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The influence of substrate surface preparation on LP MOVPE GaN epitaxy on differently oriented 4H-SiC substrates

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ABSTRACT

The influence of surface preparation and off-cut of 4H-SiC substrates on morphological and structural properties of GaN grown by low-pressure metalorganic vapor phase epitaxy was studied. Substrate etching has an impact on the surface roughness of epilayers and improves its crystal quality. The GaN layers were characterized by atomic force microscopy (AFM) and high-resolution X-ray diffractometry (HRXRD) measurements. It was observed that on-axis 4H-SiC is most suitable for GaN epitaxy and that substrate etching improves the surface morphology of epilayer.

1. Introduction

The application potential of III-N compounds, especially GaN, is widely known. Advantages of this wide bandgap, high thermal stability and high breakdown voltage material make it a good candidate for electronic devices. Most gallium-nitride-based epitaxial structures are grown on sapphire as relatively low-cost substrates, but sapphire exhibits several disadvantages for power applications. In particular, apart from the high mismatch of sapphire to GaN, high-power devices require a substrate with high thermal conductivity. This condition is not fulfilled by sapphire. Silicon carbide substrates are much better candidates for such areas. However, SiC substrates as-received from a supplier have a scratch-covered surface, an example of which is shown in the atomic force microscopy (AFM) image in Fig. 1a. Poor substrate surface morphology is not acceptable for epi-growth of GaN layers, but a meaningful improvement of the substrate surface quality is provided by wafer polishing laboratories (e.g. Novasic) or by means of an in-situ substrate preparation process [1]. Thus, the surface preparation is required and it has a big impact on device performance because it can primarily improve epitaxial layer roughness. Very good crystalline quality of the epilayers with an RMS close to 0.3 nm was reported for GaN-based HEMT structure [2]. GaN layers with the best surface roughness (RMS = 0.18 nm) were obtained for growth of *m*-plane GaN on *m*-plane SiC substrates [3]. The crystallographic structure was improved and the full-width at half-maximum (FWHM) value of the X-ray diffractometer (XRD) rocking curve reached 50 arcsec for GaN on SiC [2,4]. The advantages of on-axis SiC substrate for GaN growth have also been reported [5].

The aim of this work is to present the influence of the SiC substrate off-cut and substrate surface preparation prior to the GaN growth and its crystal quality. The presented results suggest that the growth of gallium nitride on (0 0 0 1)-oriented 4H-SiC substrate combined with surface preparation has led to improvement of epilayers' quality.

2. Experimental procedure

The experiment was divided into two parts. The first one was substrate preparation for growth and the second one was the gallium nitride growth. In the presented studies, (0 0 0 1)-oriented and also 4° and 8° misoriented toward $(11\bar{2}0)$ N-type 4H-SiC and 8°-misoriented toward $(11\bar{2}0)$ semi-insulating (SI) 4H-SiC were used as substrates for epitaxy. The surface preparation and GaN epitaxy were performed on the Si-face of SiC substrates. The substrates were cut into 1.5 cm × 1.5 cm pieces, cleaned in alcohol and deionized water and dried in nitrogen flow. Then half of them were prepared in a special surface preparation process as described below, while the other half were used "as-received" from the supplier.

