

# Electron-stimulated formation of the AlN crystalline structure on the reconstructed ( $\sqrt{31} \times \sqrt{31}$ ) $R\pm 9^\circ$ sapphire surface

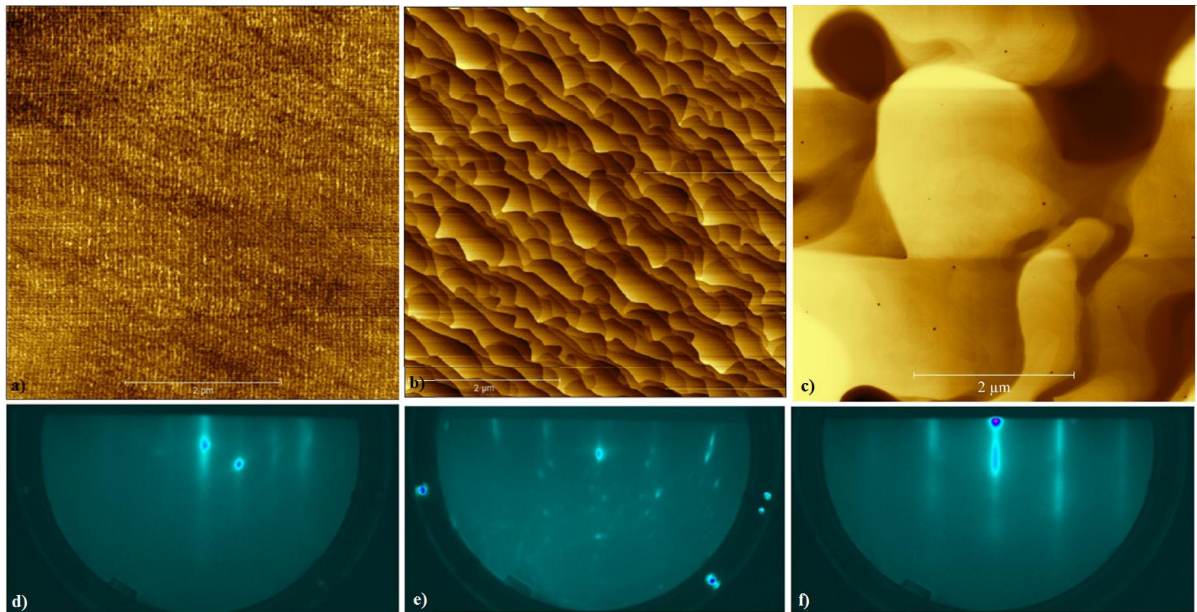
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III-nitrides are direct-gap semiconductor materials with a band gap of 0.7 eV (InN) and 3.4 eV (GaN) to 6.2 eV (AlN). III-nitride low-dimensional structures (quantum wells, superlattices and quantum dots) are of considerable interest due to their possible practical applications for creating light-emitting devices and photodetectors. III-nitride heterostructures with two-dimensional electron gas are actively used to create high-power microwave and power transistors. No less relevant at the moment is the use of III-nitrides in the form of graphene-like layers (g-AlN and g-GaN) in spintronics and electronics. To date, the most widely used substrate for the III-nitrides epitaxial growth is sapphire ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>). It is believed that when high-temperature (over 1150 °C) heating the sapphire substrate, decomposition of the surface layers of Al<sub>2</sub>O<sub>3</sub> occurs with oxygen desorption and the formation of metallic aluminum [1,2]. The high resolution AFM images of the initial Al<sub>2</sub>O<sub>3</sub> surface and after high-temperature annealing are shown in Fig. 1a,b. In this case, a reconstruction transition occurs ( $1 \times 1$ ) - ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  [3]. The sapphire reconstructed surface ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  is stable and improves the adhesion of growth components [1]. This work is devoted to the study of the fast electrons effect ( $E \sim 11$  keV) on the crystalline AlN formation on the reconstructed ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  sapphire surface. It was established experimentally that the reconstructed ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  sapphire surface under an ammonia flux nitrated at low speed compared to the unreconstructed sapphire surface ( $1 \times 1$ ), but in the presence of an electron beam the nitridation process rate increases. The order-disorder reconstruction transition ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  - ( $1 \times 1$ ) (the diffraction pattern in Fig. 1e is replaced by 1d with subsequent accelerated nitridation of the disordered sapphire surface ( $1 \times 1$ ) occurs. Since the reconstructed surface, is said to be enriched with metallic aluminum, it was fair enough to assume that the reconstruction ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  contributes to the nitridation process due to the high chemical activity of metallic aluminum, but this contradicts our experimental data. A series of experiments was carried out on deposition of 1-2 monolayers of metallic aluminum on the surface of sapphire to reproduce the reconstruction ( $\sqrt{31} \times \sqrt{31}$ )  $R \pm 9^\circ$  proposed in [1,2,4]. As a result, it was found that when aluminum is deposited at a temperature above 800 °C (this temperature is much lower than the reconstruction formation temperature ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$ ), all the adsorbed aluminum desorbs from the surface and the crystalline phase of aluminum does not form. After reducing the substrate temperature to 200 °C, deposition of aluminum on the sapphire surface led to the formation of crystalline aluminum with a 30° unit cell rotation relative to the sapphire unit cell, however, no reconstruction reflexes were detected in the diffraction pattern (Fig. 1f). These experimental results refute early work, representing the reconstructed ( $\sqrt{31} \times \sqrt{31}$ )  $R\pm 9^\circ$  sapphire surface in the form of 1-2 monolayers (111) of monocrystalline aluminum. It can be assumed that the reconstructed surface does not consist of metallic reactive aluminum, but is formed as oxides of partially reduced aluminum (Al<sub>2</sub>O and AlO) with the formation of the more stable ordered phase [5]. The formation of the crystalline AlN phase under the high-energy electrons action in this case can be explained by the fact that the electron beam initiates electron-stimulated desorption of oxygen from the sapphire surface and accelerates the nitridation reaction.

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- [1] G. Renaud, B. Villette, I. Vilfan, A. Bourret, Phys. Rev. Lett., **73**, 1825 (1994).
- [2] J.V. Lauritsen, M.C.R. Jensen, K. Venkataramani et al., Phys. Rev. Lett., **103**, 76 103 (2009).
- [3] Y. Wu, A. Hanlon, J.F. Kaeding, R. Sharma, P.T. Fini et al., Appl. Phys. Lett., **84(6)**, 912 (2004).
- [4] C. Barth, M. Reichling. Nature, **414**, 54 (2001).
- [5] T. M. French and G. A. Somorjai, J. Phys. Chem. **74**, 2489 (1970).



**Figure 1** High resolution AFM images of  $5 \times 5 \mu\text{m}$  and diffraction patterns of the initial  $\alpha\text{-Al}_2\text{O}_3$  surface close to the symmetric azimuth  $[11\bar{2}0]$  - a) and d); reconstructed ( $\sqrt{31} \times \sqrt{31}$ )  $R \pm 9^\circ$  surface of  $\alpha\text{-Al}_2\text{O}_3$  after high-temperature annealing ( $1150^\circ\text{C}$ ) - b) and e); crystalline Al close to the symmetric azimuth  $[10\bar{1}0]$  - c) and f). The scale along the z axis for a) and b) is 2nm, for c) is 20nm.