Surface Treatment for Stepped Vicinal (100) β-Ga_2O_3 Substrates for Homoepitaxial Growth by *S*-MBE

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Abstract

β-Ga_2O_3 is an ultrawide bandgap (UWBG) semiconductor of distinct interest for its high breakdown electric fields and its unique ability among UWBG semiconductors to be grown using the Czochralski method, allowing for the bulk synthesis of doped substrates.[1]

Homoepitaxial film growth of β-Ga_2O_3 on the (100) plane has been historically limited by the formation of twin boundaries, which limit the electron mobilities of devices. This work investigates the ideal cleaning, etching, and annealing of various vicinal (100) β-Ga_2O_3 substrates to obtain a smooth step-terrace surface with RMS roughness values <600 pm for the reduction of twin boundaries in films grown by suboxide-molecular beam epitaxy.

Methods

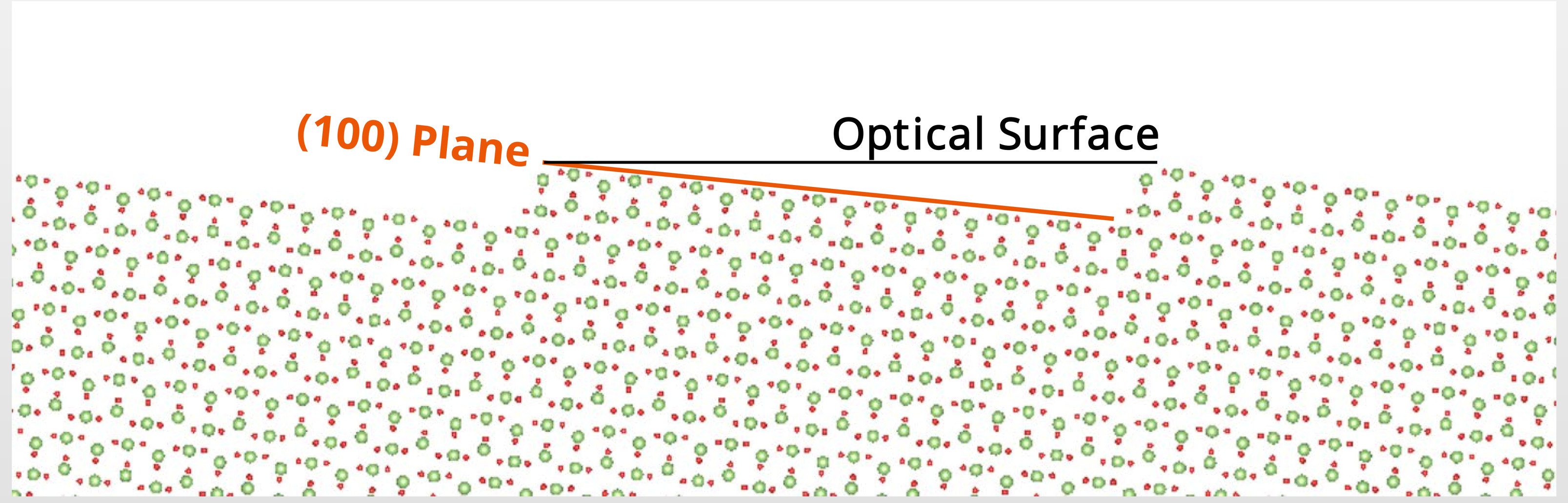
Semi-insulating vicinal gallium oxide substrates doped with magnesium and iron were purchased from CrysTec and Novel Crystal Technology (NCT), respectively.

Out of box substrates were sonicated in solutions of acetone, isopropanol, and deionized water for 30 minutes each, followed by a spin-rinse in DI water to clean their surfaces of large particles. Mg-Ga_2O_3 substrates were etched with hot H_3PO_4 at 140 °C,[2] while Fe-Ga_2O_3 substrates were etched with 47% HF at room temperature,[3] as previous reports have demonstrated successful etching of β-Ga_2O_3 at these conditions. An etch time of 15 minutes was determined to fully etch the surface layers without the formation of deep cavities on the surface.

