Microstructure of PAMBE-grown InN layers on Si(1 1 1)

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Abstract

The microstructure of InN layers grown by plasma-assisted molecular beam epitaxy on Si(111) substrates and an AlN buffer layer was investigated. InN layers with a thickness of ~500 nm were deposited at substrate temperatures between 325 °C and 375 °C under otherwise identical conditions. The structural characterization was performed by scanning electron microscopy and different transmission electron microscopy techniques including selective-area electron diffraction, electron-energy loss spectroscopy and energy-dispersive X-ray spectroscopy. The microstructure of the InN layers changes considerably despite the comparably small interval of growth temperatures.

1. Introduction

InN is a promising material due to its extraordinary physical properties such as small direct band gap of 0.7 eV, low effective electron mass, high electron mobility and high saturation velocity [1–3]. Therefore it is well suited for applications in high-speed optoelectronic devices—provided that material with good structural quality can be produced. Furthermore the ternary InAlN and InGaN alloy systems allow band-gap tuning over a wide spectral range from ultraviolet (AlN) to infrared (InN), including the whole visible spectrum, enabling applications like high-efficiency multi-junction solar cells [7–11] or high-power blue lasers [12]. Reported that nitrogen-rich plasma-assisted molecular beam epitaxy (PAMBE) growth conditions lead to granular, polycrystalline InN films whereas a N/In-ratio near stoichiometric conditions results in two-dimensional (2D) layer growth. A low-temperature InN buffer layer yields a poor crystal quality whereas a high-temperature AlN (HT-AlN) buffer layer improves the structural quality of the InN epilayer. Grandal and Sánchez-García [13] studied the influence of the growth temperature on the InN growth by PAMBE. Growth above 500 °C near the dissociation temperature leads to In-droplet formation on the surface. Below 500 °C, the growth process could be controlled by the N/In-ratio. They also reported uncoalesced, polycrystalline morphologies for N-rich growth conditions whereas increasing the In/N-ratio leads to coalesced columnar growth. Inverse pyramidal structures resulted from the growth without a buffer layer, indicating an inefficient wetting process. The deposition of a low-temperature InN (LT-InN) layer or a HT-AlN-buffer layer produced coalesced epilayers with superior crystal quality. Ajagunn a et al. [14] managed to grow compact InN layers directly on Si(111) by RF-MBE under nearly stoichiometric conditions. However, the substrates, including silicon. Its advantages are a reduced lattice-parameter mismatch of only ~7.7% for InN growth and excellent crystal quality. Another strong motivation for InN on Si(111) growth is the possible combination of InN-based devices and electronic devices, which can be implemented in the substrate by the well-established Si-technology.

Despite these advantages only few studies regarding the growth of InN on Si(111) substrates were published up to now. Hsiao et al. [12] reported that nitrogen-rich plasma-assisted molecular beam epitaxy (PAMBE) growth conditions lead to granular, polycrystalline InN films whereas a N/In-ratio near stoichiometric conditions results in two-dimensional (2D) layer growth. A low-temperature InN buffer layer yields a poor crystal quality whereas a high-temperature AlN (HT-AlN) buffer layer improves the structural quality of the InN epilayer. Grandal and Sánchez-García [13] studied the influence of the growth temperature on the InN growth by PAMBE. Growth above 500 °C near the dissociation temperature leads to In-droplet formation on the surface. Below 500 °C, the growth process could be controlled by the N/In-ratio. They also reported uncoalesced, polycrystalline morphologies for N-rich growth conditions whereas increasing the In/N-ratio leads to coalesced columnar growth. Inverse pyramidal structures resulted from the growth without a buffer layer, indicating an inefficient wetting process. The deposition of a low-temperature InN (LT-InN) layer or a HT-AlN-buffer layer produced coalesced epilayers with superior crystal quality. Ajagunn a et al. [14] managed to grow compact InN layers directly on Si(111) by RF-MBE under nearly stoichiometric conditions. However, the...
sample exhibited weak adhesion to the substrate. A LT-InN buffer layer improved adhesion but small voids were introduced at the InN/Si-interface. The highest quality InN epilayers were obtained by the deposition of a GaN/AlN-bilayer buffer. Sakalauskas et al. [15] also studied PAMBE growth of InN on Si(111) and sapphire substrates. For the samples grown on Si(111) with a HT-AlN-buffer layer they reported a narrower FWHM (917 arcsec) of the InN(0002) reflection measured by X-ray diffractometry (XRD) compared to a GaN/AlN-buffer layer (973 arcsec). InN grown on a commercial lumilog GaN:Si on sapphire template with an additional GaN buffer layer yielded an InN(0002) FWHM of 512 arcsec.

