometry of the indenter together with the strain field of the indentation and the interactions of dislocations. In the $0^\circ$ series of freestanding GaN, radial cracks propagate in $\pm [2\overline{2}0]$ and $\pm [01\overline{1}0]$ directions (figure 3a). Cracks in $\pm [2\overline{2}0]$ direction propagate in the $(01\overline{1}0)$ cleavage plane. In this direction, the cutting of surface parallel dislocations arms is low. Cracks in $\pm [01\overline{1}0]$ direction do not propagate in a cleavage plane. In this direction, two surface parallel dislocations arms intersect. This may lead to the various dislocation cuttings or the pile-up of...
dislocations, as described in figure 7. For the formation of crack seeds, different models have been proposed. Two prominent examples are the models of Stroh and of Cottrell. The model of Stroh predicts the formation of a crack seed at the pile-up of dislocations on one glide plane [19] whereas the model of Cottrell describes the formation of a crack seed due to the pile-up of dislocations on different glide planes [20]. Hence, cracks in \( \pm[01\overline{1}0] \) direction could be initiated by the interaction of \( a \)-type dislocations on neighboring dislocation arms. In the 45° series, radial cracks are crystallographically not strictly oriented (figure 3b). They emanate from the corners of the indentation in a region with a high interaction of neighboring dislocation arms (figure 2b). These cracks could be initiated by cutting processes or pile-up of \( a \)-type dislocations.

In GaN on sapphire, no intersection points of dislocations with the surface are visible (figure 5a, 5b). This indicates dislocations parallel and near to the surface. Another possibility is that the edge dislocation segments do not intersect the surface but they are bended to the interface GaN-sapphire. (0001) GaN on a (0001) sapphire substrate has a lattice mismatch of about 15 % [3]. The lattice relaxation is realized mainly by misfit dislocations with a Burgers vector in the (0001) plane [21]. However, there is only a partial relaxation, and a residual strain remains. Hence, the dislocations could be concentrated in the GaN layer. This would lead to an increased interaction between dislocations. The consequence is a modified dislocation rosette compared to freestanding GaN.

In (0001) GaN on a (0001) sapphire substrate, radial cracks occur only marginally at the highest loads used. In this orientation, the cleavage planes of GaN and sapphire do not coincide [15]. In addition, mechanical properties like hardness and fracture toughness are different [3]. Hence, the propagation of radial cracks due to cleavage may be suppressed. In GaN on sapphire, the formation of lateral cracks is the primary cracking mechanism. The formation of lateral cracks is independent from the orientation of the indenter.

In freestanding GaN, radial cracks are formed at lower loads than lateral cracks. The differences in the formation of radial cracks between the 0° series and the 45° series are not significant. The load of crack initiation and the crack resistance for lateral cracks are lower in the 45° series than in the 0° series. For lateral cracks in epitaxial GaN on sapphire, the differences between the 0° series and the 45° series are not significant. Their crack resistance is similar to the value of freestanding GaN from the 45° series. Radial cracks in GaN on sapphire occur only at the highest loads used.

6. Conclusions
In the study presented, the formation of dislocations and cracks at Vickers indentations in (0001) oriented freestanding GaN and 3 \( \mu \)m epitaxial (0001) GaN on sapphire have been investigated. Dislocation rosettes are found at all indentations. TEM investigations indicate dislocations with \( a \)-type Burgers vectors parallel to the direction of the dislocation arms. From TEM and CL images it can be concluded that the dislocations propagate as quarter loops. They emanate as \( a \)-type screw dislocations from the indentation and intersect the surface as \( a \)-type edge dislocations or 58° dislocations. Dislocations of neighboring rosette arms can interact with each other and hinder their further propagation. However, the dislocation arrangement beneath the indentation requires further investigation. For a more detailed analysis, the splitting of perfect dislocations into partial dislocations and their reactions should be considered.

Cracks occur at higher loads at the indentations. In freestanding GaN, radial cracks emanate from the corners of the indentations. The investigations indicate that the formation of crack seeds has to be clearly separated from crack propagation. Crack seeds are attributed to the interaction of dislocations such as intersection or pile-up of dislocations, whereas the crack propagation is associated with the strain field of the indentation and the mechanical and crystallographical properties of the material. In contrast to (TTT) GaAs, radial cracks in GaN are not aligned in cleavage planes in many cases. In future experiments, the formation as well as the length of radial cracks in different directions will be investigated with higher statistics in order to prove differences in the cracking behavior inside and outside a cleavage plane. The crack frequency in freestanding GaN with lower dislocation density will be investigated in more detail in order to determine the influence of in-grown dislocations on...
indentation-induced dislocations and the crack formation. In addition, this could allow a closer analysis of indentations-induced dislocations and their interactions.

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