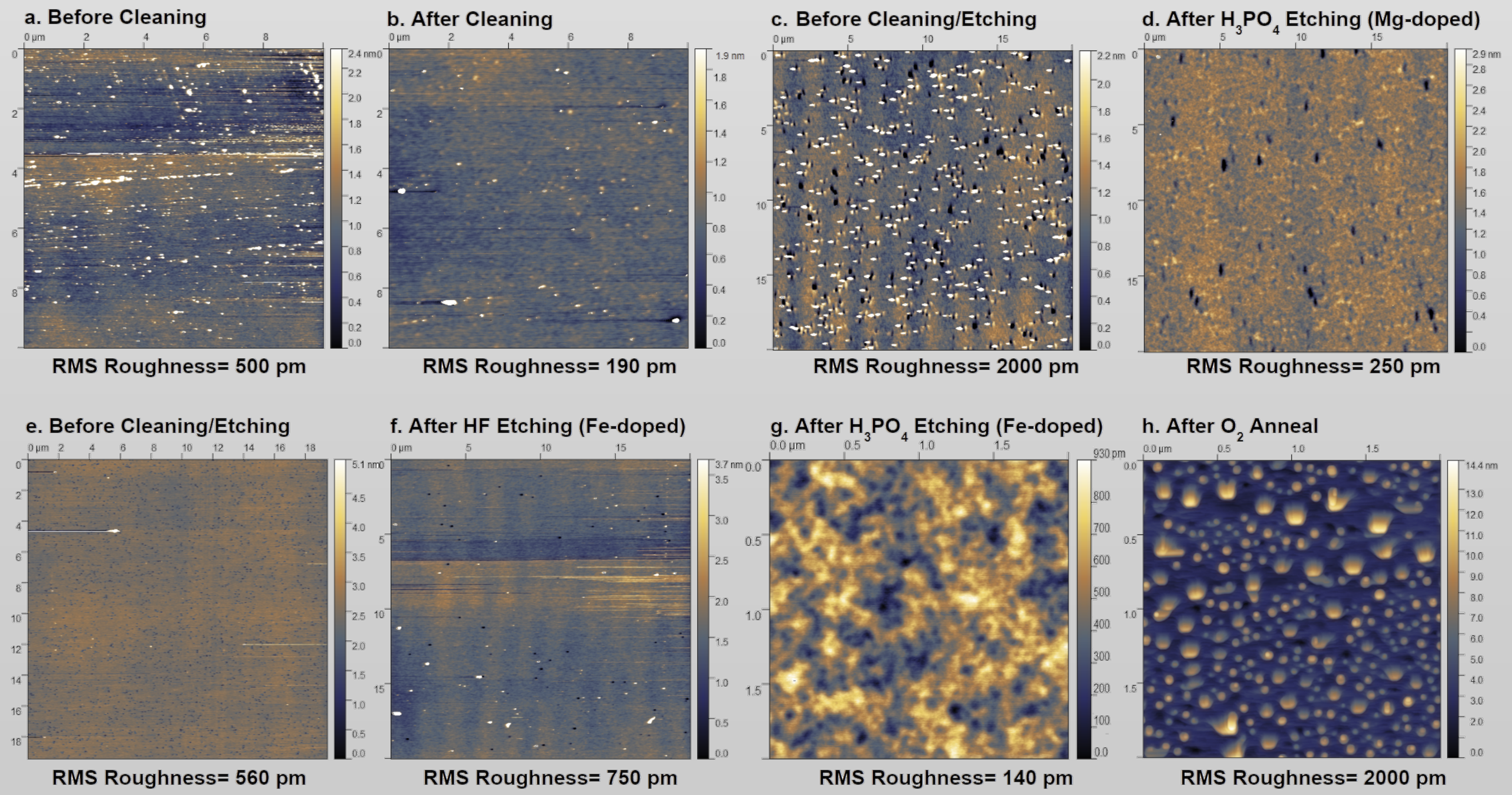
Surface morphologies at each step of the preparation process were studied using atomic force microscopy (AFM) images collected using an Asylum Research Cypher S SPM system operating in AFM tapping mode at a scan rate of 3.91 Hz and using a 10 nm radius tip.