Recent advances in metal organic vapor phase epitaxy (MOVPE) growth have also lead to high-quality InN layers. Jamil et al. [16–18] reported MOVPE growth of InN on GaN/Al$_2$O$_3$(0001) and GaN/Si(111) templates. The FWHM of the InN(0002) reflection for the GaN/Al$_2$O$_3$(0001) template was reported as 281 arcsec, indicating high crystal quality [16]. The crystal quality of InN on the GaN/Si(111) template was worse compared to the GaN/sapphire template as indicated by a higher surface roughness and the presence of metallic In reflections in XRD scans.

Despite the large lattice-parameter mismatch between AlN(0001) and Si(111) of $-19\%$ and between InN and AlN of $+13.9\%$, the introduction of an AlN-buffer layer improves the crystal quality of the InN layer. The strain at the AlN/Si(111) interface is relieved by an approximate lattice matching of 5 AlN-crystal planes to 4 Si-crystal planes leading to the introduction of geometrical misfit dislocation arrays. The same mechanism accounts for (partial) strain relief at the InN/AlN interface where approximate lattice matching is achieved for a 7:8-ratio of AlN- to InN-crystal planes [19].

In this work we have studied the microstructure of InN epilayers grown on Si(111) substrates by PAMBE. The InN layers were deposited on AlN-buffer layers with optimized structural properties [20]. The substrate temperature for the InN deposition was systematically varied between 325 $^\circ$C and 375 $^\circ$C under otherwise unchanged conditions. The microstructure was analyzed by scanning electron microscopy (SEM), X-ray diffraction (XRD) and transmission electron microscopy (TEM) combined with energy-dispersive X-ray spectroscopy (EDXS) and electron-energy loss spectroscopy (EELS). The InN layers exhibit a complex microstructure, which changes significantly even within the comparably small interval of substrate temperatures. We have in particular observed and analyzed for the first time inclusions in the substrate and InN layer, which are proposed to be formed by a complex meltback reaction.

2. Experimental details

InN epilayers were grown on Si(111) substrates using a RIBER Compact 21T equipped with an Oxford RF Atom Source H25 nitrogen-plasma source and a standard Knudsen effusion cell for indium. Prior to growth the substrate was rinsed twice in acetone and isopropanol to remove organic contaminants. Subsequently the substrate was chemically cleaned in a HF:H$_2$O (1:50) solution to remove surface oxide layers. After transfer to the loading chamber the substrate was degassed at 130 $^\circ$C for 60 min before the transfer to the growth chamber. The temperature was ramped up to 900 $^\circ$C, allowing the substrate to deoxidize for 30 min. Furthermore the transition from the $7 \times 7$ Si-surface reconstruction to the $\tilde{1} \times \tilde{1}$ reconstruction was observed by reflection high-energy electron diffraction (RHEED). Prior to the growth of InN, an AlN-buffer layer with a thickness of $\sim 30$ nm was deposited at 880 $^\circ$C. The Al-cell temperature was set to 1122 $^\circ$C and the excitation energy of the N-source was set to 400 W with a flux of 0.4 sccm. Under these conditions, AlN-buffer layers with a smooth surface are obtained. Three different samples, P325, P350 and P375, were grown at substrate temperatures of 325, 350 and 375 $^\circ$C, respectively, keeping all other parameters fixed. The In-cell temperature was 810 $^\circ$C, the N-source was excited with 400 W with a flux of 0.5 sccm. The N/III-ratio was controlled via the temperature of the Knudsen effusion cell, the plasma-source excitation energy and the nitrogen flux. All samples were grown in the metal-rich growth regime. The III/N-ratios were determined from post-growth measured beam-equivalent pressures of the constituents. Although the cell parameters were kept constant for all samples, post-growth measurements of the Al/N-ratio indicated that the Al/N-ratio could have been higher due to fluctuations of the cell temperature.