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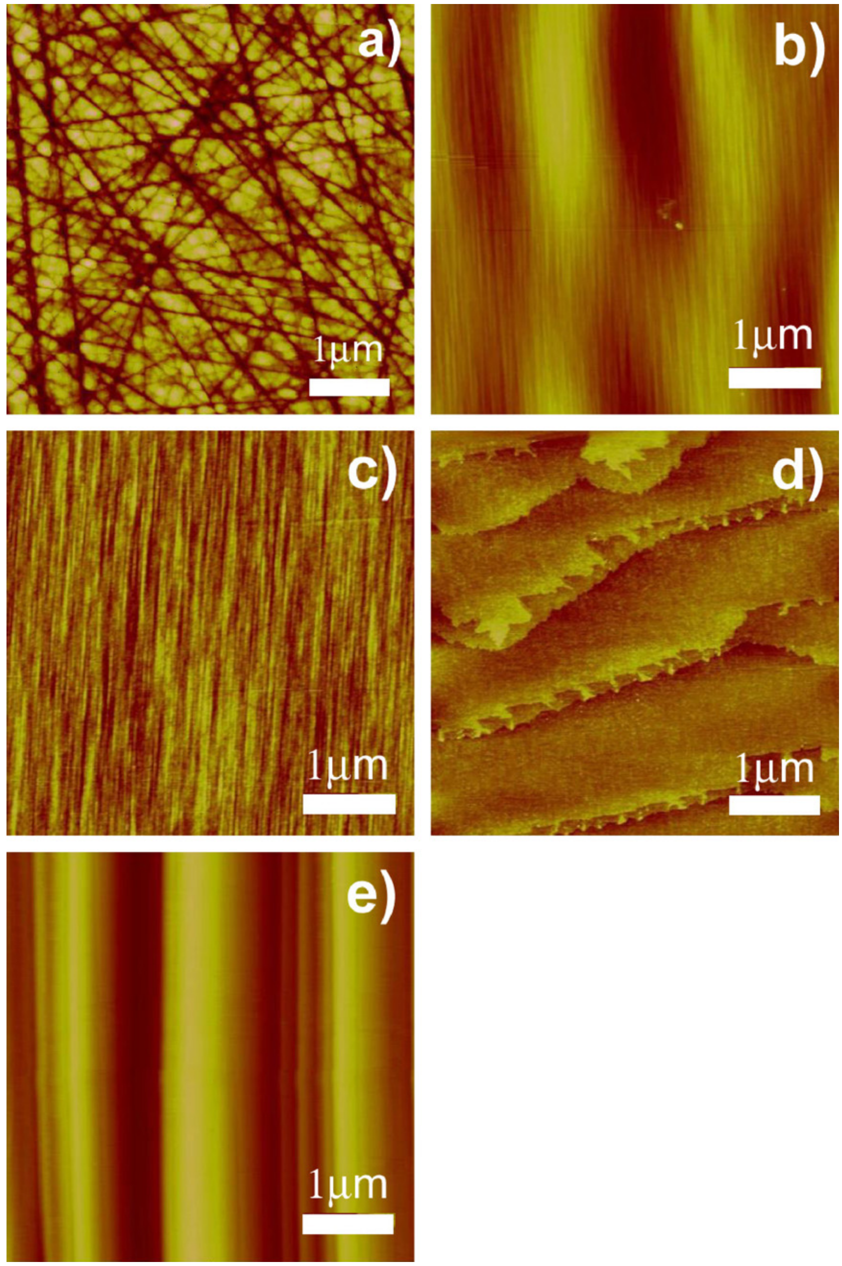

Fig. 1. 4H-SiC substrates' surface morphology of (a) as-received substrate (orientation 8° off-axis), (b) 8E, (c) 8SIE, (d) 0E and (e) 4E, after surface preparation (scan size: 5 μm × 5 μm).

Substrate surface etching was done in a horizontal hot-wall chemical vapor deposition (CVD) reactor with a SiC- and TiC-coated graphite susceptor inductively heated by an RF generator. In-situ H_2+C_3H_8 [6] etching was prepared at ~1600 °C temperature for 10 min, which are optimal conditions for 8° off-axis SiC substrates according to Strupinski et al. [7]. To keep all conditions stable all of the prepared substrates were etched in the same process. Next, the gallium nitride epitaxial layers were deposited also during one growth process on the etched substrates (marked with E) and on as-received ones (marked with N). GaN layers were grown on silicon carbide (as-received and etched) substrates in an AIX 200/4 RF-S metalorganic vapor phase epitaxy low-pressure reactor (LP MOVPE). In the beginning 100-nm-thick aluminum nitride layers were deposited as a wetting layer [8,9], followed by 1400 nm of gallium nitride. The source gases were trimethylaluminium (TMAl, 20 sccm), trimethylgallium (TMGa, 20 sccm) and ammonia (NH_3, 2000 sccm for both AlN and GaN). The growth temperatures were 1070 and 1115 °C, respectively. The reactor pressure was 50 mbar for both layers. High-purity hydrogen was used as a carrier gas. In contrast to the growth on sapphire, the high-temperature surface desorption step was absent from the recipe.