Results

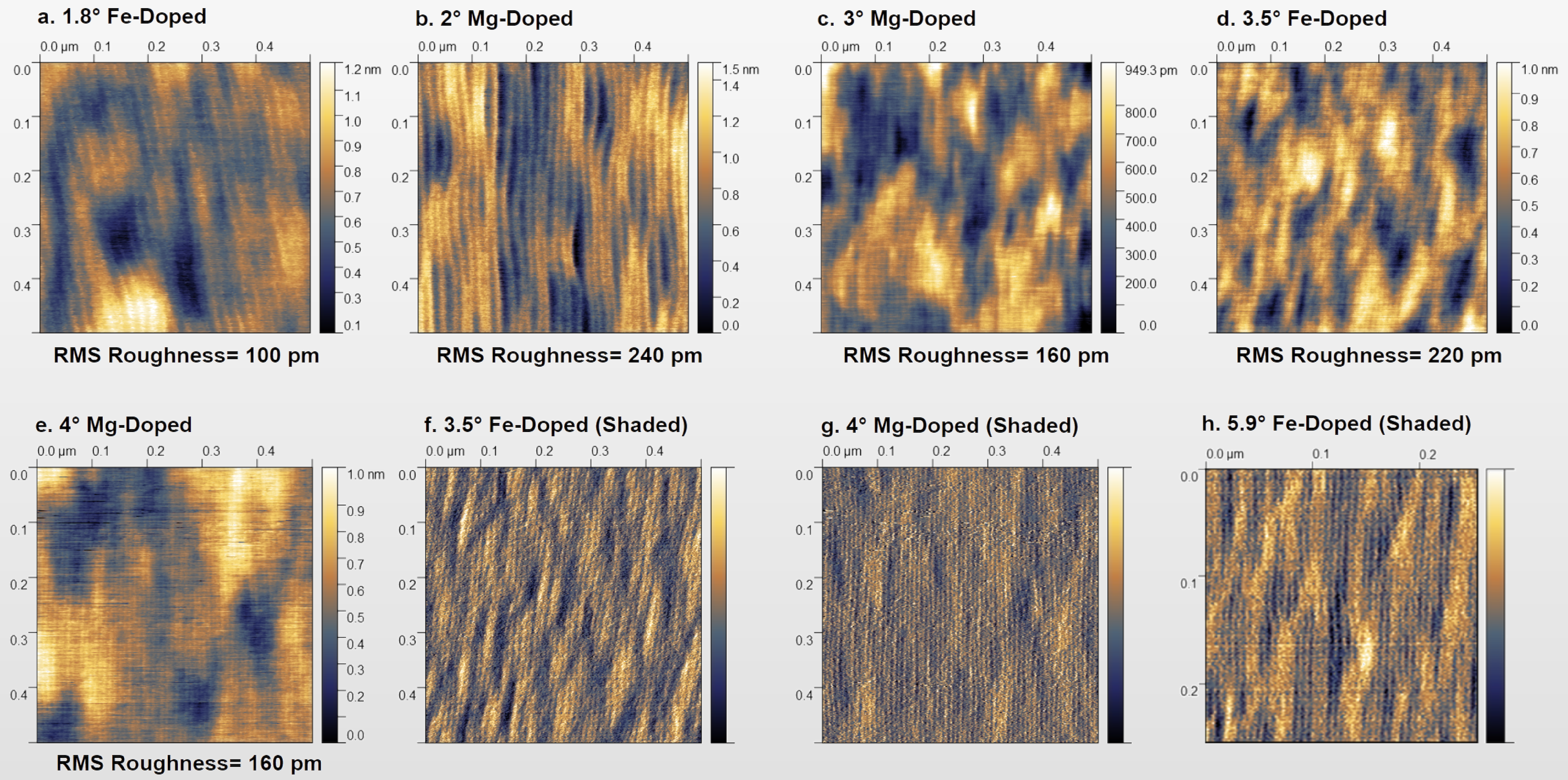
Vicinal substrates, as shown below, suppress the formation of twins within films by forcing the orientation of the crystal and favoring step-flow growth over islanding. This work prepares substrates 1.8° - 5.9° miscut from the (100) normal, which lack step-terrace structures and have rough surfaces when purchased.