XRD rocking-curves of the (0002) reflection were acquired with a Bruker-AXS D8 Discover diffractometer. SEM was employed to study the surface morphology of the samples. Cross-section TEM specimens were prepared by mechanical grinding and Ar$^+$-ion polishing. The TEM cross-section samples contain two specimen pieces along the [1–10]- and [112]-zone axes of silicon which are oriented perpendicular with respect to each other. Bright-field (BF) and dark-field (DF) TEM imaging as well as high-resolution TEM (HRTEM) etc. was performed using a Philips CM 200 FEG/ST operated at 200 kV. Energy-filtered TEM (EFTEM) images were recorded using a Zeiss 922 Omega [21]. EDXS was performed in an FEI Titan$^3$ 80-300 to determine the chemical composition with high spatial resolution. Composition-sensitive images were taken by high-angle annular dark-field scanning transmission electron microscopy (HAADF STEM).

3. Results and discussion

The SEM images in Fig. 1 visualize the surface structure of the three samples. Sample P325 (Fig. 1a) shows the best surface morphology with only a small density of little dips and few deeper holes (e.g. on the lower right side of Fig. 1a). The density

![Fig. 1. SEM micrographs of (a) P325, (b) P350 and (c) P375.](image-url)
and depth of dips increase for sample P350, which is depicted in Fig. 1b. Sample P375 (Fig. 1c) shows the roughest surface morphology. The density of dips appears to be unchanged compared to Fig. 1b, but the depth of the dips increases further. The shape of the dips, especially in the upper left of Fig. 1c, indicates that the dips are generated as a consequence of island coalescence as suggested in Ref. [22]. Generally, the surface morphology tends to degrade with the increasing growth temperature.

Fig. 2 shows representative cross-section BF TEM images of all samples. Fig. 2a was taken along the [1–100]InN-zone axis of sample P325. SAED patterns (not shown here) were analyzed to determine the orientation relationship between InN and Si which is [1–100]InN || [1–100]AlN,InN and [111]InN || [0001]AlN,InN as already suggested by Grandal et al. [19]. The Si-substrate (bottom of Fig. 2a) shows strain contrast induced by the large lattice-parameter mismatch with respect to the AlN layer. The highly defective AlN-buffer layer is clearly discernible between the substrate and the InN layer. The surface of the InN layer is covered by a thin layer of glue from TEM cross-section sample preparation which was not fully removed during Ar$^+$-ion polishing. The deteriorated surface region on the left-hand side is a TEM-sample preparation artifact. A small surface dip is visible at the right-hand, which is consistent with the SEM image in Fig. 1a. The InN layer contains dislocations predominantly oriented parallel to the [0001]-growth direction. The layer is composed of columnar regions, which show different contrast because the columns are slightly tilted against each other. Dark blotches are visible in the InN layer, which most likely originate from TEM-sample preparation. Towards the left-hand side of Fig. 2a segmented columnar inclusions are observed in the InN layer and a broad inclusion in the substrate (marked by arrows). The segment height of the first inclusion type ranges from 10 to 50 nm, whereas their widths extend between 10 and 200 nm in the whole specimen. These inclusions are typical for P325 and will be discussed in detail later. Fig. 2b shows a BF image of sample P350. The microstructure of the AlN-buffer layer and the strain contrast in the substrate are similar to sample P325. However, the structural properties of the InN layer differ considerably from P325. P350 is characterized by a lower dislocation density and a less pronounced columnar structure. Some voids are formed at the InN/AlN interface (marked by white arrows). The layer surface contains steps and a deeper hole on the right-hand side (black arrow), consistent with the SEM image in Fig. 1b. The deeper hole at the surface is situated directly above a void at the InN/AlN interface indicating that both could have been formed by only partial coalescence of two growth islands. A BF image of sample P375 is shown in Fig. 2c. It shows the same structural features as P350, but the size and density of the voids at the InN/AlN interface are increased.