The influences of SiC surface etching and substrate off-cut on gallium nitride layers' properties were examined. The surface morphologies of etched substrates and grown epilayers were studied using differential interference contrast (DIC) or Nomarski optical microscopy and AFM.

The method of defect-selective etching of GaN surface in molten NaOH–KOH eutectic with addition of 10% of MgO powder was used for revealing dislocations [10]. SEM images of etched epilayers allowed to count the dislocation density.

The X-ray measurement (FWHM) of the rocking curve of the 200 reflection by means of a double-crystal diffractometer was applied also to analyze an epilayer structure [11]. The X-ray measurements were performed on a high-resolution X-ray diffractometer (HRXRD) equipped with a Ge 440 Bartels monochromator using $CuK_{\alpha 1}$ radiation with wavelength amounting to 0.15405 nm from a 2.2 kW ceramic Philips tube.

3. Result and discussion

The images from the Nomarski microscope show that surface preparation improves GaN layer morphology. The effect of the surface improvement occurred for all investigated substrates, suggesting that during the substrate preparation hydrogen and propane effectively react with SiC surface and remove scratches.

The investigation on the nano-scale (AFM) suggests that the surface preparation process for the analyzed off-cut has occurred in a different way. The applied conditions of substrate etching were developed for 8° off-cut. For such a substrate misorientation the AFM results (Fig. 1b and c) of surface roughness for samples 8E and 8SIE are better than for 8N and 8SIN. Table 1 summarizes the AFM statistics obtained for the investigated substrate orientations. In case of the 0N and 0E (shown in Fig. 1d), the surface preparation process also improved the surface roughness. The applied etching process has changed the value of RMS from 1.84 to 0.05 nm (R_a from 1.49 to 0.11 nm). Unfortunately, for substrates with 4° off-cut after removing the scratches, the surface starts to be etched selectively and the surface roughness increases strongly as shown in Fig. 1e. The gallium nitride epilayers deposited on the investigated substrates were characterized by AFM. The surface roughness results are also presented in Table 1. We conclude that in case of 8° off-axis samples the surface preparation has an impact on the GaN epilayer roughness only for N-type SiC. For the SI SiC there is no improvement of the RMS value. Epilayers deposited on the 4° off-axis etched substrate duplicate the substrate's morphology, resulting in a very rough GaN epilayer.

The best results were obtained for the substrates with 0° off-cut. The surface roughness of the GaN epilayer deposited on the 0E substrate reached the lowest value compared to the other substrate orientations. It was possible to improve the RMS from 1.90 nm ($R_a = 1.50$ nm) to 0.06 nm ($R_a = 0.14$ nm) by surface preparation.

The substrates' off-cut has a crucial influence on GaN epitaxy. GaN epitaxial layers on the substrates with misorientation from (0 0 0 1) direction are characterized by higher surface roughness than the layers grown on the substrates with 0° off-axis. For the off-cut substrates a huge number of defects (nanopipes) appear on the epilayer surface (Fig. 2a). In case of growth on 0° off-axis substrate (Fig. 2b), there are edge, screw and mixed type dislocations (for 0E-GaN the dislocation density is $(3–4)\times 10^8\,cm^{-2}$), but in comparison with off-cut substrates it is quite a low number (e.g. for 8E-GaN the dislocation density is $(1–2)\times 10^9\,cm^{-2}$). The results from X-ray measurements are presented in Table 2. In case of this method the broadening of the symmetric reciprocal lattice point (RLP) of the GaN epitaxial film in the direction perpendicular to the surface $\sigma_\perp$ depends only on a small correlation length normal to the substrate surface and a heterogeneous strain along the *c*-axis, while the broadening of this point in the direction parallel to the surface σ_{II} is influenced only by the tilt, i.e. out-of-plane misorientation and the small correlation length parallel to the substrate surface.