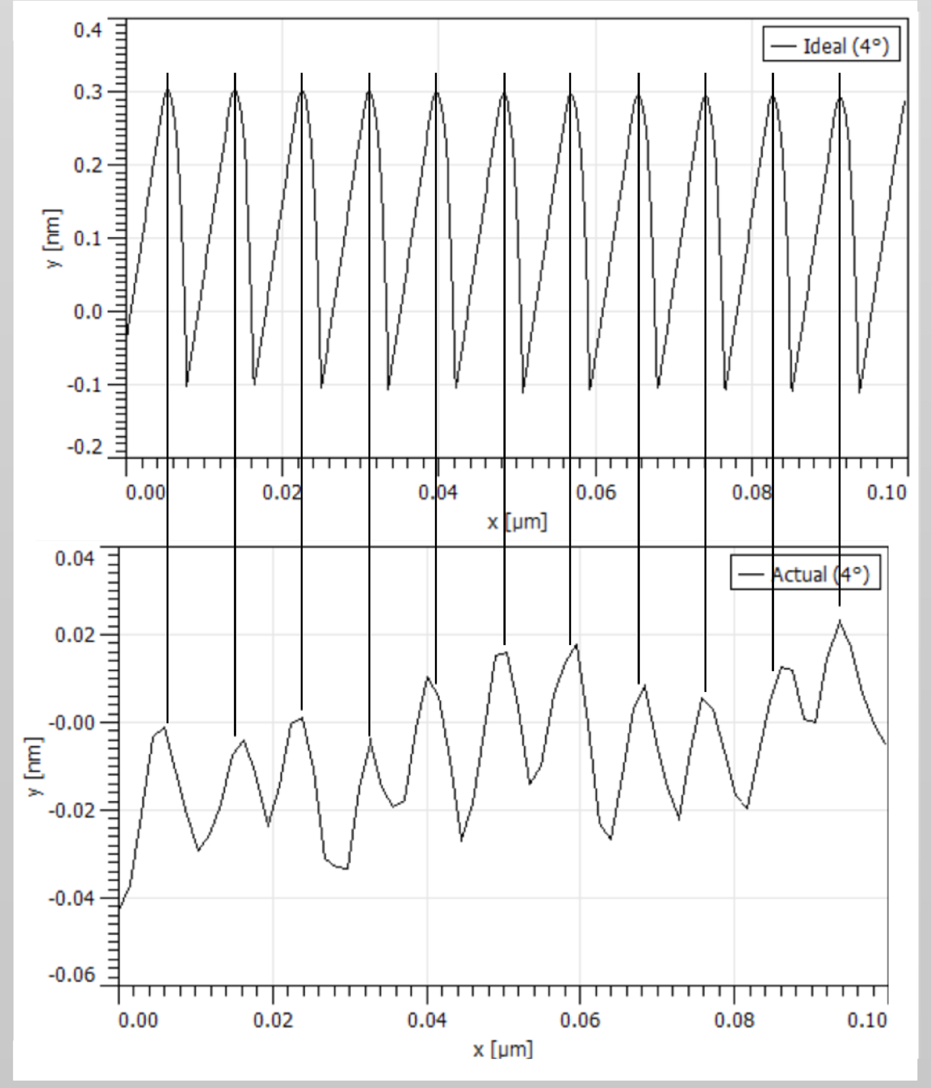
5.9° miscut substrate, viewed along the (010) direction.