InN-(0002) XRD rocking curves of the samples are presented in Fig. 3. The largest full-width at half-maximum (FWHM) value is observed for P350. The minimum FWHM occurs for P375 indicating superior crystal quality compared to the samples grown at lower temperatures. Generally it is expected that the FWHM decreases with increasing growth temperature due to the improvement of crystal quality related to higher adatom mobilities. This leads, e.g., to a lower density of larger islands and fewer boundaries due to island coalescence. This process was suggested in Ref. [23] to be responsible for the majority of threading dislocations due to slight misorientations of the growth islands. In this sense, it is surprising that the FWHM of P350 exceeds the value of P325.

To investigate the origin of this unusual behavior, the threading-dislocation (TD) types and densities were analyzed by $g\cdot b$ analysis of dark-field (DF) TEM images [24]. Perfect dislocations in materials with hexagonal crystal structure have Burgers vectors of the type $b=1/3\times<11–20>$, $b=[0001]$ and $b=1/3\times<11–23>$ [25], which correspond to edge-, screw- and mixed-type dislocations for the dominant [0001]-line direction. The [0001]-line directions are expected from the coalescence of slightly tilted or twisted islands during the early stages of growth which leads to an upward bending of the dislocations at island boundaries as outlined, e.g., by Gallinat et al. [23]. The fractions of each TD type were determined by counting the number of TDs in the cross-section TEM

![Fig. 2](image-url)

Fig. 2. Cross-section TEM micrographs of the three samples: (a) P325 in [1–100]InN-zone axis orientation. White arrows indicate inclusions in the substrate and in the InN layer. (b) P350 in [1–100]InN-zone axis orientation. White arrows indicate voids at the AlN/InN interface, black arrow a dip at the surface. (c) P375 in [11–20]InN-zone axis orientation.

![Fig. 3](image-url)

Fig. 3. XRD rocking curves of the InN-(0 0 0 2) reflection including the measured full width at half maximum (FWHM) values for (a) P325, (b) P350 and (c) P375.
specimens, which are plotted in Fig. 4. For each sample, dark-field images taken under two-beam conditions over a width of \(-10\) \(\mu m\) were analyzed. The fraction of mixed-type TDs remains constant within the error margin in contrast to the fraction of pure edge- and pure screw-type TDs, which varies as a function of the growth temperature. In P325 and P375, the majority of TDs have an edge component whereas TDs of pure screw character are less frequent as already reported by other groups [22,23,26]. P350 on the other hand contains almost the same fraction of screw- and edge-type TDs. The origin of the increased density of screw TDs in specimen P350 is unknown. However, the high density of screw dislocations can explain the increased FWHM of P350 because screw-type dislocations distort the (0002) planes resulting in a broadened (0002) rocking curve [27].

The AlN/Si and InN/AlN interfaces were analyzed by HRTEM. Fourier-filtered images (not shown here) using reflections of planes perpendicular to the interface reveal a high density of geometrical dislocations, which is sufficient to relax the lattice-parameter mismatch at these interfaces in a good approximation. The c-lattice parameters of InN and AlN were determined by \(\Theta/2\Theta\) XRD scans. The measured values for InN are \(5.695 \pm 0.007\) Å, \(5.698 \pm 0.007\) Å and \(5.697 \pm 0.007\) Å for P325, P350 and P375, respectively. With respect to the bulk-lattice parameter of \(5.703\) Å, the InN layers are fully relaxed within the error margin. The same applies to the AlN-buffer layers, which are characterized by c-lattice parameters of \(4.991 \pm 0.027\) Å, \(4.992 \pm 0.027\) Å and \(4.987 \pm 0.027\) Å for P325, P350 and P375, respectively. The increased error margins result from the small signal-to-noise ratio of the (0002) reflections due to the small thickness of the AlN buffer layers.