Using triple-axis HRXRD it is possible not only to determine the broadening $\sigma_\perp$ of the 220 RLP by measuring the FWHM of the diffraction curve obtained in the radial scan ($2\theta/\omega$-scan), but,

Table 1
The AFM surface roughness results (in nm) for: as-received (N) and etched (E) differently off-cut substrates and also for GaN layer on the as-received (N-GaN) and etched (E-GaN) substrates

Disorientation angle (deg)	(nm)	N	E	N-GaN	E-GaN
0	RMS	1.84	0.05	1.90	0.06
	Ra	1.49	0.11	1.50	0.14
4	RMS	1.64	11.39	1.60	7.48
	Ra	1.25	9.53	1.36	6.40
8	RMS	1.75	1.37	2.21	1.36
	Ra	1.37	1.22	1.89	1.36
8SI	RMS	1.32	0.10	1.31	1.59
	Ra	0.96	0.17	1.17	1.26

Table 2
XRD FWHM results (in arcsec) for GaN layers deposited on 4H-SiC N-type with misorientation angles 0°, 4°, 8° and for semi-insulating 4H-SiC 8° off-axis marked as 8SI

Disorientation angle (deg)	$\sigma_\perp$ (arcsec)		σ_{II} (arcsec)	
	N-GaN	E-GaN	N-GaN	E-GaN
0	136	40	275	80
4	199	95	276	206
8	72	85	197	184
8SI	100	78	188	60

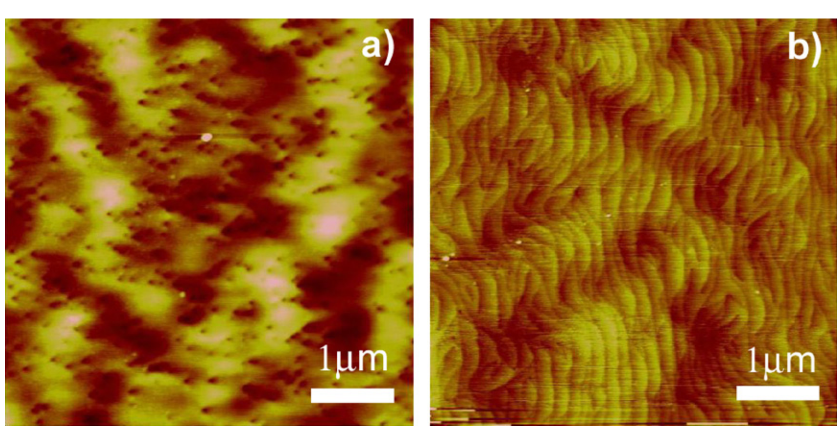

Fig. 2. Surface morphology of a GaN grown on: (a) 4H-SiC 8° off-axis (8E) and (b) 4H-SiC 0° off-axis (0E). The scan size is 5 μm × 5 μm.

performing angular scan (ω-scan), it is possible also to determine the σ_{II} as a FWHM of this diffraction profile.

The results show improvement of the crystallographic structure of GaN deposited on etched substrates. Surface preparation has an impact on the epilayer mosaicity by reducing grain misorientation (twist and tilt) which is observed in σ_{II} FWHM value. From the point of view of the device performance, the most important is the degree of crystalline order in the growth direction measured by $\sigma_{\perp}$. The best results were obtained for 0N-GaN (FWHM = 40 arcsec) and 8SIE-GaN (FWHM = 78 arcsec).

4. Summary

The growth of GaN epilayer on (0 0 0 1)-oriented and also 4° and 8°-misoriented toward $(11\bar{2}0)$ 4H-SiC substrates was analyzed. The influence of substrate surface preparation was also compared. Results of surface roughness measurement showed that the substrate etching before the growth had a crucial impact on the deposited layer surface. The best result of RMS = 0.06 nm (R_a = 0.14 nm) was obtained for GaN epilayer deposited on the etched 4H-SiC 0° off axis.

From X-ray analysis we concluded that the crystallinity of GaN epilayers is good on 4H-SiC 0°-off and 4H-SiC 8°-off substrates, but that the latter is not acceptable due to the high number of nanopipes in the GaN epilayer surface and a high value of RMS roughness.

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