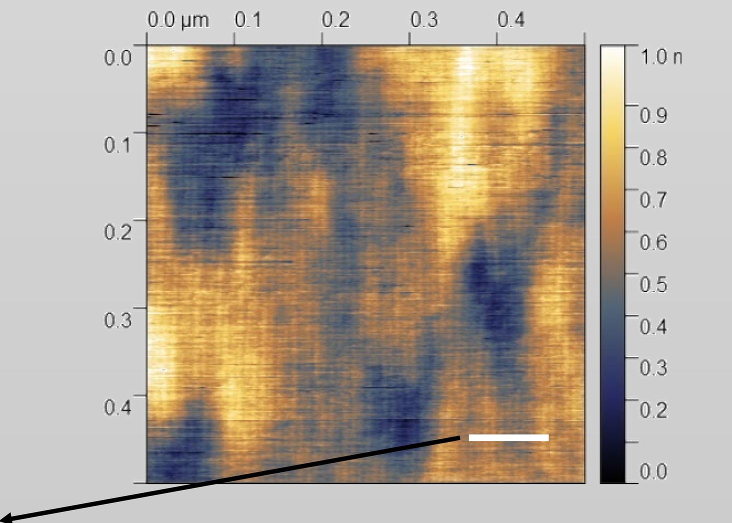
Success of cleaning and etching process from typical out-of-box substrate (a) is demonstrated above. (a-b) demonstrate the acetone/isopropanol/water cleaning process, (c-d) demonstrate the cleaning and phosphoric acid etch on a magnesium-doped substrate, (e-f) demonstrate the cleaning and hydrofluoric acid etch on an iron-doped substrate, and (g-h) demonstrate the failure of phosphoric acid etching on an iron-doped substrate after anneal. Unlike the H_3PO_4 etch, the HF etch increases surface roughness, however, we believe it is still essential for removing particles not able to be removed during the following anneal.



Utilizing previous reports of O_2 annealing conditions[5] and testing across a range of miscuts, an annealing model was created: $t\ (\text{hours}) = 0.00366 \cdot \cot(\theta)^2$, at 900 °C.
Figure above demonstrates success following the above model. Substrates were annealed at 900 °C for (a) 3.7 hr, (b) 3 hr, (c) 1.3 hr, (d/f) 1 hr, (e/g) 0.75 hr, (h) 0.34 hr. Shaded AFM scans (f-h) allow for improved viewing of step shapes, though remove numerical height accuracy of surface. Sample roughness at a scale of 20 μm x 20 μm are all <600 pm.



4° Mg-Doped, Line profile from white bar



Step widths match closely with theoretically calculated values, as seen in ideal and actual AFM profiles (left). Step heights calculated from widths and miscut angles range from 0.54 nm to 0.61 nm, consistent with the theoretical, ideal 0.59 nm.[5]

Conclusion and References

We demonstrate the successful cleaning, wet etching, and annealing of (100) vicinal semi-insulating β-Ga_2O_3 substrates doped with magnesium or iron. The use of isopropanol, acetone, and DI water in cleaning and the use of wet etching in H_3PO_4 (CrysTec Mg-doped substrates) or HF (NCT Fe-doped substrates) demonstrate the successful removal of surface contaminants with low surface roughness. We demonstrate a successful recipe for 900 °C anneals with lengths dependent on miscut angle, forming straight, consistent-height steps on a range of miscut angles from 1.8° through 5.9°, paving the way for future high-quality epitaxial growth. Future studies will apply these high-quality substrates in suboxide molecular beam epitaxy for the growth of Si-Ga_2O_3 at silicon doping concentrations relevant for devices. Using these films, we will investigate the suppression of twin boundary formation and the resulting mobilities of films as a function of miscut angle.

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