The voids at the InN/AlN interface in samples P350 and P375 were analyzed by HRTEM. Fig. 5a presents an overview image of such a void in sample P350. The interfaces of the AlN-buffer layer are marked by dashed lines. The void is filled with amorphous material, most likely glue from the TEM-sample preparation. The surface of the AlN-buffer layer in the region of the void appears rough and highly defective. Fig. 5b shows the Fourier transform of the AlN-buffer layer from the region marked by a square in Fig. 5a. Numerous reflections are discernible that do not belong to the \([1\overline{1}00]_{\text{AlN}}\)-zone axis or the Si-substrate. Circles connected by a dashed line mark reflections of cubic AlN along the \([10\overline{1}]\) -zone axis. Square symbols connected by a dash-dotted line denote wurtzite AlN oriented along the \([11\overline{2}3]\)-zone axis. The deteriorated surface along with the polycrystallinity of the buffer layer may have prevented the direct deposition of InN in this region. The increasing size and density of the voids with increasing growth temperature may be correlated with the increasing adatom mobility, which reduces the probability that InN is directly deposited on defective buffer layer regions. Another feature of the void is the small precipitate at the top surface (arrow in Fig. 5a). A Fourier transform of this region is shown in

\[\text{Fig. 5.} \] High-resolution TEM image of a void at the AlN/InN interface. (b) Fourier transform of the defective AlN-buffer layer indicated by black square in (a). Circles denote cubic AlN reflections in \([1\overline{1}0\overline{1}]\)-zone axis orientation, squares wurtzite AlN reflections along \([1\overline{1}23]\). Those not marked are Si and AlN reflections along \([1\overline{1}0\overline{1}]\) and \([1\overline{1}20]\), respectively. Dashed lines are guides to the eye. (c) Fourier transform of the region marked by the black arrow. Squares denote InN reflections in \([1\overline{1}20]\)-zone axis orientation, circles In\(_2\)O\(_3\) reflections in \([10\overline{1}]\)-zone axis orientation.
Fig. 5c where InN oriented along the [11\textendash 20]-zone axis is marked with squares. Reflections marked with circles belong to In$_2$O$_3$ in [101]-zone axis orientation. The presence of In$_2$O$_3$ can be correlated with the large band gap energy of 1.9 eV formerly reported for bulk InN [28]. The presence of In$_2$O$_3$ is undesirable but the influence of a small volume fraction of precipitates on the physical properties of the whole InN layer is negligible.

Fig. 6 shows two DF images recorded with the (000\textendash 2) and (0002) reflections of sample P325. A narrow band-like contrast is visible in the center of both images that shows complementary contrast with respect to the contrast of the surrounding layer. In Fig. 6a it is darker than the surrounding matrix whereas it appears brighter in Fig. 6b. Such line-like features with complementary contrast are inversion domains (IDs) where the occupation of the In- and N-sublattices is inverted. The ID found in sample P325 is about 10 nm wide, starts inside the layer and propagates to the surface. The region below the ID appears dark under these imaging conditions, indicating a different structure or crystal orientation but not a void. Previously described IDs in InN layers are characterized by a V-shaped morphology [29,30] and were observed in Mn-doped layers. Jasinski et al. [29] concluded that IDs nucleate at Mn or other impurities. Wang et al. [30] reported the formation of IDs if the Mn-doping exceeds a critical value. In this sense, the observation of IDs in undoped InN is in contradiction to IDs reported previously.

Fig. 6 shows in addition the aforementioned segmented columnar (white arrow in the center of Fig. 6a) and trapezoidal inclusions (black arrows) in the InN layer and Si. The occurrence of the trapezoidal structures in the substrate is usually correlated with inclusions in the InN layer above as already shown in Fig. 2a. Further analyses were performed to clarify the origin of these two inclusion types. Fig. 7a shows a HAADF STEM image of sample P325. A trapezoidal inclusion is discernible in the Si substrate as well as segmented columnar inclusions in the InN layer above. The inset of Fig. 7a shows a SAED pattern of a trapezoid in the Si substrate.

Fig. 6. Cross-section TEM dark-field micrographs of P325 taken with (a) $g_{\text{0002}}$ and (b) $g_{\text{0002}}$. The upper white arrow marks the inversion domain. The white arrow below points to a segmented inclusion (segment interspaces only visible in bright-field imaging, not shown here). Black arrows mark trapezoid inclusions in the substrate.

Fig. 7. (a) HAADF STEM image of P325. A black square indicates the region for the elemental mappings. The inset shows a SAED pattern of a trapezoid in the Si-substrate. (b), (c) and (d) Elemental distributions for In, N and Si, respectively. White arrows indicate the interspaces of the segmented inclusion.
crystalline nature of the inclusion. Reflections belonging to Si along the [11–2]-zone axis are observed. The remnant reflections can be assigned to indium with a tetragonal structure (a = 3.25 Å, c = 4.95 Å), which is oriented along the [−1−31]-zone axis. A clear orientation relation between the Si-substrate and the In-inclusion is evident with [111]_{Si} || [101]_{In}. Other SAED patterns taken along the [1−10]_{Si}-zone axis also show well-defined orientation relationships between Si and In with [111]_{Si} || [101]_{In} and [1−10]_{Si} || [100]_{In} or [−111]_{In}. The borders of the trapezoidal structures appear to be aligned parallel to the [111] planes of the Si-substrate. Typical inclusion thicknesses are between 30 and 50 nm, and widths extend from 50 to 1500 nm.

To analyze the element distribution, EDXS mappings were recorded using the N-K, In-K and Si-K lines, which are shown in Fig. 7b–d for In, N and Si, respectively. The location of the mappings is marked with a black square in Fig. 7a. The In-distribution in Fig. 7b shows that the inclusion contains In but the signal weakens towards the left edge of the trapezoid whereas the Si-signal weakens towards the right-hand side. The lack of N (Fig. 7c) in this region indicates that the inclusion may consist of pure indium. The complementary behavior of the Si- and In-signals results from the partial embedding of the inclusion in Si along the electron-beam direction. In is also detected in the AlN-buffer layer directly above the inclusion. Focusing on the segmented inclusions in the InN layer, Fig. 7c indicates a N-deficit in these regions whereas the interspaces (one is marked with a white arrow) contain a higher N-concentration. As the analyzed TEM-sample region is rather thick, element distributions were additionally recorded by EFTEM in thinner specimen regions to ensure that only the inclusions are analyzed without the surrounding InN matrix along the electron-beam direction. The EFTEM images (not shown here) confirm that the segments do in fact not contain any N, and that the In-signal is stronger in the segments compared to the InN layer. The Si-signal in the trapezoid contains Si. Si is also detected in the interspaces between the segments, which are marked by white arrows suggesting that the interspaces consist of SiN.

Fig. 8 presents an HRTEM image of a segmented inclusion in P325. The surrounding InN matrix is oriented along the [11−20]-zone axis. The material in the interspaces shows an amorphous structure consistent with the presence of SiN. Fourier analysis of the bottom segment yields a [100]-zone axis orientation of indium.

Trapezoidal inclusions in the substrate were described by Kaiser et al. [31] for the growth of AlN on Si(111). In their specimens, the trapezoids consist of Al. They concluded that the trapezoids are formed by a local meltback reaction. The latter is related to a significant reduction of the melting temperature of Si if it is solved in a liquid metal, which is typically present during group-III nitride growth under metal-rich conditions. Ishikawa et al. [32] reported a meltback reaction for GaN grown on Si(111). Recently, Ishikawa and Shimanaka [33] studied meltback reactions of Ga, Al and In with Si by depositing these metals on Si-substrates. Atomic force microscopy revealed the depth of the meltback regions, which corresponds well to the depth of the trapezoidal structures in our specimens and the samples analyzed by Kaiser et al. [31]. We therefore conclude that the inclusions in our specimens are also formed by a meltback reaction.

However the fact that the trapezoids are filled with In is striking. The Si-substrate is only in direct contact to AlN during growth, suggesting the trapezoids should be filled with Al. The following three possibilities can be envisioned to know how In-filled trapezoids can be formed.

(a) The trapezoids may first contain Al during AlN growth, which is replaced by In during InN growth. Indium can reach the Si-substrate through holes in the AlN-buffer layer, which can be observed in HRTEM images, e.g. in the inset of Fig. 9, where the AlN is missing on top of an In-inclusion. The replacement of Al by indium in the trapezoids is possible by a meltback reaction in which the melting temperature is reduced by In. If Al in a trapezoid is replaced by In, Al should be detectable within the InN layer. One Al-precipitate (black arrow in Fig. 9) could be indeed detected as shown in the composition-sensitive HAADF-STEM image in Fig. 9. Chemical analysis by EELS confirms that the precipitate consists of Al. The rare occurrence of Al-precipitates compared to the high trapezoid density suggests that only few Al-filled trapezoids are formed initially.

(b) Alternatively, the Al-meltback reaction leads to the formation of voids in the Si-substrate as reported by Kaiser et al. [31], which
are filled with In during InN deposition as suggested in (a). The presence of Si (Fig. 8d) in the InN layer indicates, that In enlarges the voids and the molten Si is deposited in the InN layer.

(c) The meltback reaction is originally initiated by indium in defective regions of the AlN-buffer layer.

Processes (b) and (c) can also explain the formation of the segmented In-inclusions in the InN layer in the following way. The molten Si forms a solution with the liquid In-surface film during growth. If the Si-fraction reaches a critical value, a eutectic transformation occurs, which leads to spatially separated In- and Si-segments. As growth continues, part of the precipitated Si forms amorphous Si$_x$N$_{y}$ due to the arriving nitrogen flux, whereas the other part is meltback-etched again. The eutectic transformation then reoccurs, creating another segment. This process continues until there is not enough Si anymore to reach the critical Si-fraction for the eutectic transformation.

Sample P325 shows a high density of In-trapezoids and segmented inclusions whereas the other two samples P350 and P375 rarely contain any inclusions. However, these samples contain voids at the InN/AlN interface with a density in the same order of magnitude. This indicates a possible connection between the two phenomena, because the AlN-buffer layer is defective above trapezoids in P325 as well as below voids in P350 and P375. In P325 indium penetrates the AlN-buffer layer during growth and dissolves the substrate. In the other two specimens, overgrowth of these faulty buffer layer regions may be inhibited by higher adatom mobilities. Another plausible explanation is provided by the Al/N-ratio because the AlN-buffer layer of P325 was grown with the highest Al/N-ratio of all three samples and filled trapezoids only occur for metal-rich growth conditions [31].

4. Summary

The microstructure of PAMBE-grown InN layers was investigated by different electron microscopic techniques and XRD. Only the substrate temperature was varied between 325°C and 375°C under otherwise fixed growth conditions which leads to the following temperature-dependent changes of the microstructure:

- The surface roughness increases with the temperature due to the formation of large and deep holes.
- The FWHM of the (0002) rocking curves indicates a superior crystal quality of the sample grown at 375°C compared to the other samples. A particularly large FWHM value for the sample grown at 350°C can be correlated with a high fraction of screw-type threading dislocations.
- Voids are formed at the InN/AlN-buffer layer interface at temperatures of 350°C and 375°C. Their size and density increases with the growth temperature. The formation of voids is correlated with defective regions of the AlN-buffer layer.
- Inversion domains are observed in all samples. This is in contradiction to the previous work where the formation of inversion domains was correlated with Mn-doping.
- The sample grown at 325°C contains two types of inclusions, which occur in high densities. In-filled trapezoidal inclusions in the Si-substrate and segmented In$_x$Si$_{1-x}$N$_y$ inclusions above the In-trapezoids. A meltback reaction is suggested to be responsible for the formation of these defects, which is based on the significant lowering of the melting temperature if silicon is in contact with liquid indium or aluminum. We conclude that these defects are absent if high III/N-ratios are avoided during growth.

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